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Static and dynamic critical behaviour of the freezing transition in the random-exchange $Ho_5Co_{50}Al_{45}$ alloy

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Abstract. Random-exchange metallic spin glass $Ho_5Co_{50}Al_{45}$ is investigated by static and dynamic magnetization measurements. The specimen is initially characterized as spin-glass-like below the freezing point $T_f \sim 14$ K. The freezing phenomenon at T_f is examined by static and dynamic scaling analysis. The results support the existence of a paramagnetic–spin-glass phase transition at $T_g \approx 14.2$ K, with critical exponents of the transition as $\delta = 6.4$, $\phi = 3.3$ and $z\nu = 11.8$.

1. Introduction

$Co_{50}Al_{50}$ alloy is a strong paramagnetic with the CsCl (bcc) crystal structure [1]. The Co atoms are situated at the corners of the cubic lattice and the Al atoms are in its centre. By doping with 3d elements and Pd for Al, the $T_xCo_{50}Al_{50-x}$ system develops a well observable spin-glass- (SG-) like state [2].

In this work the parent $Co_{50}Al_{50}$ matrix is doped with 5 at.% of the 4f, rare earth magnetic element Ho for Al. The obtained $Ho_5Co_{50}Al_{45}$ alloy is characterized by standard SG-like behaviour. Then the problem of the freezing phenomenon below the freezing temperature, T_f, [3, 4], is approached by detailed study of the specimen's response to dc and ac magnetic susceptibility measurements.

It is known [5] that the magnetization of the specimen, $M(T, H)$, can be presented in terms of the odd powers of the field:

$$M(T, H) = \chi_1(T)H - \chi_3(T)H^3 + \chi_5(T)H^5 - \cdots \quad (1)$$

$\chi_1(T)$ in equation (1) is the linear susceptibility and the rest of the coefficients stand for the nonlinear terms. It is obvious from equation (1) that the contribution of the nonlinear terms becomes significant when the measuring field exceeds a low-limit value. For this low-limit value, only the contribution of the first nonlinear coefficient $\chi_3(T)$ can be taken into account [6–8]. The temperature dependence of the linear susceptibility $\chi_1(T)$ and the first nonlinear term $\chi_3(T)$ in equation (1) can be obtained from the least-squares fit of the isothermal magnetization curves [6–8]. The increase of the nonlinear coefficient $\chi_3(T)$ should be three orders of magnitude for a typical SG [5, 6].

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As the total magnetic susceptibility, $\chi(T, H)$, and its linear component, $\chi_1(T)$, are non-divergent, if a thermodynamic phase transition is undergone at the temperature of T_g, the nonlinear terms should diverge at T_g [9]. So an important physical quantity to identify a phase transition is the total nonlinear susceptibility, $\chi_{nl}(T, H)$

$$\chi_{nl}(T, H) = \chi_1(T) - M(T, H)/H. \tag{2}$$

To describe $\chi_{nl}(T, H)$ in the critical region, different scaling relations are proposed [4, 10]. To minimize the experimental error [6, 8], preference is given to the scaling relation proposed in [10]:

$$\chi_{nl}(T, H) \propto H^{2/\delta} f[\iota/H^{2/\phi}]. \tag{3}$$

In equation (3), the reduced temperature, defined as $T/T_g - 1$, is denoted by ι; δ and ϕ are critical exponents of the spin-glass transition. It is assumed in equation (3) that the freezing temperature might coincide with the phase-transition temperature. The parameter ϕ is related to the parameter δ as $\phi = \delta\beta$ [6, 10], where β is the exponent of the SG order parameter below T_g.

The asymptotic behaviour of equation (3) [6, 10]:

$$f(x) = \text{const} \qquad \text{with } x \to 0 \tag{4}$$

and

$$f(x) = x^{-\gamma} \qquad \text{with } x \to \infty \tag{5}$$

has simple physical meaning.

