

THERMOMAGNETOMETRY

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Abstract

This brief tutorial and somewhat personal review discusses general experimental aspects and several examples of how thermomagnetometry has proven valuable in a variety of studies. The three areas of focus concern 1) determination of the magnetic transition temperature to characterize the material or to serve subsequently as a potential temperature standard for thermogravimetry; 2) detection of magnetic reactants, intermediates, or products during reactions and 3) following the reaction rates for such reactions. A broad range of materials is examined, metals, ceramics, catalysts, minerals, and inorganic complexes.

Keywords: magnetic transition temperature, temperature standard for TG, thermomagnetometry

Introduction

Thermomagnetometry, TM, is essentially thermogravimetry, TG, with a magnetic field gradient imposed upon the sample. The effect of the resulting attraction upon a ferro- or ferri-magnetic sample is a significant apparent increase or decrease in the mass of the sample, depending upon the direction of the magnetic field gradient. The much weaker effects involving antiferromagnetic, paramagnetic, or diamagnetic materials are certainly a major field of investigation, but not generally considered as TM in the thermoanalytical sense.

The topic has had several prior reviews that have focused on metals [1-3], minerals [4-5], thin films [6] or the general method [7]. Obviously, this technique is currently only applicable to inorganic materials. The field of organic magnets is of growing interest, however, and the use of polymer/magnetic material composites currently dominates the computer memory and recording tape markets. Hence the technique is of value to most materials scientists and engineers.

Apparatus for TM consists of a conventional thermobalance having sufficient sensitivity and a practical means of imposing a magnetic gradient on the sample position [8, 9]. Since the concern is with magnetically ordered materials, a strong magnetic field is not needed and actually may be undesirable for many applications [10]. Consequently a small permanent magnet is frequently adequate. Stronger permanent magnets or electromagnets can be used when required [11, 12]. The field gradient at the sample position may be measured but usually the specific value is irrelevant, provided that it is of adequate strength to detect the desired magnetic effects. There are times when the magnetic field produced by the furnace itself is adequate [13, 14].

The magnet is outside of the furnace so that it is unheated and maintains a constant field. Reproducible positions for the magnet and sample are very desirable. Confusion may result if the sample is free to rotate or move about, since the magnetic attraction will change due to simply geometric considerations rather than for the chemical or thermal reasons as presumed. It should also be kept in mind that the magnetic attraction will almost always decrease with increasing temperature. So as one approaches the T_c of the sample the effect will be weaker and consequently more difficult to detect.

Determination of T_c

Accurate measurement of the magnetic transition temperature, T_c , is important from several standpoints. Besides its significance in magnetic theory, it also has value for temperature calibration and for the identification and analysis of magnetic materials. Once the T_c has been accurately determined for a series of materials, then that information can be used to identify unknown samples or materials of known T_c and can be used for calibration of the temperature in the sample position.

The calibration of temperature is of particular interest in TG where intimate contact between the sample and the temperature sensor may be impractical due to experimental factors or because of chemical reactivity. The value for qualitative analysis arises from the relatively simple fast measurement and its ability to distinguish between various substituted spinels, garnets, etc. whose X-ray diffraction patterns can be very similar.

The accepted definition for T_c is the temperature at which the material becomes paramagnetic, i.e., the disappearance of the strong magnetic attraction. This translates to the return to the baseline and is conveniently measured as the extrapolated end point, see Fig. 1 [10]. It can be measured from either the TG curve directly or from the derivative (DTG) curve. The latter will be somewhat more sensitive and yield a slightly higher apparent T_c .

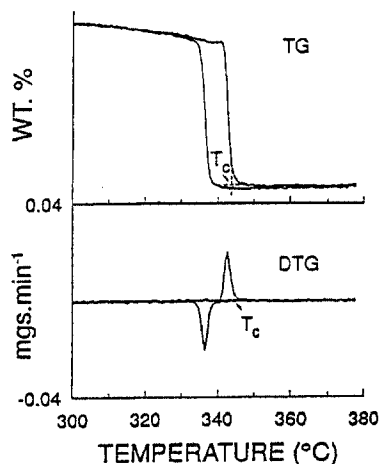


Fig. 1 TM and DTM curves for nickel heated at $10^{\circ}\text{C min}^{-1}$ in nitrogen [10]

The purpose for the calibration is to remove both the primary uncertainty in the correlation between the temperature of the sensor and sample and the secondary uncertainty in the calibration of the sensor itself. The former is related to the difference in the position of the sample and sensor and is exacerbated by the rate of heating. Varying the rate of heating and/or cooling followed by extrapolation to zero rate should yield a value similar to an isothermal situation as seen in Fig. 2 [10]. The difference between this temperature of convergence and the "true" temperature reflects the inaccuracy of the sensor.

