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ORIGINAL PAPER

Determination of the Transition Temperature of a Weak Ferromagnetic Thin Film by Means of an Evolution of the Method Based on the Arrott Plots

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Abstract The ferromagnetic transition temperature (T_c) of a weak ferromagnetic thin film $Cu_{0.38}Ni_{0.62}$ has been estimated by using an evolution of the Belov, Goriaga, and Arrott (BGA) model. In fact, the typically used measurements of the zero-field-cooled-field-cooled magnetic moment (m) as a function of temperature (T), the magnetic hysteresis loops ($m(H)$) as a function of field, and the temperature dependence of the remanent magnetic moment, performed on our sample, did not allow us to individuate precisely the value of T_c, mainly due to the influence of thermal effects and noise on the weak ferromagnetic response of the sample, resulting in a large uncertainty in the estimation of T_c. On the other hand, also the commonly used method of determining the T_c by means of a linear extrapolation of the high-field region of m^2 as a function of the normalized magnetic field (H/m) at different temperatures (Arrott curves), working though for our analyzed material, could not completely fulfill the basic assumptions of the BGA model in general. For these reasons, we have considered the analysis of the derivatives of the Arrott curves, starting from observing that the T_c of the sample corresponds to the temperature where the curvature of the Arrott curves at low fields inverts. In this way, the sensitivity in the determination of the Curie point from the inductive measurements of the magnetic moment is improved while the correct assumptions of the Arrott model are also fully respected.

Keywords Magnetism · Curie temperature · Magnetization measurements · Arrott plot · Thin film · Weak ferromagnet · CuNi

1 Introduction

In the recent years, weakly ferromagnetic alloys, like CuNi and PdNi, have been the subject of analysis, also for what concerns their electrical and magnetic properties, on thin films, polycrystals, massive samples, and nanoalloys [1–6]. The renovated appeal about these weak ferromagnets (Fs) comes also from their relatively low value of the exchange energy, which suggested to characterize their properties in conjunction with superconducting (S) materials, so that the interplay between superconducting and ferromagnetic properties could be more easily investigated in S/F-layered structures [7–13]. In order to analyze the magnetic properties of these materials, measurements of the magnetic susceptibility in dc and ac field are widely used. The dc magnetic techniques are generally used when it is not necessary to take into account the dynamic behavior of the system. On the other hand, the ac susceptibility measurements, both in first and higher harmonics, are used if one is interested to the dynamic magnetic response of the analyzed sample both for magnetic [14–17] and for superconducting [18–22] materials. In order to determine the ferromagnetic transition temperature (or Curie temperature, T_c), different dc approaches can be considered. The first approach consists in the comparison of the sample magnetization

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measurements as a function of the temperature ($M(T)$) performed in zero-field-cooling (ZFC) and field-cooling (FC) conditions. More precisely, the sample is first cooled down to a sufficiently low temperature (presumably lower than T_c) in the absence of any magnetic field, and then the field is applied and the measurement is taken for increasing temperature (ZFC) up to a value presumably higher than T_c. After that, still leaving the same field on, the measurement is performed by cooling down the sample (FC) back to the starting temperature. In this way, the temperature at which the ZFC and FC curves start to separate from each other corresponds to the estimation of T_c [23–26].

Another approach consists in the measurements of the magnetization loops (M vs H loops) at different temperatures around the value T^* of the presumed transition temperature in zero magnetic field. The field is applied up to a maximum value of $H_{\max}$ and back to $-H_{\max}$ and then again to $H_{\max}$ and finally to zero, while the corresponding magnetization (M) is detected. Before the loop measurements at each temperature, the magnetic history of the sample is canceled by warming it at a temperature above T^*. With this approach, the ferromagnetic transition temperature (T_c) is estimated as the temperature at which a magnetic hysteresis starts to appear in the M-H cycle due to the presence of a ferromagnetic state [27].