Equation (4) regards the case when $T = T_g$ at a finite applied magnetic field. It is known [9] that if an alloy is undergoing a phase transition, at the temperature of the transition $\chi_{nl}(T, H)$ will depend on H as:

$$\chi_{nl}(T = T_g, H) \propto H^{2/\delta}. \tag{6}$$

The parameter δ in equation (6) is the exponent at the temperature of the transition.

Equation (5) applies the case for temperatures $T > T_g$ and for small applied fields. Then the total nonlinear susceptibility of an alloy $\chi_{nl}(T, H)$ (and especially the $\chi_3(T)$ term) will diverge around the transition temperature as [9]:

$$\chi_{nl}(T) \propto \iota^{-\gamma}. \tag{7}$$

The parameter γ in equation (7) is the susceptibility exponent.

The critical exponent δ can be estimated from the field dependence of $\chi_{nl}(T = T_g, H)$ for the temperature of the freezing point. The obtained value of δ can be used to scale the nonlinear isotherms for $T > T_f$, according to equation (3), with varying T_g and ϕ. The best scaling will give the values of the free parameters T_g and ϕ.

The critical exponents γ, δ and ϕ are related through the hyperscaling relation [11]:

$$\gamma = \phi(1 - 1/\delta). \tag{8}$$

The value of γ obtained by the asymptotic slope of the scaling plot (equation (3)) should be close to the value of γ, defined by equation (8). This is used partly as a criterion that the investigated alloy is an SG [6].

Spin-glass-like materials exhibit dynamical critical properties [6, 8]. The in-phase component of the ac susceptibility shows a peak indicative of the freezing temperature T_f. Its position on the temperature axis coincides with inflection point after the peak of the out-of-phase component of the ac susceptibility, for the same frequency of the measuring field. In the framework of a general slowing-down model, which assumes a true phase below T_g, the relaxation times $\tau(= 1/w)$ diverge as the critical temperature is approached from above [12] as:

$$\tau = \xi^z \propto [T_f(w)/T_g - 1]^{-z\nu}. \tag{9}$$

In equation (9) z is the dynamic exponent relating τ to the correlation length ξ; ν is the critical exponent for ξ. The product $z\nu$ is often regarded as one parameter.

It is known [9] that only two of all the parameters defining the SG behaviour and the SG transition are independent. The related SG parameters can be calculated using the relations connecting them [9]. The comparison of the results from the relations and the independent assessment can indicate the existence of a true SG phase below the freezing point.

2. Experiment

The random-exchange $Ho_5Co_{50}Al_{45}$ alloy is obtained by melting the high purity ingredients in an arc-melting furnace. The ingot, in 300 Torr argon atmosphere, was sealed in a quartz ampoule, and heat-treated at 1200 °C for 92 hours. Afterwards, the temperature of the treatment was decreased to 600 °C with a rate of ∼200 degrees h^{-1}. Upon reaching 600 °C the ampoule was quenched into air to prevent formation of unnecessary crystallographic phases. The ingot was checked for magnetic homogeneity. X-ray measurements confirmed the CsCl crystal structure, characteristic of the $T_xCo_{50}Al_{50-x}$ system [2]. A small, 5.6 mg, needle-like sample was used for the magnetic investigations.

The magnetic measurements were made with a commercial Quantum Design SQUID magnetometer with measuring sensitivity of about 10^{-6} emu, measuring temperature interval from 1.5 to 800 K, and applicable magnetic-field interval (−7, 7) T.

Diagnostic dc zero-field-cooled (ZFC) and field-cooled (FC) magnetization measurements were made using the field of 1 Oe, by a standard magnetization-measurement technique. The temperature dependence of the thermoremanent magnetization, M_{TR}, was measured using the same technique, after cooling the sample in the measuring field to 4.2 K and reducing the field to zero.