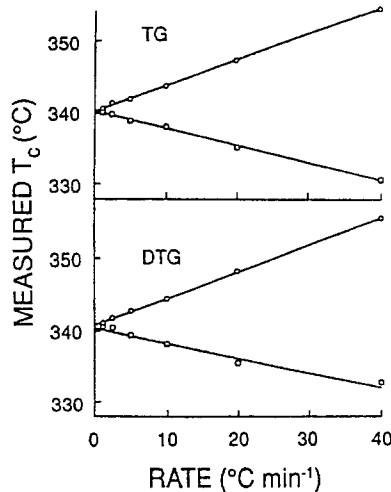


Fig. 2 The effect of heating and cooling rates on the measured T_c of nickel [10]

A comparison of temperature based on the TM method vs. the "fused link" approach suggests excellent agreement [10]. The TM method was first suggested by Norem *et al.* [15] and has the advantages of 1) suitability for cooling as well as heating, 2) generally allowing reuse of the standards, and 3) being more convenient for simultaneously running several different samples or repeating the experiment. This was immediately adapted for digital data acquisition and processing [16].

The Standardization Committee of ICTAC certified some magnetic materials for calibration by conducting a large international round robin, however, the results were not particularly encouraging because of the wide variability [17, 18]. The advent of simultaneous TG/DTA, however, has dramatically opened the way for tremendous improvements in both precision and accuracy [19–21]. It is possible now to use the very standards that define the International Temperature Scale of 1990 [22] for simultaneous calibration of the TM experiment.

Addition of selected pure metals, whose melting points bracket the T_c of the sample, to the sample pan enables correction of the observed value of T_c based on the observed and true melting temperatures. These corrected values of T_c show greatly enhanced precision and the accuracy is traceable directly to the standards which define the temperature scale. Alloys in the systems Ni-Co and Ni with a non magnetic metal such as Cu, Pd, or Zn should provide standards for values of T_c be-

low about 1400 K. Such standards then could be supplied and used for the calibration of traditional TG instruments with confidence. These simultaneous TM/DTA techniques should also be the most accurate (but not necessarily the most precise) means of determining T_c for any new materials.

Many of the earliest applications concentrated on understanding alloys. Very nice analytical methods were developed based on correlating the value of T_c with the concentration of an alloying species. Cemented carbide materials are widely used and TM has been successfully employed to determine the dissolved carbide content in the metallic cobalt phase by the depression of T_c [23, 24]. Similarly, alloys of nickel with titanium and/or carbon have been analyzed using the relationships between saturation magnetization, T_c , and concentrations of titanium and/or carbon [25].

Detection of magnetic intermediates or products

This section focuses on the overall nature, stoichiometry and mechanism of a reaction, while the following section concerns the rate of a reaction.

Metals

Phase identification and formation are the main advantages offered by TM in this section. The evolution of phase development during the heat treatment of steels and the austenite martensite type transformations has been a topic of study [26, 27]. Grain growth and nucleation processes that are critical to the application of "met-glasses" are amenable to study by TM [28, 29]. The effects of mechanical alloying were studied by TM in the copper-iron system [30].

The thermal decomposition of oxalates has always been an intriguing topic in thermal analysis. The contrasting behavior towards oxidation and reduction behavior in these materials has been especially interesting. Many transition metal oxalates will decompose directly in an inert atmosphere to yield metals through the evolution of CO_2 while others form the oxides by the release of both CO and CO_2 . Among those forming the metal under inert conditions are cobalt and nickel. These elements also form solid solutions of the oxalate dihydrates, $\text{MC}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$. Do these solid solutions decompose directly to yield a homogeneous alloy or do they yield mixtures of the individual metals?

The X-ray diffraction patterns of the two materials are so similar that it is very difficult to distinguish them apart by this means. The metals, like the oxalates form a complete range of solid solution and the T_c varies smoothly from about 1130°C for cobalt to 358°C for nickel. Hence, TM is a convenient technique to answer the question above. The TM results clearly supported the direct formation of the alloys during the thermal decomposition in an inert atmosphere [31]. The reactions of copper with iron, nickel and cobalt have also been carefully studied, however, the formation of metals is incomplete for these systems [32].