A further method to determine the ferromagnetic transition temperature is based on the study of the temperature behavior of the remanent magnetization (M_r). Within this method, the sample is firstly cooled down to the lowest temperature (presumably lower than T_c) reachable by the apparatus in zero magnetic field; after that, the field is increased until the magnetization of the sample reaches the saturation value and then is lowered to zero again. This produces a remanent magnetization (M_r) in the sample due to the ferromagnetic ordering. Finally, the temperature of the sample is increased while the temperature dependence ($M_r(T)$) of M_r in zero field is measured, showing a decreasing behavior due to the increase of the thermal excitations of the elementary magnetic dipoles with increasing temperature. When the temperature reaches T_c, the magnetic correlation between the dipoles disappears and the M_r value is expected to be equal to zero [28].

Beyond the most common methods described above, the one described by Belov, Goriaga, and Arrott (BGA) [29–31] can be adopted in order to have an accurate estimation of the Curie temperature. This method, which is appropriate only at weak magnetic field, is based on the Landau theory of the second-order phase transitions. Within this approach, the free energy (f_m) is expanded as a function of the order parameter, which here is the magnetization (M)

$$f_m = \frac{\alpha}{2}M^2 + \frac{\beta}{4}M^4 \ldots - \mu_0 HM \tag{1}$$

with α and β as positive functions of the temperature. Then, the equilibrium magnetization is the value that minimizes f_m with respect to M

$$\alpha M + \beta M^3 = \mu_0 H \tag{2}$$

where M is small, as it is expected in weak fields close to T_c, and one can consider only the first order of the expansion in (2) which can be written as

$$\alpha M = \mu_0 H. \tag{3}$$

From the comparison with the Curie-Weiss law, the coefficient α can be determined as

$$\alpha = \frac{\mu_0}{C}(T - T_c) \tag{4}$$

where C is the Curie constant [27]. By using (4), (2) can be written in the form

$$M^2 = \frac{\mu_0}{\beta}\frac{H}{M} - \frac{\mu_0}{\beta C}(T - T_c). \tag{5}$$

According to (5), by plotting M^2 versus H/M (Arrott plots), a series of straight lines are expected at different temperatures, and a straight line passing through the origin is expected at $T = T_c$.

The straight lines predicted by this simplified model are not frequently observed indeed, but nevertheless, a reliable value of T_c can be obtained by the linear extrapolation of the curve in correspondence to a larger value of the magnetization. Moreover, as we will see more ahead in this work, at sufficiently high fields, in addition to a linear behavior, a change in the concavity of the Arrott curves may be present, above and below T_c.

In this paper, we will determine experimentally the Curie temperature of a weak ferromagnetic thin film Cu_xNi_{1-x} (x = 0.38) by means of different techniques. Performing and analyzing the measurements of the magnetic moment versus temperature and field, we have underlined that the correct estimation of the Curie temperature can be influenced by several factors, like the thermal and electromagnetic noise as well as artefacts coming from a not-complete control of the experimental parameters used for the magnetic measurement. Moreover, also the use of the Arrott curve to determine T_c is often done without full respect to some basic assumptions of the BGA model. For these reasons, in order to suggest a more objective, usable, and reliable criterion for the evaluation of T_c by means of dc magnetic measurements, the way in which the Arrott plots are treated have been slightly modified, and a more precise and correct value of T_c has been so obtained.

2 Experimental Details

In this work, we have analyzed the magnetic response of a film of $Cu_{0.38}Ni_{0.62}$ with dimensions 3×5 mm^2 and a thickness of 440 nm, deposited by UHV dc diode magnetron sputtering on a Si(100) substrate in an Ar atmosphere. More details about the fabrication procedure are reported elsewhere [32]. The measurements of the magnetic momentum (m) as a function of the temperature have been performed on the $Cu_{0.38}Ni_{0.62}$ sample in the presence of a dc magnetic field by using a Quantum Design PPMS 9T equipped with the vibrating sample magnetometer (VSM) option. The measured temperature range goes from 2 to 400 K, and a dc magnetic field (H) up to about 2 T maximum, parallel to the film plane, has been applied, paying attention to avoid any field overshoot [33]. Moreover, a particular care has been put in order to delete the effect on the used material of an eventual magnetic history, by heating the sample at temperature of 350 K for about 20 min before each measurement, while performing demagnetization cycles on the superconducting magnet of the PPMS to reduce its trapped field below 1 Oe [33, 34].