Dc relaxation of $M_{ZFC}(t, T)$ [8] is measured at some temperatures below T_f. The specimen was cooled in zero field for ∼600 s, and kept at the measurement temperature for a waiting time of $t_{wait} \approx 2400$ s. A weak probing field of 0.5 Oe, stabilized in the non-overshoot mode for ∼100 s, is applied after t_{wait}. The resultant waiting time, t_w, is defined as the sum of t_{wait} and the time ∼100 s needed for stabilizing the magnetic field. The $M_{ZFC}(t, T)$–t plot was recorded for a time period of $t \sim 10^4$ s.

Multiple $M_{FC}(T, H_i = \text{const}, \Delta t \approx \text{const})$–$T$ measuring procedures are used to obtain the points in the field–temperature space (H–T). (H_i is a field in the range of 1 to 5000 Oe and Δt is the time to take a point in an M_{FC}–T plot.) This measuring technique provides the values of the acting magnetic fields more precisely with the used equipment in this investigation [7]. Conventional measuring of the $M(T, H)$–H isotherms seems not good enough against hysteresis of the superconducting magnet, which could affect the correct values of the magnetic fields, and, therefore, the results.

In the M_{FC}–T measuring cycles, the sample was initially cooled down from room temperature in zero field, to a temperature of $T \sim 3T_f$. The magnetic interactions in the specimens were considered to be negligible at $T \sim 3T_f$. Then a magnetic field in the range of 1 to 5000 Oe was switched on, and the M_{FC}–T dependence, at a fixed applied field, was measured, down to a temperature below T_f.

Afterwards the sample was warmed back in the same field to $\sim 3T_f$, the next measuring field was switched on, and the corresponding M_{FC}–T plot, was recorded.

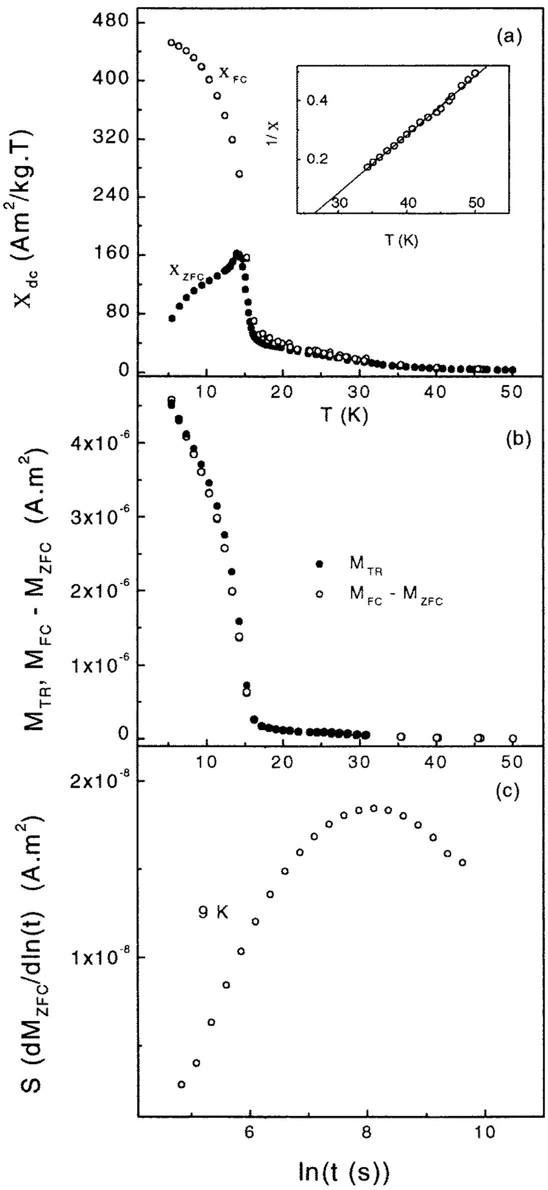

Figure 1. (a) Temperature dependences of the dc zero-field-cooled and field-cooled magnetic susceptibilities X_{ZFC} and X_{FC} for $Ho_5Co_{50}Al_{45}$. The inset shows the temperature dependence of the reverse magnetic susceptibility above 30 K. The line in the inset is the linear extrapolation of the data. (b) Temperature dependences of the thermoremanent magnetization M_{TR} (solid circles), and of $M_{FC} - M_{ZFC}$ (open circles). (c) The rate of change of the magnetization $S \sim \mathrm{d}M_{ZFC}/\,\mathrm{d}\ln(t)$ against $\ln(t(s))$ for the temperature of 9 K.