The study of Chromindur (Cr10.5-Fe28-Co61.5) proved particularly informative for the range of the immiscibility gap in the phase diagram [33]. Samples quenched from the single phase regime into the two-phase area retained a single

value of T_c that was indicative of the overall composition. When annealed, however, growth of a phase having a higher T_c appeared indicative of a lower Cr concentration. The absence of a peak associated with the Cr rich phase was initially puzzling. The spinodal microstructure that forms, however, has the two phases in thin alternating layers. The intense internal magnetic field of the higher T_c phase is sufficient to clamp the lower T_c phase in the magnetically aligned structure, so that it behaved magnetically as its lower Cr neighbor.

Reactions with other material have also proved interesting. TM has been valuable in the detecting the formation of carbide or nitride phases in carbonyl iron [34]. Decomposition of supported nickel salts to form supported Ni catalysts has recently been investigated by TM [35]. Not only can one follow the formation of the metal, but also reactions with the underlying alumina or silica support.

The final example of the use of TM in this section is for studies on the recovery of heavy metals from tailings. Mikhail *et al.* [36] have indicated that such valuable materials as titanium and zirconium can be recovered by oxidation of the tailings to form magnetic oxides of iron with the desired elements. These can be separated magnetically and the oxides of Ti and Zr recovered through subsequent processing.

Geosciences

The proximate analysis of coal has been a subject of great interest. One frequent component in coals that presents problems is iron pyrite. The major problem concerns the emissions of SO_2 that result during combustion. Consequently the ability to evaluate the pyrite content along with the other major components of the coal is valuable. Efficient accurate schemes have been developed using TG with controlled atmospheres to evaluate the moisture, volatiles, carbon, and total ash, see for example the reviews by Warne [4, 5]. The iron oxide resulting from the combustion process is an unknown part of the total ash content. Several investigators, e.g., Aylmer and Rowe [37], have utilized a subsequent TM step in which the Fe_2O_3 in the ash is reduced by hydrogen to form the metal as shown in Fig. 3. The apparent increase

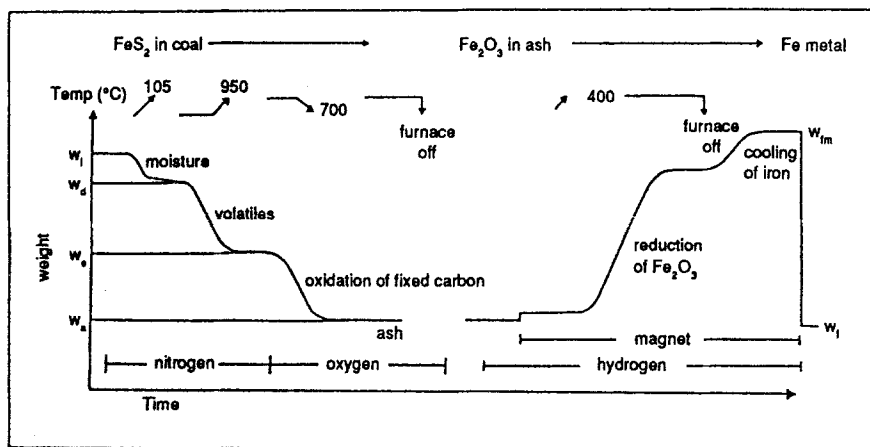


Fig. 3 Proximate analysis of coal by TG and TM in controlled atmospheres [37]

in mass during the TM step is related to the iron content of the ash and hence the starting coal. Comparison of the relatively fast and convenient TG/TM method with the ASTM standard at that time was very favorable.

The decomposition of the mineral siderite, FeCO_3 , serves as a nice example of the formation of magnetic intermediates during the thermal decomposition of materials [38, 39]. The iron is initially present as Fe^{2+} , but after decomposition it appears as Fe^{3+} . The question was does the mixed valent oxide magnetite, Fe_3O_4 , form as an intermediate? In an oxidizing atmosphere no apparent gain in mass occurs as a result of a magnetic field gradient. For a relatively pure sample in an inert atmosphere, however, there is a very large gain in apparent mass associated with the formation of the magnetic intermediate. The attraction disappears at about 585°C , the T_c for magnetite.

