

On the Curie temperature determination of nickel-zinc ferrites

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In this work, we carried out the determination of the Curie temperature of nickel-zinc ferrites. We conducted a thorough review of the available methods for its determination. From the methods considered, we selected the determination based on inflection points and on the intersection of two tangents. The resulting comparison of the obtained results showed that the latter method generally leads to higher values, differing by several degrees Celsius.

Keywords: nickel-zinc ferrites, Curie temperature

1 Introduction

Nowadays, the increasing demand for a higher standard of living for an ever-growing number of people necessitates development in many areas of human activity. This trend is accompanied by technological progress and the need for new, more powerful technologies and effective materials that find applications across various industries. Among these materials are magnetic materials which are becoming increasingly integrated into our daily lives. From this perspective, ferrites with a cubic spinel structure are highly effective materials for a wide range of industrial and technological applications.

Their unique and versatile properties – such as high resistivity, higher saturation magnetization and permeability, low dielectric loss, and minimal eddy currents – enable their potential use in electronics, chemical engineering, environmental, and automotive industries [1]. Due to their soft magnetic properties [2, 3], Ni-Zn spinel ferrites are used as multifunctional materials in various applications, including microwave electronic devices [4], magnetic recording media [5], magneto-optical devices [6], gas sensors [7], humidity sensors [8], and lithium-ion batteries [9]. Nanosized ferrites also find applications in medicine, serving as contrast agents in magnetic resonance imaging (MRI), in targeted drug delivery, and in magnetic hyperthermia [10, 11].

Spinel ferrites with the formula MFe_2O_4 , where M is typically a divalent metal ion (such as Ni^{2+} , Zn^{2+} , Mn^{2+} , Co^{2+}), are characterized by a face-centered cubic lattice structure in which the cations are distributed across

tetrahedral (A) and octahedral (B) sublattices. The differing nature and ionic radii of the constituent elements lead to preferential occupancy of these sites, which significantly influences the magnetic properties of the ferrites [12]. Substituting even small amounts of metal ions with rare earth ions RE^{3+} can alter the structure and enhance the magnetic, electrical, dielectric, and optical properties of ferrite materials. This change may be due to the presence of 4f-3d bonds formed by rare earth ions in the octahedral (B) sites which notably affect the magnetocrystalline anisotropy in spinel ferrites. Since RE^{3+} ions replacing Fe^{3+} ions have larger ionic radii, the substitution causes internal stresses and deformation within the crystal lattice. As a result, only a limited amount of RE^{3+} ions can be incorporated into the lattice. Beyond this solubility limit, the excess RE^{3+} ions tend to accumulate at grain boundaries, inhibiting grain growth [13].

In our previous studies, we examined the influence of substituting different types and amounts of rare earth ions on the structural, electromagnetic, and microwave properties of Ni-Zn ferrites [14, 15]. Based on this knowledge, even a small amount of RE ion substitution can induce changes in the structure and magnetic behavior of spinel ferrites. One particularly sensitive parameter affected by these structural modifications is the Curie temperature T_C , which is also known as the transition temperature at which the ferromagnetic materials or ferrimagnetic materials (ferrites) subject a transition from an ordered magnetic phase ($T < T_C$) to a disordered paramagnetic phase ($T > T_C$). T_C is a peerless property of each ferromagnet (or ferrite) that yields a second-order phase transition in a zero magnetic field

and depends on the material itself only. The value of this critical temperature needs to be known so that in practical use of material there is no loss of magnetization at temperatures exceeding the T_C .

2 Related works

Determining the T_C is not unambiguous, different procedures are used, which may not give the same result. When plotting graphical dependencies, there are also several methods, where the temperature is on the horizontal axis, and the dependent variable on the vertical axis (such as magnetic susceptibility χ , saturation magnetization M_s , magnetic permeability μ or another measured physical quantity) [16]. Absolute but also relative values of the dependent variable are used. However, it is certain that the T_C is in the temperature range where a sharp change in the characteristic occurs. This sharp change is represented by at least one order of magnitude decrease in the dependent variable. It should be noted that the mentioned concepts of T_C determination may also be utilized in ferroelectric materials (or multiferroic with ferroelectric parts) only the dependent variable is different (e.g., dielectric susceptibility χ_e , saturation polarization P_s , dielectric permittivity ϵ , etc.) [17, 18].