The ac susceptibility measurements were made in zero dc field, using an amplitude of the ac field, $h_{ac} \approx 0.1$ Oe, and frequencies of the ac field, $\nu = \omega/2\pi = 0.85$; 4; 10; 22; 40; 80; 120; 180 and 210 Hz. The results for M_{ac} against T, obtained using amplitudes of 0.5 Oe and 1 Oe, showed linearity of M_{ac} against h_{ac} for the investigated field region.

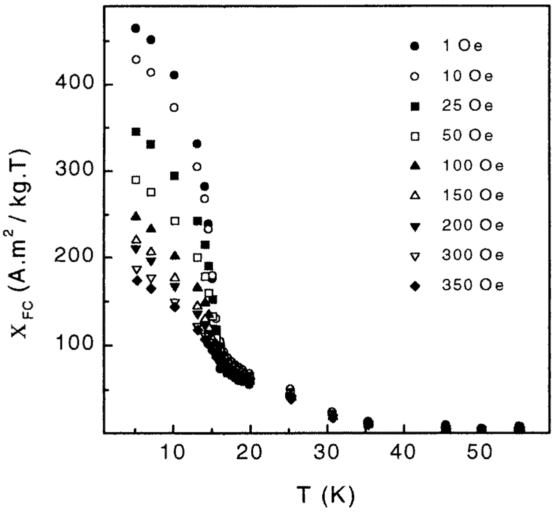

Figure 2. Temperature dependence of the field-cooled magnetization, M_{FC}, of $Ho_5Co_{50}Al_{45}$, measured in the fields of 1; 10; 25; 50; 100; 150; 200; 300 and 350 Oe.

3. Results and discussion

Figure 1 presents the general magnetic behaviour of the $Ho_5Co_{50}Al_{45}$ alloy. In figure 1(a) are shown the temperature dependences of the dc zero-field-cooled and field-cooled magnetic susceptibilities, $X_{ZFC}(T, H, t)$ and $X_{FC}(T, H, t)$. There is a peak at ∼14 K in the $X_{ZFC}(T, H, t)$–T plot, and there is a splitting of the $X_{ZFC}(T, H, t)$ and $X_{FC}(T, H, t)$ plots, characteristic for SGs.

The doping with the localized 4f element Ho for Al results in some specific differences when we compare $Ho_5Co_{50}Al_{45}$ with the $Al_{45}Co_{55}$ alloy. The magnitude of $X_{ZFC}(T, H, t)$ at the peak is ≈2 times smaller for $Ho_5Co_{50}Al_{45}$ than the corresponding value for $Co_{55}Al_{45}$ [2]. The freezing temperatures differ significantly: $T_f \approx 14$ K for $Ho_5Co_{50}Al_{45}$ and $T_f \approx 55$ K for $Co_{55}Al_{45}$ [2].

The inset of figure 1(a) shows the temperature dependences of $1/X_{ZFC(FC)}(T, H, t)$ above 30 K, where the alloy is paramagnetic. The Curie–Weiss temperature θ_c, obtained from the linear extrapolation of $1/X_{ZFC}(T, H, t)$ against T, is ≈26 K. Its positive value shows ferromagnetic-type interactions in this material. This value of θ_c is on the low side of the reported values for spin-glass materials [9].