3 Results and Discussion

First of all, the Curie temperature (T_c) due to the ferromagnetic transition of the $Cu_{0.38}Ni_{0.62}$ sample has been evaluated by comparing the $m(T)$ measurements in ZFC and FC conditions, performed with the procedure previously described. In particular, the sample has been cooled down to $T = 2$ K, and then a 100 Oe magnetic field has been applied and the measurement has been taken by warming up the sample up to 400 K (ZFC) and then again cooling it back to 2 K (FC). The results are reported in Fig. 1, where a reversible temperature behavior is visible above approximately 200 K, with a low paramagnetic signal at temperatures higher than about 225 K. In order to determine the Curie temperature, corresponding to the temperature where the ZFC and FC curves start to separate from each other, the region between 180 and 240 K has been magnified, and it is shown in the inset of Fig. 1. Here, two different contact points between the ZFC and FC curves are visible (as indicated by the arrows), so making ambiguous the determination of T_c, which can only be placed in the range of 200–225 K. The exactness of this range has been verified also by means of magnetization loop [moment (m) vs field (H)] measurements performed at different temperatures, as described in the previous section, and is reported in Fig. 2. The $m(H)$ loops reveal that a hysteresis is evident in the curve measured at temperatures as low as 50 K. The highest temperature at which the hysteresis still objectively appears is $T = 210$ K, whereas it is not clear at 220 K due to its found dependence on the field sweep rate, allowing us to state that $T_c = 215 \pm 5$ K. It is worth to underline that in general, the presence of a hysteresis in a $m(H)$ curve depends both on the field sweep rate (not specifically treated in this work) and on the magnification of the plot. In fact, in the inset of Fig. 2, it can be seen that also the $m(H)$ at 300 K presents a hysteresis, i.e., a non-zero remanent magnetization. In this sense, one must be very careful when this criterion is used for the estimation of the T_c, especially when the sweep rate is not taken into account and reported as the parameter used for the experimental curves.

In order to reduce the margin of error in the determination of T_c, the method based on the temperature dependence of the remanent magnetic moment (m_r) has been used. Within this method, the sample has been first cooled down to 2 K in zero field, and after waiting enough time for its thermal stabilization, a magnetic field of 2 T has been applied to magnetically saturate the sample. Then, the field has been slowly reduced to zero without any field overshoot, so determining the presence of a remanent magnetization, and after that, the temperature of the sample started to be increased, allowing the measurement of the $m_r(T)$ curve as reported in Fig. 3. As expected, the m_r decreases for increasing temperature, due to thermal energy which determines an increase of the average excitation of the magnetic elementary dipoles and therefore a decrease of the magnetic ordering, but it remains positive as long as the ferromagnetic interaction energy is larger than the thermal energy. At $T = T_c$, the two energies become equal, so the correlation between the magnetic dipoles is lost, and the sample starts to behave as a paramagnet with a remanent magnetic moment reducing to zero. The linear fit of the decreasing portion of the curve in Fig. 3 intercepts the $m_r = 0$ axis at $T \approx 215$ K, although a closer look to the curve (inset Fig. 3) shows that