In the following description, the criterion for determining the T_C will be a physical phenomenon, a measurement method of the temperature dependence of the monitored parameter, and the subsequent numerical method of determination.

2.1 Determination of T_C by means of galvanomagnetic effects [19, 24]

- a) Determination of the T_C from the maximum of the temperature coefficient of electrical resistance $(1/R_0)(dR/dT)$.
- b) Determination of the T_C from the extreme of the negative galvanomagnetic effect (caused by the para-process) $-(\Delta R/R)_n$.

In this case the critical temperature of a material is determined by identifying the temperature at which the electrical resistance drops to zero, which occurs at the maximum of the temperature coefficient of resistance, where the resistance-temperature curve shows a steep decline. This approach is especially suitable for magnetic materials exhibiting also semiconducting properties (according to the value of electrical conductivity). Note that when the galvanomagnetic effects are measured, the extremum values of these effects occur only if spontaneous magnetization is large enough.

2.2 Determination of the T_C by Mössbauer spectroscopy and neutron scattering [19]

Mössbauer spectroscopy and neutron scattering belong to markedly more accurate ways how to determine T_C . In the case of the Mössbauer method, it is known that at T_C a hyperfine magnetic nuclear field disappears. In the case of neutron scattering, magnetic scattering iso-thermal experiments enable monitoring of the break-down of the long-range magnetic arrangement at T_C . However, none of the two approaches mentioned is used to determine T_C routinely because of the requirements upon the measurement apparatus and probably also because of a considerably high cost of such examinations.

2.3 Determination of T_C from temperature dependence of selected magnetic parameters [19]

- a) Determination of the T_C from the fall of the temperature dependence of saturation magnetization $M_s(T)$, or from the minimum of dM_s/dT .

In the past, some scientists proposed a method leading to finding precise values of T_C from the measurements of magnetization. This method is also known as the Arrott technique [20, 21] and it is very convenient to compute T_C because it utilizes experimental data for supporting a fit based on the thermodynamic theory. The technique utilizes Landau's expansion of the thermodynamic potential V into a series of magnetization powers with convenient parameters

$$V = \Phi_0 + \frac{1}{2}aM^2 + \frac{1}{4}bM^4 + \dots - HM \quad (1)$$

In the case of equilibrium condition $\partial\Phi/\partial t = 0$, and in terms of the reduced temperature $\tau = T/T_C$ and magnetization $\sigma = M/M_s$, Eqn. (1) is changed to $H = a\sigma + b\sigma^3 + \dots$. Parameters $a, b, \dots$ which are considered as functions of reduced temperature are expanded in the Taylor series about the T_C , i.e., about $\tau = 1$. Parameter a can be determined from isothermal field dependences of magnetization and since $a = 0$ at T_C , this can be used to find out the Curie point. The method outperforms the external aspects such as temperature and applied magnetic field effects. This method is convenient for applications where magnetocaloric properties of magnetic materials can be utilized.

It should be noted that the procedure of determining the spontaneous magnetization M_s by linear extrapolation of the $M(H)$ curves to zero field cannot be used near the T_C since in this region the $M(H)$ curves are extremely nonlinear and therefore determining T_C from the $M(T)$ dependence appears to be somewhat complicated in some cases.

- b) Determination of the T_C from the disappearance of initial magnetic permeability μ_i or susceptibility χ [19, 22, 23].

To our knowledge, determination of T_C from the initial permeability temperature dependence is the least accurate way due to the existence of such phenomenon as the paraprocess (even in low fields). In the vicinity of T_C , magnetostriction and magnetic anisotropy are poor, which may reduce displacements and rotations of spins.

- c) Some authors made efforts in order to find out the T_C by means of remanent magnetization M_r and the coercive force H_c temperature dependences [19]. However, the mentioned approaches fall to the ground as the value of any of these quantities may be nonzero even over T_C as a result of non-disappearance of spontaneous magnetization.