If the starting mineral is quite impure (the usual impurities are magnesium and manganese), then there was no gain in mass evident during decomposition in an inert atmosphere. The spinel phase, magnetite is still formed, however, Mg^{2+} and Mn^{2+} have now partially substituted for some of the Fe^{2+} and markedly reduced T_c so that it is below the temperature for the thermal decomposition. Hence, the material is paramagnetic by the formation temperature and does not give rise to the magnetic attraction until during cooling or on subsequent heating. The high degree of chemical reactivity during a thermal decomposition is evident from both the total incorporation of these impurity ions into the spinel phase during that initial heating in this instance and in the alloy formation described earlier for solid solutions of oxalates.

Ceramics

The examples above of solid state reactivity lead nicely into the following topic concerning the formation of ferrites by direct reaction of the appropriate oxides or the thermal decomposition of suitable precursors. In such instances, the formation of the final ordered, homogeneous, magnetic oxide is the goal and its achievement is indicated by a sharp magnetic transition having the proper value of T_c . These TM curves offer supplementary information to X-ray diffraction which has difficulty distinguishing these subtle substitutions in the spinel structure. Examples of such studies are the formation of nickel ferrite from citrate precursors [40], the synthesis of the nickel cobalt analog from various salts [41], and the formation of magnesium ferrites from solid solutions of the oxalates [42].

The formation of magnetic oxides having structures other than spinels, e.g., garnets ($\text{M}_3\text{Fe}_5\text{O}_{12}$) and the hexagonal magnetoplumbite ($\text{MFe}_{12}\text{O}_{19}$), have also been studied using TM [14]. This work of Moskalewicz offers the opportunity to distinguish between "hard" and "soft" ferrites. These are terms used to describe the relative ease of domain wall motion. A soft ferrite is one in which the magnetic domains can rearrange readily and thus conform to the current conditions. The domain walls in hard ferrites, in contrast, are more resistant to motion and therefore, are better suited for permanent magnet applications. This increasing resistance to domain reversal is indicated by an increase in the field (coercive force) necessary to accomplish zero magnetic induction.

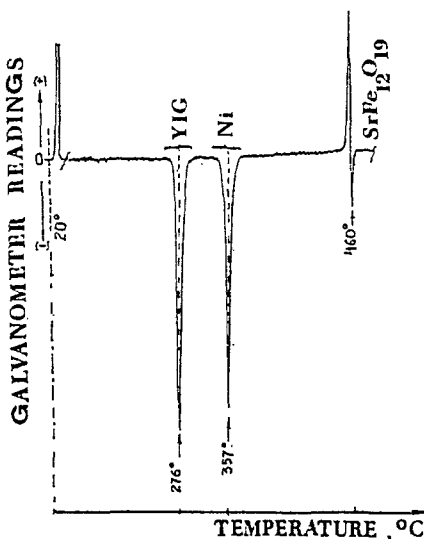


Fig. 4 TM curves for soft magnetic materials (Yttrium iron garnet and nickel metal) and a hard magnetic material $\text{SrFe}_{12}\text{O}_{19}$ [14]

Figure 4 shows a DTM curve for a garnet and a metal having a relatively low coercive force (soft) and a hard magnetic material, $\text{SrFe}_{12}\text{O}_{19}$. The soft materials show curves similar to those shown in previous figures. The hard material, however, exhibits an increase in the magnetic attraction approaching the transition and then the drop in apparent mass associated with T_c . Since the mobility of domain wall motion increases with temperature it is not surprising that at some point the energy associated with kT would allow the domains to begin to move and align with the magnetic field gradient. This would depend upon the microstructure of the material, the strength of the field gradient, etc. As further alignment of the domains occurs the apparent mass will increase until the value of T_c is closely approached, at which point normal behavior takes over as shown in Fig. 4.

Determining the rates of chemical reactions

Using TM to follow the relative concentration of magnetic species as a function of time and/or temperature provides the opportunity to study rates under unusual conditions. Two examples are selected to demonstrate the unique possibilities. In contrast to most of the previous examples, it is advisable to have a relatively large magnetic field gradient in order to maximize the apparent weight change and thus the ability to determine the fraction reacted, α . This raises the question of the potential effects of magnetic fields on the rates of chemical reactions. A very brief discussion of this is added.