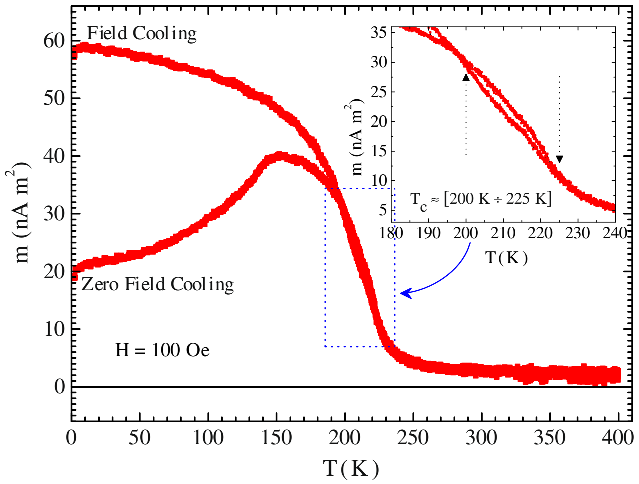

Fig. 1 ZFC and FC dc magnetization curves as a function of the temperature for the $Cu_{0.38}Ni_{0.62}$ with an applied field $H = 100$ Oe. The inset shows the local enlargement between 180 and 240 K

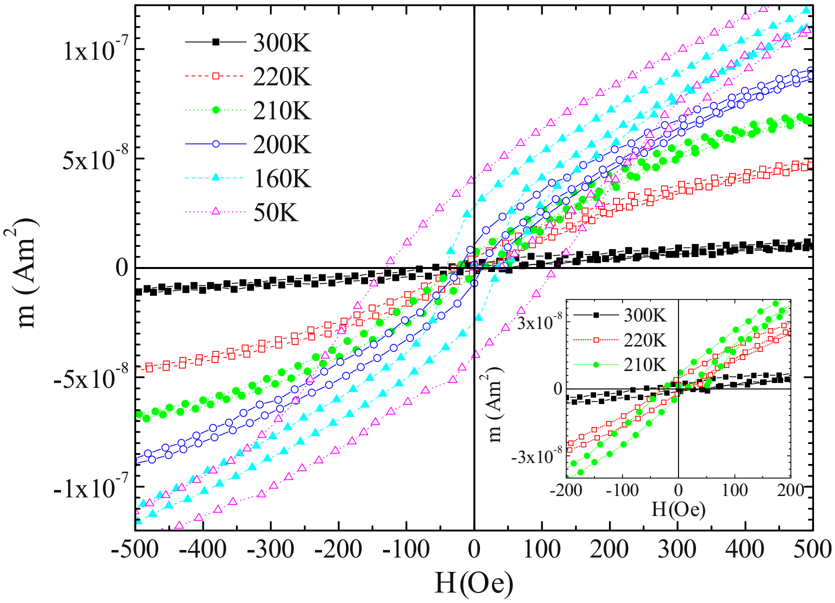

Fig. 2 Magnetic moment (m) as a function of the magnetic field (H) for the $Cu_{0.38}Ni_{0.62}$, measured at temperatures $T =$ 50, 160, 200, 210, 220, and 300 K. Inset: a magnification near zero field for the $m(H)$ at $T =$ 210, 220, and 300 K is shown. In particular, the curve at 300 K still appears to behave ferromagnetically, contrary to what is suggested by the curve in Fig. 1 at the same temperature, due to the presence of a non-zero remanent magnetic moment

the lowest temperature corresponding to $m_r \approx 0$ is $T_0 \approx 210$ K. Since the noise in the measurement does not allows us to univocally identify T_0 as the temperature where effectively the remanent magnetization disappears, with this method we can only estimate the critical temperature as $T_c = 215 \pm 5$ K. Moreover, it is worth underlining that the remanent magnetic moment at $T = 300$ K is null, as expected for a paramagnetic state and in contrast with the presence of the hysteresis in the $m(H)$ loop at the same temperature reported in the inset of Fig. 2.