2.4 Determination of T_C from temperature dependence of other non-magnetic parameters [19, 25, 26]

- a) Determination of the T_C from the isothermal heat capacity measurements $C_{\text{heat}}(T)$ in zero and nonzero magnetic fields; the peak of the specific heat is observed at T_C [19]. Since the extreme of specific heat (or heat capacity) may be very wide, the finding out of T_C can also be hard using this approach.
- b) A technique of temperature gradient appreciation for rapid finding out the T_C based on thermal conductivity and diffusivity as functions of temperature [26]

The required temperature T_C is obtained by means of an inverse numerical method, giving the temperature dependent thermal diffusivity with another variation in slope at T_C . The gradient of the steady temperature profile adjusts according to the local thermal conductivity and changes suddenly at T_C . The high-resolution finding out of the temperature dependent thermal properties allows the identification of T_C by two independent evaluations during a single experimental run, solely relying on data of thermal conductivity and thermal diffusivity. The obtained T_C agrees well with values presented in the literature obtained using other methods.

- c) T_C may be determined precisely also by means of the magnetocaloric effect. In this case, T_C can be approximated to the temperature at which the change of magnetic entropy has its extreme value, T_{peak} . However, both T_C and T_{peak} do not necessarily coincide, and the deviation depends on the applied magnetic field [25].

2.5 Numerical techniques for T_C determination

- a) Determination of the T_C using inflection points [29]

The inflection point is the point at which the shape of the curve changes. The curve changes from a concave part to a convex part, or vice versa. So, using this method, the T_C corresponds to the point on the graph of the magnetic susceptibility versus temperature dependence, where the concave shape of the curve changes to a convex shape. To calculate the inflection points, it is necessary to construct a graph of the magnetic susceptibility versus temperature and find a suitable function of the graph (which sufficiently describes the measured course). The inflection point of the magnetic susceptibility versus temperature dependence curve is considered to be the T_C . However, it is necessary to add that several inflection points may appear on the curve over the entire temperature range. The increased number of inflection points may be caused by inaccuracies in the measurement, or by the approximation of the curve. The curve can be approximated by an n th degree polynomial. Only the inflection point that is located in the temperature interval of a sharp decrease in magnetic susceptibility is considered the T_C .

- b) Determining the T_C using the intersection of two tangents [27]

In this procedure, we will use a part of the graph of the dependence of magnetic susceptibility on temperature, and we are only interested in the part of the graph that is located around the sharp decrease in magnetic susceptibility. We will use this part to determine the T_C . This procedure uses two tangents to the graph. The first tangent is located in the temperature region just below the T_C . In this temperature region near the T_C , we observe a steep linear decrease in magnetic susceptibility with temperature. The second tangent is located in the temperature region just above the T_C . In this region, we can also observe a slight linear decrease in magnetic susceptibility with increasing temperature. These two regions are connected by the so-called knee of the curve. We consider the T_C to be the intersection of these two tangents, which is located in the lower knee of the characteristic.

- c) Determining the T_C using the maximum flexure of the curve [28]

In this approach, it is necessary to fit the measured data to a function (for example, a fourth-order polynomial) that describes the given course of magnetic susceptibility versus temperature with sufficient accuracy, so we need to construct a graph of the temperature dependence of magnetic susceptibility (similar to the inflection point method). Then, we look for the maximum curvature of the given function, which we consider to be the desired T_C . To determine this maximum curvature, it is necessary to calculate the first derivative of the function, and then the second derivative of the function. It is the second derivative that will help us determine the maximum curvature of the given function, and therefore the T_C of the given sample. If we create a graph where the second derivative of magnetic susceptibility (or magnetization) is on the y-axis and the temperature of the material is on the x-axis, then the peak of this curve indicates the T_C .

3 Results and discussion

3.1 Determination of T_C by means of inflection points

To determine the T_C using inflection points, we used data from our previous study [14]. In that study, we analyzed the effect of changing the stoichiometry of Ni-Zn ferrites through partial substitution of Fe^{3+} ions with Eu^{3+} , Gd^{3+} and La^{3+} ions on selected structural and magnetic properties, with a total sample size of fifteen. For each ferrite sample, we constructed a graph of the temperature dependence of magnetic susceptibility $\chi(T)$. We extracted the part of the graph where a sharp change (decrease) in magnetic susceptibility occurs, since the T_C is located in this region of the graph (see example in Fig. 1). By applying a suitable approximation – a fourth-degree polynomial, which sufficiently accurately describes the measured dependence – we determined the inflection points. From the resulting inflection points, we selected the one located in the steep decrease of the curve. Each sample has exactly one second-order phase transition, and thus exactly one T_C .