Charles and his colleagues have performed a number of very interesting studies on corrosion in aqueous and liquid sodium media [3]. Figure 5 is a schematic of his apparatus. A small non-inductively wound furnace fits between the pole faces of an

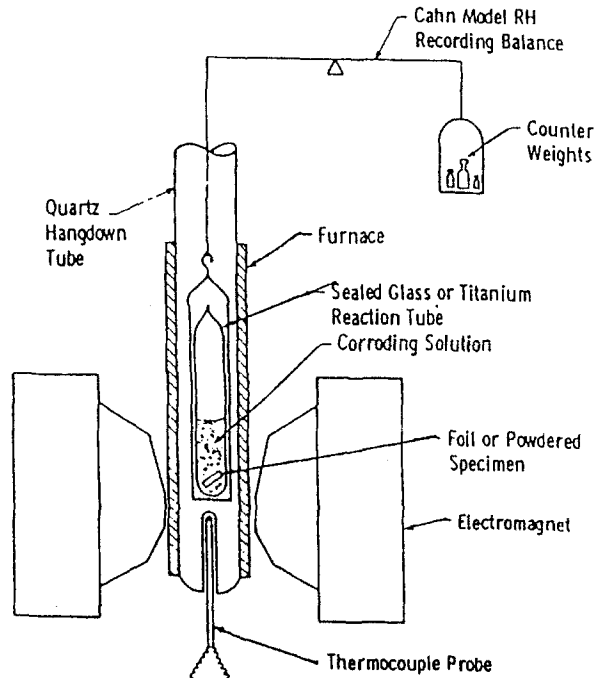


Fig. 5 Schematic of a TM apparatus for work with sealed tubes in a high magnetic field gradient [3]

electromagnet. These pole faces are shaped so as to provide a vertical homogeneous field over as wide a range as possible. The strong fields limit the amount of magnetic material in order to prevent the sample container from swinging sideways. The clever feature is that closed systems using a non-magnetic container, e.g., fused quartz, for aqueous media or titanium for studies in liquid sodium, are possible. Even though there is no change in real mass under sealed conditions, there is a change in apparent mass as the magnetic material is consumed.

Numerous studies were made in aqueous media on the effects of *pH*, chelating agents, inhibitors, etc. on the rates of corrosion of steels and spinel ferrites. A wide range of studies were made using liquid sodium as a corrosive (strong reducing agent) media or as a solvent for the reaction of oxides, carbides, etc.

Another area where the amplification possible through TM is important concerns thin films. Following the kinetics and mechanism of reactions involving thin films is complicated by the general presence of a supporting substrate [6, 43]. The presence of this overwhelming amount of "inert" material greatly reduces the sensitivity of most thermoanalytical techniques, except for TM as also seen in the previous example. There is the added complication of the potential reactions with the support as mentioned earlier for the supported catalyst.

Thin films of cobalt on alumina and silicon substrates were studied using a TM approach [12]. Both the oxidation in air and the subsequent reduction in hydrogen could be easily followed by the demise of the magnetic attraction as the cobalt was

consumed or the increase in attraction as the metal was reformed via reduction of the oxide. One must keep in mind, however, that the resulting kinetics for the oxidation are those due to the consumption of cobalt and not necessarily that of the formation of the final oxide. The analogous comments apply to the reduction reaction where the formation of cobalt is being measured and not the disappearance of the initial oxide. In addition there may be changing effects when the islands of cobalt remaining or forming are so small that they become superparamagnetic and lose their attraction. Obviously, as the temperatures approach the value of T_c , the sensitivity is markedly diminished and precise control of temperature becomes critical.

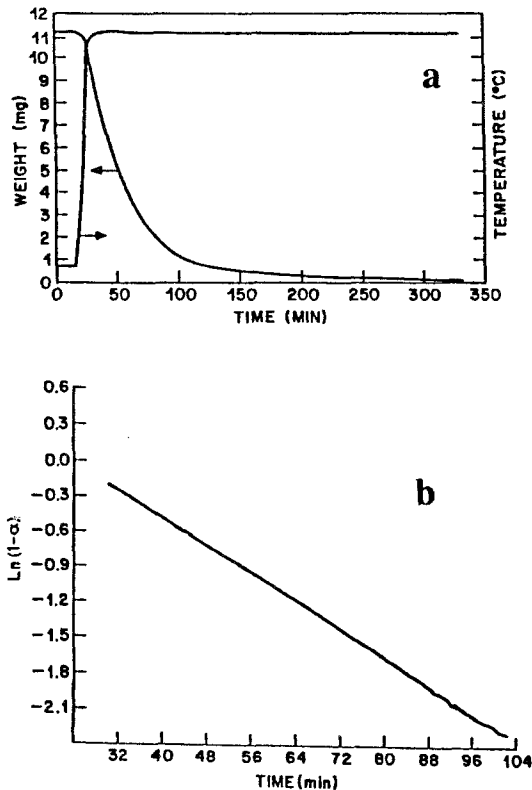


Fig. 6 Isothermal, 278°C, kinetic plots for a 900 Å film of cobalt on a lightly oxidized silicon substrate [13]