In figure 1(b) are shown the temperature dependences of the measured thermoremanent magnetization $M_{TR}(T, t)$, and the calculated difference of the $M_{FC} - M_{ZFC}$ data. Figure 1(b) presents good coincidence of $M_{TR}(T, t)$ and $M_{FC} - M_{ZFC}$ over the measured temperature interval, analogously to results obtained for some 3d-element-doped $T_xC0_{50}Al_{50-x}$ SGs [2].

The change of M_{ZFC} against time, on an imposed field disturbance of 0.5 Oe, obeys standard SG behaviour [8, 9]. In figure 1(c) is shown the calculated relaxation rate, $S \sim \mathrm{d}M_{ZFC}/\mathrm{d}\ln(t)$, against $\ln(t)$, for temperature of the maximal rate of the relaxation [13], ∼9 K.

The rate of relaxation, S, should have a maximum at $t \approx t_w$, which is typical for SGs [8, 9]. (Some contribution to t_w, due to a finite (small) rate of cooling below the freezing point is possible, which can explain the slight displacement of the relaxation rate, $S(t, H)$, from the position $t \approx t_w$.)

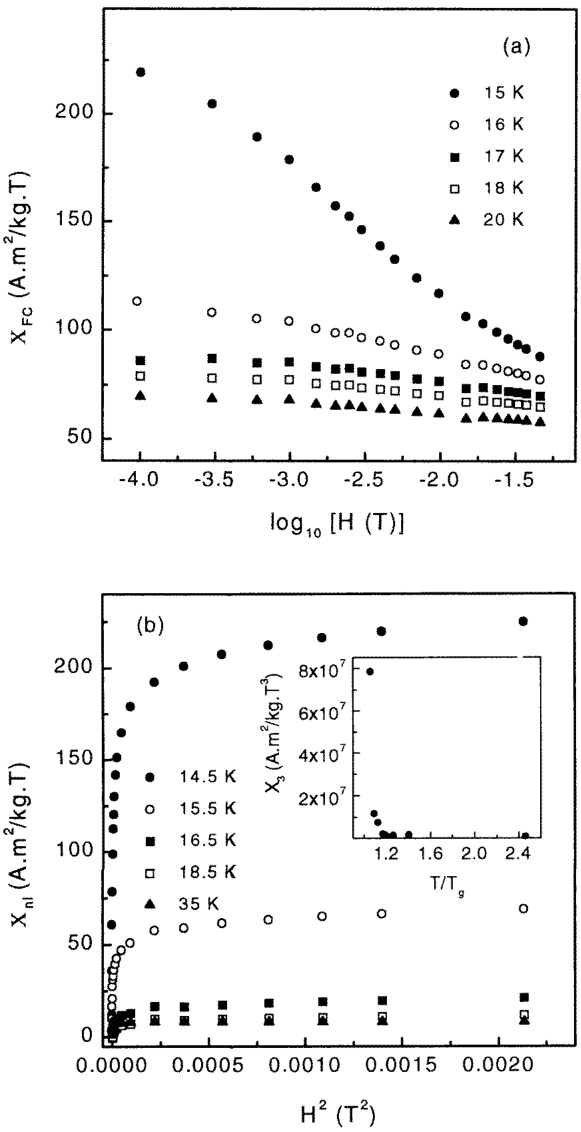

Figure 3. (a) Total field-cooled magnetic susceptibility at selected temperatures as a function of the logarithm of the applied dc magnetic field. (b) The nonlinear susceptibility, χ_{nl} against H^2 for the five indicated temperatures. The inset shows the χ_3 coefficient against the reduced temperature T/T_g.

3.1. dc FC magnetic measurements

In figure 2 are presented the temperature dependences of the dc FC magnetic susceptibilities, measured in applied fields from 1 to 350 Oe. The FC susceptibility of $Ho_5Co_{50}Al_{45}$ shows freezing of the magnetic moments below 15 K (figure 2). When the measuring field is increased, the susceptibility flattens below the freezing point, which is indicative of the increasing contribution of its nonlinear part.

