TA Instruments

Thermal Analysis & Rheology

THERMAL SOLUTIONS

INVESTIGATION OF THE CURIE POINT BY MDSC™

PROBLEM

The Curie transition is the point at which a material (usually a pure metal or alloy) loses ferromagnetic character and becomes paramagnetic. This phenomenon can be monitored using thermogravimetric analysis (TGA) as an apparent weight change created by the change in attraction to a magnetic field. In fact, the Curie transition is often used to temperature calibrate TGA (1). However, the use of TGA is cumbersome and temperature precision is not always optimum.

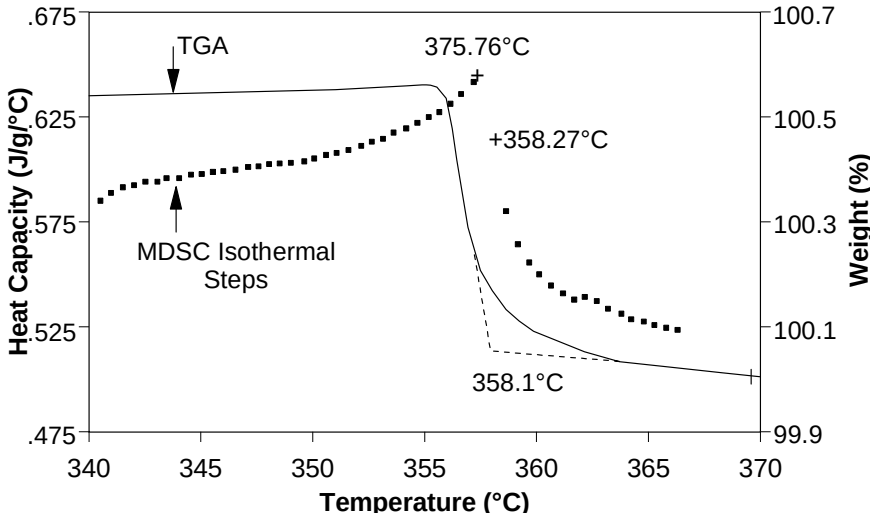
Since the Curie transition is accompanied by a change in heat capacity, it should also be detectable by DSC. However, conventional DSC cannot readily detect the Curie transition because the associated heat capacity change is very small (roughly 0.1 J/g/°C) and can be lost in baseline curvature and related DSC cell effects.

SOLUTION

Modulated DSC™ is a technique which subjects a material to a linear heating method which has a superimposed sinusoidal temperature oscillation (modulation) resulting in a cyclic heating profile. Deconvolution of the resultant heat flow profile during this cyclic heating provides not only the "total" heat flow obtained from conventional DSC, but also separates that "total" heat flow into its heat capacity-related (reversing) and kinetic (nonreversing) components. In the case of the Curie transition, modulated DSC separates the heat capacity change from nonreversing phenomena like baseline curvature. As a result, the sensitivity for detecting the Curie transition is greatly enhanced.

Figure 1 shows the comparative results obtained for pure nickel using modulated DSC and TGA. The agreement between the commonly used TGA inflection point (358.1°C)

Figure 1. DETERMINATION OF NICKEL CURIE TRANSITION (TGA and MDSC™ Analysis)



and the modulated DSC abrupt change in heat capacity between 357.8 and 358.3°C is excellent. The modulated DSC results in this case were obtained using a series of "quasi-isothermal" experiments at 0.5°C intervals across the temperature range of the transition. This "quasi-isothermal" approach, based on modulated DSC's ability to determine heat capacity from the instantaneous sinusoidal heating profile rather than the underlying heating rate, is preferred since it eliminates time-dependent (hysteresis) effects.

REFERENCE

1. TA Instruments Thermal Applications Note TN-24.

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