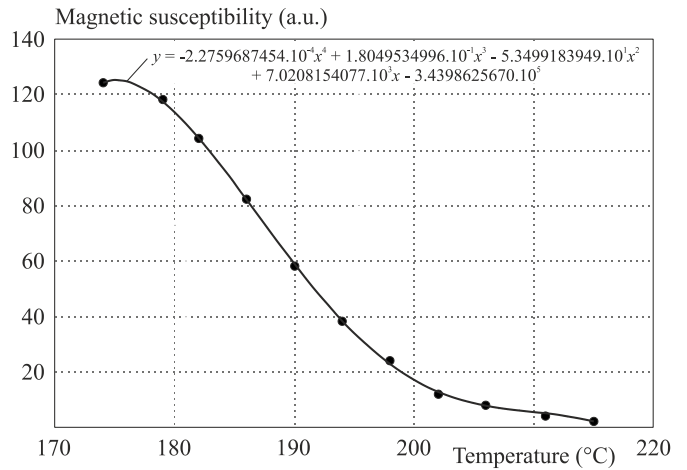


Fig. 1. Section of the magnetic susceptibility temperature dependence

3.2 Determination of the T_C by means of the intersection of two tangents

To determine the T_C using the intersection of two tangents, we employed the same data as in the method of inflection points. For each sample, we again constructed a $\chi(T)$. We extracted the part of the graph where a sharp change in magnetic susceptibility occurs (as in the use of inflection points), see example in Fig. 2. In this procedure, it is necessary to construct two tangents to the graph. One tangent is drawn in the temperature region below the T_C , and the other tangent is drawn in the temperature region above the T_C . The intersection of these tangents is considered to be the T_C .

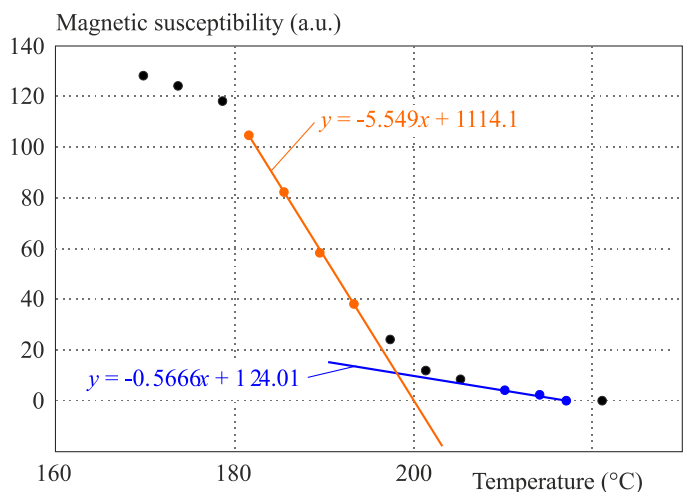


Fig. 2. Section of magnetic susceptibility temperature dependence with tangents indicated

3.3 Comparison of the obtained results

Table 1 presents the obtained T_C values for all samples. The T_C determined using the method based on the intersection of two tangents is higher by several degrees Celsius compared to the method employing inflection points. Nevertheless, both methods provide important information, namely that the T_C increases with the rising number of additives.

Table 1. T_C (°C) of $\text{Ni}_{0.42}\text{Zn}_{0.58}\text{Me}_x\text{Fe}_{2-x}\text{O}_4$ ferrites

x	inflection-point method			tangent-intersection method		
	Eu^{3+}	Gd^{3+}	La^{3+}	Eu^{3+}	Gd^{3+}	La^{3+}
0	186.28	186.28	186.82	198.72	198.72	198.72
0.01	191.56	199.66	194.17	204.87	206.31	200.85
0.02	197.26	209.61	195.48	211.13	216.45	200.82
0.04	209.84	216.24	197.05	219.91	225.48	201.82
0.06	219.40	226.85	200.16	229.26	234.80	206.23

4 Conclusion

In this work, we determined the Curie temperature of nickel-zinc ferrites. We carried out an extensive review of the methods used. From these, we selected the inflection-point method and the tangent-intersection method. Our results show that the tangent-intersection method consistently yields Curie temperature values higher by several degrees Celsius than those obtained by the inflection-point method. The extracted value of the Curie temperature therefore depends on the type of method chosen.

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