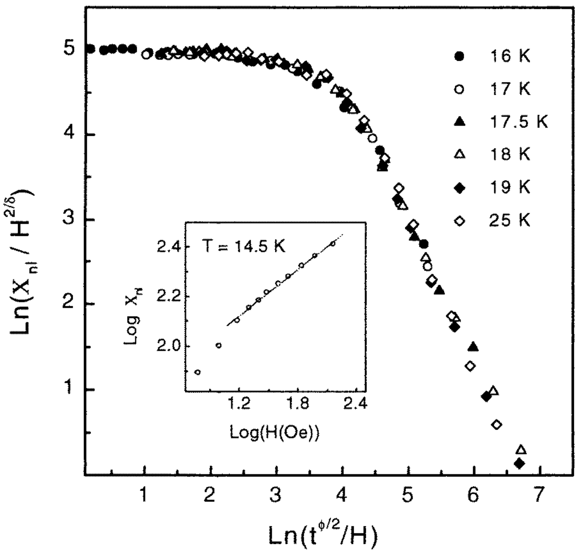

Figure 4. Scaling plot of the nonlinear part of the susceptibility, χ_{nl}, for $Ho_5Co_{50}Al_{45}$, according to equation (3), for the temperature interval $1.18\,T_g \leqslant T \leqslant 1.32\,T_g$. The inset shows the $\log \chi_{nl}$–$\log H$ plot for 14.5 K, from which the value of δ is determined.

In figure 3(a) is depicted the magnetic field dependence of $X_{FC}(T, H)$ for the temperatures of 15, 16, 17, 18 and 20 K. A sharp decrease of the maximal value of the isotherms, combined with flattening, is observed above 15 K. As obvious from the figure, the nonlinear part of the susceptibility is substantial mainly for the temperatures close to the freezing point.

The nonlinear susceptibility isotherms are obtained from the total susceptibility, according to equation (2). The linear susceptibility for each measured temperature is obtained from the approximation to zero of the low-field values of $X_{FC}(T, H)$. Figure 3(b) displays the derived nonlinear susceptibility, $\chi_{nl}(T, H)$–H^2 isotherms, for the temperatures of 14.5, 15.5, 16.5, 18.5 and 35 K. There is a sharp decrease of the nonlinear susceptibility above the freezing point. Also, there is a small low-field region of linear dependence of $\chi_{nl}(T, H)$ against H^2 in figure 3(b), where the first nonlinear-susceptibility term $X_3(T)$ is predominant.

The temperature variation of the first nonlinear term $X_3(T)$ is shown in the inset of figure 3(b). The calculated values of $X_3(T)$ show the increase of $X_3(T)$ when the freezing temperature is approached upon cooling is about 600 orders of magnitude.

The critical exponent δ is obtained by using equation (6). The inset of figure 4 shows the dependence of $\log_{10}\chi_{nl}$–$\log_{10} H$ for the temperature of 14.5 K, corresponding to the nearest measured temperature to that at which X_{ZFC} against T shows a maximum, and $X_3(T, H)$ diverges [5]. From the $\log_{10}\chi_{nl}$–$\log_{10} H$ plot in figure 4, the value of $\delta \approx 6.45 \pm 0.3$ is obtained. The limiting values of δ (6.75 and 6.15) are obtained from criteria of indistinguishability of the experimental data when scaled according to equation (3). The value of $\delta \approx 6.45 \pm 0.3$ is valid for the field region of 15–200 Oe. Below 15 Oe, larger experimental error in the values of H adds to the nonlinearity of the $\log_{10}\chi_{nl}$–$\log_{10} H$ plot.

The data for $\chi_{nl}(T, H)$ were scaled according to equation (3). Figure 4 displays the $\ln(\chi_{nl}/H^{2/\delta})$–$\ln(\iota^{\phi/2}/H)$ isotherms for some temperatures between $1.1\,T_g < T < 2\,T_g$. The phase transition temperature, T_g, is assumed to be approximately 14 K.

