Evolved Gas Analysis of Iron Powder Using a TG/DTA System with a High -resolution Multi-turn Mass Spectrometer

Introduction

Thermogravimetry/differential thermal analysis (TG/DTA) systems are often used to analyze the thermal behavior of metal materials. These systems measure changes in sample weight as they relate to programmed oven temperatures. It is generally known that during the measurement process, as the weight decreases, gases are evolved. TG–MS is a hyphenated technique that combines TG/DTA with mass spectrometry (MS) to measure both the weight/thermal change and analyze the evolved gases simultaneously during a programmed temperature ramp. Typically, low-resolution MS systems (e.g., quadrupole MS) are used in TG–MS. However, these systems cannot separately detect ions such as carbon monoxide (CO – *m/z* 27.99491) or nitrogen (N_2 – *m/z* 28.00615). Consequently, the resulting evolved-gas data for these gases, N_2 and CO, can be misinterpreted.

Recently, we combined a TG/DTA system with a compact high-resolution multi-turn MS (TG/HR–MS). In this application note, we show the measurement results of iron powder by using TG/HR-MS.

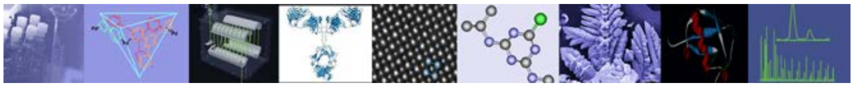
Experiment

Figure 1 shows the TG/HR–MS system in which the TG/DTA (STA 449 F1 Jupiter®; manufactured by NETZSCH) is combined with a HR–MS (INFITOF JMS-MT3010HRGA; manufactured by JEOL Ltd.). The system also includes a gas chromatography (GC) oven (7890B; manufactured by Agilent) in order to provide a heated transfer line into the MS. We measured 5 g of a commercial iron powder with the TG/HR–MS. Table 1 shows the TG/HR–MS conditions.

Figure 1. TG/HR–MS system.

Table 1. Measurement conditions.

TG/DSC; STA 449 F1 Jupiter®(NETZSCH)	
Furnace temp.	200˚C→20˚C/min→1480˚C
Transfer line temp.	300˚C
Purge gas flow	He (100 mL/min)
Split ratio	100 : 1
GC; 7890B (Agilent Technologies)	
Oven temp.	300˚C
Column	Deactivated capillary metal tube 5 m x 0.32 mm i.d.
HR-MS; JMS-MT3010HRGA (JEOL)	
Ion source temp.	300˚C
Interface temp.	300˚C
Ionization mode	EI (70 eV, 50 µA)
Measurement mode	Extended
Resolution	R ≒ 5000
Scan range	*m/z* 0.8 – 200