An alternative way to determine univocally and in a physically transparent way the T_c in a weak ferromagnet is the use of the Arrott plots [31], which allows also to overcome some of the problems related to the influence of the noise in the measurements. Although according to (5) the plot of m^2 versus H/m should represent a series of straight lines, very often the obtained curves do not behave linearly, so making the analysis of the curves controversial. In these cases, the critical temperatures are often obtained by considering the high-field extrapolation of the $m^2(H/m)$ curves. These extrapolations effectively behave as straight lines with a given slope, which is almost independent of the temperature at which the measurement is taken. As shown in Fig. 4, if the straight line with this slope is forced to pass by the origin of the axis, it identifies the temperature corresponding

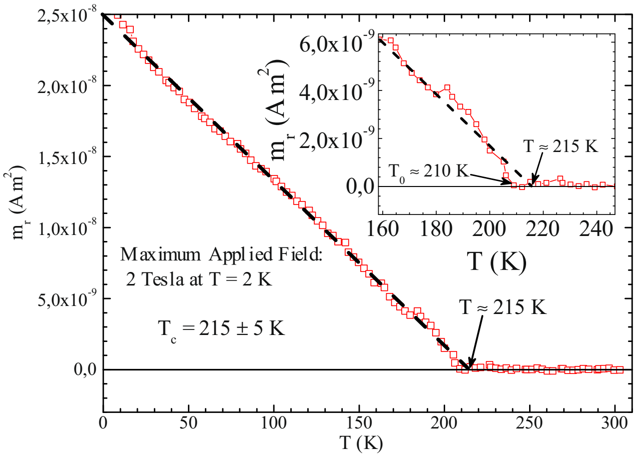

Fig. 3 Temperature dependence of the remanent magnetization produced in the sample at $T = 2$ K by applying a magnetic field from 0 to 2 T and back to 0 T again. In the inset, the region near $m_r = 0$ is magnified

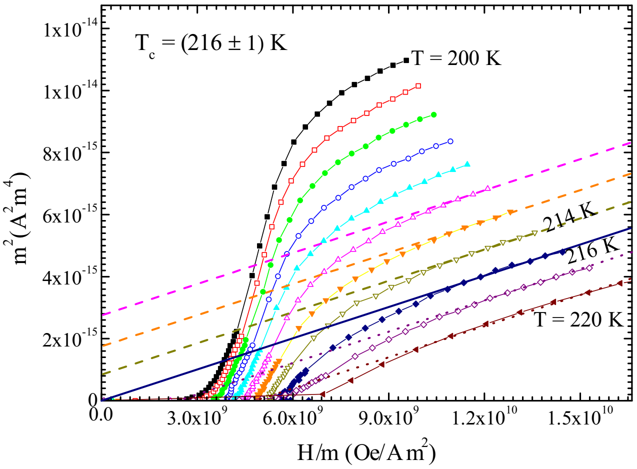

Fig. 4 Arrott plots m^2 versus H/m made with the $m(H)$ curves measured in the temperature range of 200–220 K with a step of 2 K. The solid straight line passing by the axis origin and having the same slope as the linear extrapolation of the curves at high H/m values is reported, so individuating the value of $T_c = 216 \pm 1$ K. The additional lines parallel to the one passing by T_c are also drawn for $T < T_c$ (*dashed lines*) and for $T > T_c$ (*dotted lines*)

to the critical temperature. In the case of Fig. 4, T_c is found from the solid straight line laying on the m^2 versus H/m curve taken at $T = 216$ K, and therefore, it indicates that the critical temperature is $T_c = 216 \pm 1$ K.

As we have shown, the determination of the T_c from the Arrott plots requires a linear extrapolation which can be realized only if the magnetic field is high enough to establish a clearly linear behavior in the m^2 versus H/m curves. On the contrary, as discussed in the first section, the Arrott criterion requires a low magnetic moment, in order to be considered appropriate for the analysis of the curves. For this reason, the procedure used to determine the T_c from the curves in Fig. 4, which is still valid for weak ferromagnets and is often used also for not-weak ferromagnets, cannot be considered totally correct regardless.