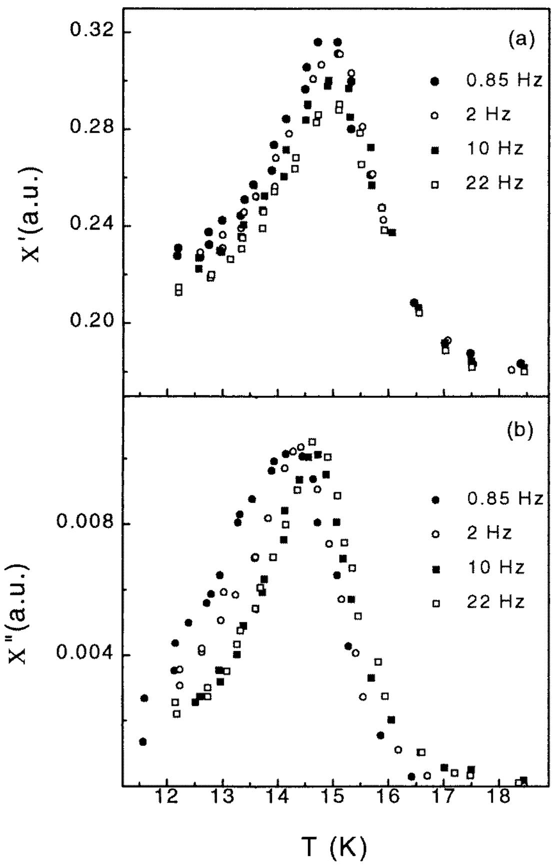

Figure 5. Temperature dependence of the in-phase-susceptibility, $X'(w, T)$ (a), and the out-of-phase susceptibility, $X''(w, T)$ (b), of $Ho_5Co_{50}Al_{45}$, measured with the frequencies of $\omega/2\pi =$ 0.85, 2, 10 and 22 Hz, and the amplitude of $h_{ac} = 0.1$ Oe.

The parameters T_g and ϕ are changed to obtain the best data collapse, shown in figure 4. The results, depicted in figure 4, correspond to $T_g = 14.2 \pm 0.1$ K, and $\phi = 3.3 \pm 0.1$. When T_g or ϕ increase, the isotherms shown in figure 4 are displaced to the left, and vice versa.

Then, according to equation (8), the susceptibility exponent $\gamma = 2.8$. The spin-glass ordering parameter, $\beta = \phi/\delta$, is $\approx$0.51.

The specific heat exponent, α, is calculated by the relation [9] $\alpha = 2 - 2\beta - \gamma$. With the obtained values of β and γ, α equals -1.82. The negative value of α indicates that there should be no divergence in the specific heat, since it scales as $\iota^{-\alpha}$ [14].

The exponent of the correlation length, $\xi = \iota^{-\nu}$ [9], can be calculated by the relation $\nu = (2 - \alpha)/d$ (d is the spatial dimension). With $\alpha = -1.82$ and $d = 3$, we get $\nu = 1.27$.

The obtained values of the SG parameters for $Ho_5Co_{50}Al_{45}$ are within the range of the parameters, reported for other SGs [6–9].

The reliability of the scaling behaviour is then tested at the limiting points. It is seen from figure 4, that the asymptotic slope, which is equal according to equations (3) and (5) to $-2\gamma/\phi$ when $x \to \infty$, is ≈ -1.49. This slope results in the value of $\gamma \approx 2.46$, which is $\sim$12% lower than the value of 2.8, obtained above, using the hyperscaling relation (equation (9)). In the