Results

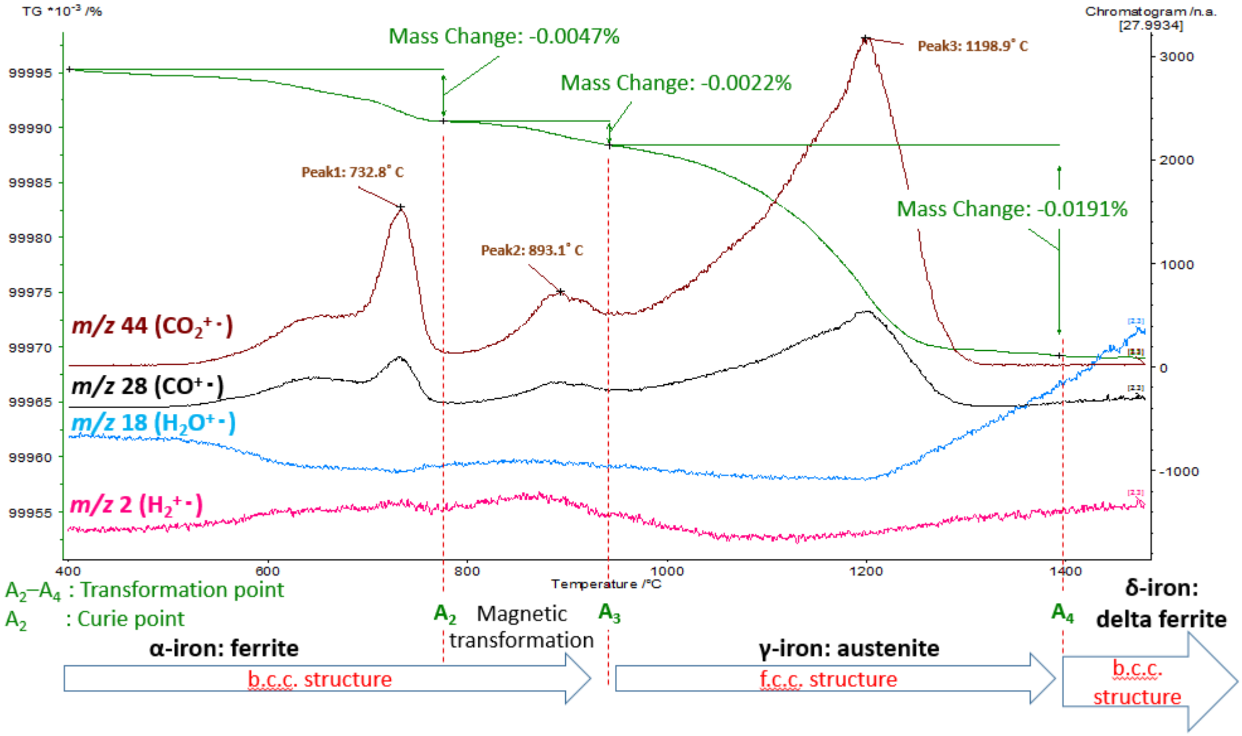


Figure 2. TG signal and evolved-gas behavior of ion powder in the He purge gas.

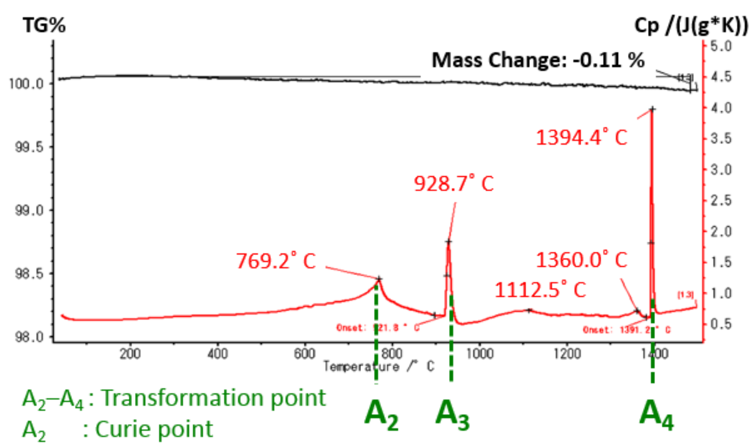


Figure 3. TG/Cp-specific heat capacity signal using TG/DSC.

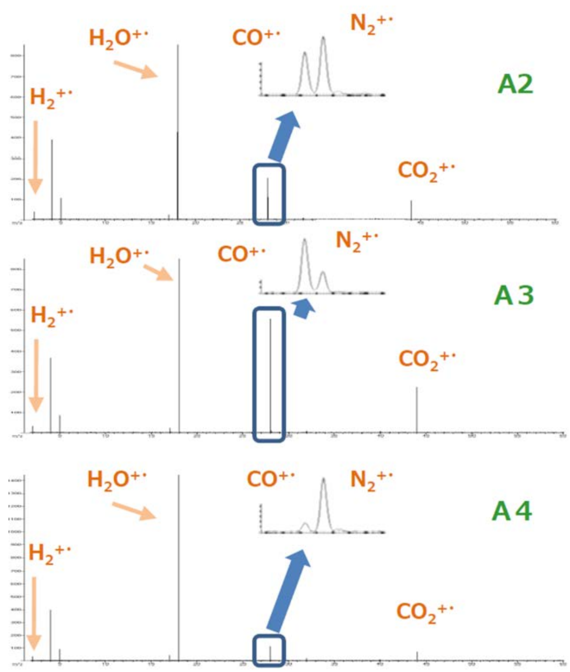


Figure 4. Mass spectra at A_2, A_3, and A_4.

Figure 2 shows the TG signal and extracted-ion chromatogram (EIC) for the gases detected from iron powder in a helium atmosphere. There were three stepwise regions of iron powder weight loss. The evolved gases were measured as the weight loss occurred, and the exact masses were estimated as H_2, H_2O, CO, CO_2, etc. Since the EICs for both CO and CO_2 showed exactly the same behavior, these results suggest that the observed CO was actually a fragment ion from CO_2. A comparison between this data and the TG/Cp data obtained with the TG/DSC (STA 449 F1 Jupiter®) in Figure 3 suggests that all of the CO_2 EIC peaks were detected before each transformation point (A_2, A_3, and A_4). The mass spectra at each transformation point (Fig. 4) suggest that CO_2 evolved from the inside of the sample as the crystal structure changed near the structural-phase transition points (A_3 and A_4). CO_2 also evolved before the Curie point (A_2) with weight loss of 0.0047%, which suggests that CO_2 evolved as the magnetic domains and domain walls changed during the magnetic-phase transition process. Based on our experience, CO_2 gas volume differs from sample to sample as a result of differences/variations in the manufacturing process.

Conclusion

The NETZSCH STA 449 F1 Jupiter® TG, combined with the JEOL INFITOF JMS-MT3010HRGA, provided high-quality TG/HR–MS data that made it possible to easily follow the thermal behavior of metal in greater detail. These results also suggest that this TG/HR–MS system would be useful for quality control of metal products.

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