However, a further characteristic of the m^2 versus H/m curves in the Arrott approach is that their curvature is different for curves taken above and below T_c. In particular, the curves are concave for $T < T_c$ and convex for $T > T_c$ [31]. This behavior is visible at low fields, in particular lower than the field needed to establish a linear behavior in the curves, and it is due to the fact that all curves must tend to go to zero for $H = 0$ without crossing the straight line corresponding to T_c. Looking at our plots of Fig. 4, this characteristic is not evident even at the lowest temperatures considered, because the curves tend to go to zero even at $H \neq 0$ and the values of the data, although at intermediate H/m and in particular at $T > T_c$, are too small to clearly distinguish the signal from the noise. In order to highlight the change in the curvature in the Arrott plots, their first-order derivatives have been numerically performed. These derivatives are reported in Fig. 5. In particular, in the main panel of Fig. 5, the derivative curves show a very clear peak at the lowest temperatures, in correspondence to $(H/m) \approx 5 \times 10^9$ Oe/Am^2, indicating a change in the curvature of the experimental Arrott plots. This peak decreases for increasing temperatures, and as expected at $T = T_c$, it disappears in the noise for the curve at $T = 216$ K, being still present in the curve at $T = 214$ K, as reported in the inset of Fig. 5. In this way, a more precise and correct value of the T_c of a weak ferromagnetic material has been determined without forcing the application of the Arrott plot to consider the portion of the curves at high fields, but only analyzing the low field part of the curves.

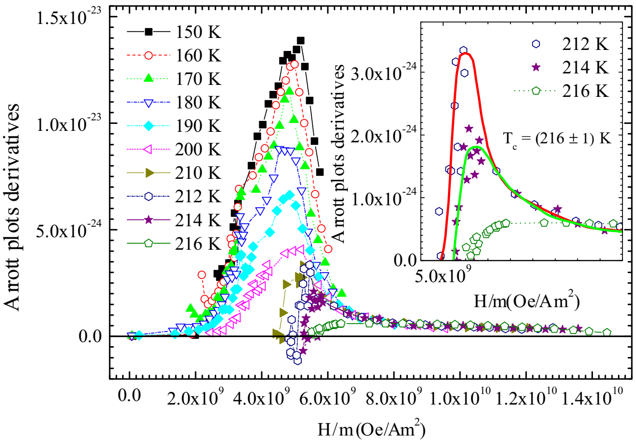

Fig. 5 The behavior of the derivatives of the Arrott plot, i.e., of m^2 with respect to H/m, is reported at increasing temperatures, suggesting as the value of T_c, the temperature corresponding to the first curve where the peak in the derivative disappears. In the inset, the details of the curves at 212, 214, and 216 K are reported, allowing to individuate the value of $T_c = 216 \pm 1$ K. The red and green solid lines are guides for the eye

4 Conclusions

In this work, we have analyzed different techniques used to determine the ferromagnetic transition temperature (T_c) of a weak ferromagnetic thin film Cu_xNi_{1-x}, and we have underlined the effect of noise and of the measurement parameters on the accuracy and correctness of the transition temperature value. In particular, we have focused the attention on the Arrott plots of the isotherms of m^2 as a function of H/m, where a linear extrapolation of the high-field portion of the curves is generally used to define which curve corresponds to T_c, so giving no importance to one of the main assumptions of the Arrott technique which requires small values of m. This condition can be considered valid for weak ferromagnets but can be fulfilled for strong magnetic materials only at low fields. In order to solve this contradiction, the Arrott plot approach for the determination of T_c has been slightly modified by considering the derivatives of the curves of m^2 as a function of H/m, due to the observation that these curves have to show a change in the curvature in the region of low H/m when the temperature crosses T_c. The results obtained by using this modified Arrott plot method appear to be in agreement with those obtained by other widely used magnetic characterization techniques, but their higher reliability and precision can be ensured by avoiding the most relevant problems and weak points encountered with the other alternative approaches. The condition for the application of this method, namely the analysis of the region at low H/m, suggests that it could be extended to characterize, in particular, strong ferromagnetic materials in the region of low magnetic fields.

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