In spite of these caveats, it is gratifying to see how effective TM can be. Figure 6 shows results for the isothermal oxidation of a 900 Å film of cobalt on oxidized silicon in a field of 9000 Oe with a gradient of 2300 Oe cm^{-1} . The apparent gain in mass is over 11 mg in Fig. 6a. If one has a sensitivity of 1 μg , then it is possible to obtain meaningful kinetic data from much thinner films, perhaps by a factor of 100. The kinetic rate law appears to be a simple logarithmic function as seen in Fig. 6b, where $\ln(1-\alpha) = -0.0294t + 0.6933$. It is also possible to measure the reaction of the

cobalt with the underlying silicon substrate in the absence of the silicon oxide barrier layer. Such a reaction to form cobalt silicide has no change in mass associated with the reaction, only the apparent change due to the conversion of a magnetic phase into a paramagnetic phase.

Working in these higher fields raises concerns of the effect of high magnetic fields on the rates of chemical reactions involving magnetic materials. This is a highly controversial topic, see for example Ref. [44] and references therein. Some dependence of apparent T_c on the external magnetic field strength has been observed, but this is believed to be a function of the measurement and can be corrected by extrapolating to zero field [10].

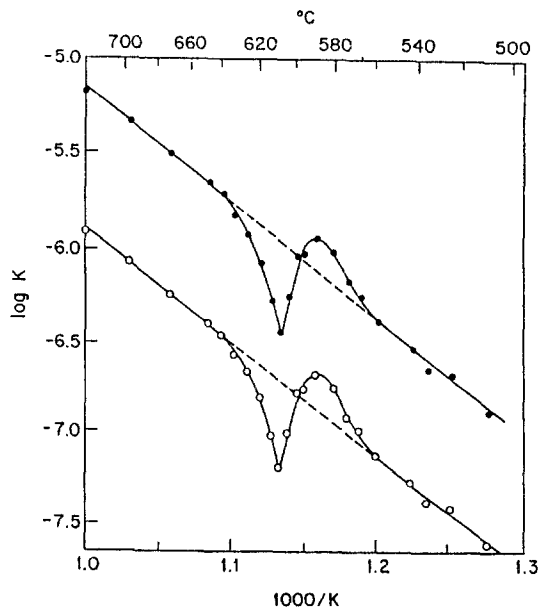


Fig. 7 Arrhenius plots for the oxidation of magnetite in oxygen [45]. Open points are for the Jander kinetic equation and closed points are for the contracting sphere kinetic equation

The presence of high internal fields, however, is another matter. Earlier the clamping effect observed in the spinodal decomposition of the chromindur alloy was shown to completely mask the intrinsic magnetic of the chromium rich phase. Perhaps the most intriguing and direct observation of such a phenomena is shown in Fig. 7 which displays Arrhenius plots for the oxidation of magnetite to hematite, $\alpha\text{-Fe}_2\text{O}_3$, by oxygen [45]. There is a striking anomaly in the rate constant within the narrow range of temperature surrounding the value T_c for magnetite and $\gamma\text{-Fe}_2\text{O}_3$, a metastable intermediate product. Clearly there is a pronounced effect on the rate of the reaction associated with the magnetic transition. Many questions remain unanswered in that system.

Conclusions

The potential value of TM is enormous under the proper circumstances. These circumstances, of course, include the requirement that magnetic phases are somehow involved in the system or reactions of interest. Simultaneous TG/DTA now allows for the very accurate determination of T_c by TM and the subsequent use of these values for calibration of temperature or the qualitative analysis of phases that may be difficult to determine otherwise. Reactions that involve no real changes in mass, e.g., within sealed tubes or of a thin film or catalyst with its support may be investigated. Using high fields provides the opportunity to study thin films with a sensitivity not possible by conventional TG. The ill-defined effects of high magnetic fields on the rates and mechanisms of reactions, however, must be kept in mind. The TM is under utilized. Hopefully this tutorial will make you more alert and better prepared to take advantage of the technique

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The presentation of the GEFTA-Netzsch Award. The recipient was Professor Patrick K. Gallagher. The award was presented by the President of Netzsch Gerätebau GmbH., Dr. W.-D. Emmerich. Congratulations!