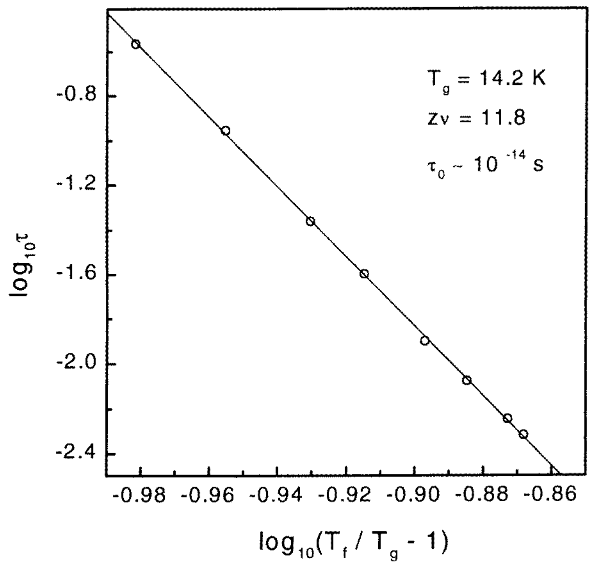

Figure 6. The best fit to $\tau/\tau_0 = (T_f/T_g - 1)^{-z\nu}$ of the measured freezing temperatures T_f, at different observation times $t = \tau$. $T_g = 14.2$ K, $z\nu = 11.8$ and $\tau_0 = 10-14$ s.

limit of $x \to 0$, an asymptotic constant value is observed from figure 4. So, the scaling plots in figure 4 may have physical meaning, and from the point of view of the scaling theory, it seems possible to conclude that a conventional SG transition takes place at $T_g = 14.2$ K for $Ho_5Co_{50}Al_{45}$.

3.2. ac magnetic measurements

The ac magnetic susceptibility measurements on $Ho_5Co_{50}Al_{45}$, made as described above, display standard SG behaviour [6, 8, 9]. The in-phase ($X'(w, T)$) and out-of-phase ($X''(w, T)$) components of the ac susceptibility are frequency dependent. The position of the peak in $X'(w, T)$ at the temperature of $T \approx 15$ K coincides with the inflection point after the peak in $X''(w, T)$ against T for the corresponding frequency. In figures 5 (a) and (b) this is shown for $Ho_5Co_{50}Al_{45}$ for the frequencies of $\omega/2\pi = 0.85$, 4, 10, 22 and 30 Hz, and the amplitude of the ac field, $h_{ac} = 0.1$ Oe.

The data for all observation times τ, used in the experiment, with the relevant freezing temperatures T_f, were analyzed assuming a conventional slowing-down process (equation (9)). The experimental data for τ and T_f were plotted in a $\log_{10}\tau - \log_{10}(T_f/T_g - 1)$ scale. Different T_g values were tested for the best linear approximation of the data points. The best-fit line to the experimental data (figure 6) resulted in the values of $z\nu = 11.8$ and $\tau_0 = 10^{-14}$ s, with $T_g = 14.2$ K.

The obtained value of $z\nu$ in $Ho_5Co_{50}Al_{45}$ is typical for SGs [8]. Although the value of the shortest relaxation (observation) time in $Ho_5Co_{50}Al_{45}$, τ_0, is among the reported ones [8] it is one decade shorter than the spin flip time of the atomic magnetic moments (10^{-13} s). This small discrepancy can be related to the sensitivity of these calculations to the experimental data, obtained at small frequencies for which the experimental error is larger.

4. Conclusions

General characterization of the random-exchange metallic alloy $Ho_5Co_{50}Al_{45}$ by dc magnetization measurements shows spin-glass-like behaviour below ~14 K with the characteristic breakaway between X_{ZFC} and X_{FC}, and ageing on imposed field perturbation. The calculated dc nonlinear susceptibility of $Ho_5Co_{50}Al_{45}$ was used to analyse the freezing phenomenon at $T_f \approx 14$ K. The results from the static and dynamic scaling analysis prove that the freezing at ~14.2 K can be considered as a phase transition to the SG phase. The values of the parameters describing the PM–SG transition, $\delta = 6.45$, $\phi = 3.3$, $z\nu = 11.8$ and $\tau_0 = 10^{-14}$ s, are among those reported for conventional spin-glasses [6–9].

Acknowledgments

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