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CONSTRUCTION OF A THERMOANALYZER FOR DTA–TD–TMAG–T MEASUREMENTS ON METALS UP TO 1100 °C*

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A thermoanalyzer is described for the simultaneous testing of metals and alloys by three methods: differential thermal analysis (DTA), thermal dilatometry (TD) and thermomagnetometry (TMAG). One reference specimen is used as a standard for each of the above kinds of analysis. Measurements can be performed in vacuum or in a static or dynamic atmosphere of gases at any pressure between normal and 5 x 10^{-5} Torr. The temperature of the sample can be changed linearly in the range 20–1100 °C. DTA and TD are performed classically, whereas TMAG is based on the temperature and magnetic field–dependences of the reversible magnetic susceptibility of the sample. Some analysis results are presented.

Most of the instruments designed for thermal analysis permit different kinds of measurements simultaneously. A combination of the different methods of thermal analysis is very fruitful from the aspect of the ease of interpretation of the physical or chemical phenomena observed during heating or cooling of the sample under investigation [1].

One of the aims of this paper is a short description of a thermoanalyzer designed for the differential thermal analysis (DTA), thermal dilatometry (TD) and thermomagnetometry (TMAG) of metals during one thermal run. Evolved gas analysis (EGA) is also possible.

The coupling of TMAG with other techniques of thermal analysis is usually difficult. In the presented thermoanalyzer, this problem is solved by means of a magnetic system which permits use of the Gans law [2] for this purpose.

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Description of the thermoanalyzer

1 Experimental chamber

The central part of the thermoanalyzer is the measuring cell (Fig. 1) incorporating a silica glass support [1], the samples [14] (one of the samples is the standard for comparative measurements), a radiative heater [9], a reflection tube [10], and a stainless steel tube [11]. The measuring cell is connected with a suitable

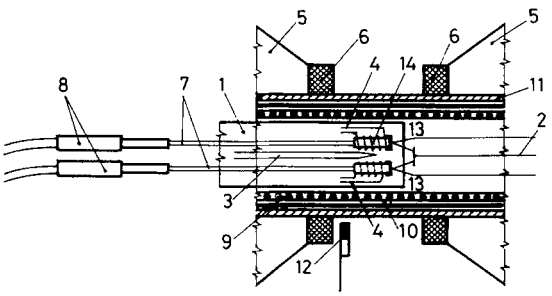

Fig. 1 Experimental chamber of the thermoanalyzer (description in the text)

vacuum or gas inlet system. The sample and reference are both cylindrical, with appropriate dimensions (∅ 3 × 30 mm). The temperature difference between the sample and reference (DTA) is measured with differential connected Pt/PtRhlO thermocouples [2] in thermal contact with the samples by means of two thin platinum plates [13]. For regulation, the temperature of the furnace is measured by a third thermocouple [3] in the vicinity of the specimens.

Dilatation or contraction of the samples (TD) is investigated with two sensors (linearly variable differential transformers) [8] connected with the specimens by means of two silica glass tubes [7].

Information on the magnetic state of the ferromagnetic samples (TMAG) is taken from the coils [4] surrounding the samples.

The sample is magnetized by an external electromagnet [5] and Helmholtz coils [6]. The value of the static or slowly changed magnetic field is measured with a Hall probe [12].

2 General description

The electrical signals generated by the probes in the measuring cell are measured with a suitable electronic apparatus controlled by the microcomputer; they can be depicted, for example, with a multi-pen recorder.

The temperature of the samples can be changed up to 1100° at different rates from 0.75 deg·min^{-1} to 20 deg·min^{-1}, with linearity better than 0.5%.

The calorimetric sensitivity of the instrument is estimated to be 0.01 cal g^{-1}·s^{-1} at the point of the magnetic phase transition of nickel (heating rate: 20 deg·min^{-1}).

The dilatometric measurements (TD) can be conducted in three ranges per full scale of the recorder: 20 μm, 200 μm and 1000 μm, with an accuracy of about 1%.

Two kinds of TMAG are possible in this thermoanalyzer. Firstly, a simple registration of the initial or reversible susceptibility *vs.* temperature is possible for a ferromagnetic sample with or without standard, giving information on its Curie point. In this way a qualitative analysis is provided.

Secondly, quantitative TMAG is possible from the knowledge of the temperature-dependence of the saturation magnetization of the sample. This dependence can be obtained by recording the reversible susceptibility *vs.* a slowly changed magnetic field at constant temperature. Through use of the semi-empirical Gans equations:

$$\frac{J}{J_s} = L(x)$$

$$\frac{\chi_r}{\chi_{rp}} = 3\,L\cdot(x)$$

where J = magnetization,
J_s = saturation magnetization,
χ_r = reversible susceptibility,
$L(x)$ = Langevin function,
χ_{rp} = initial susceptibility and
x = a certain parameter

the saturation magnetization is determined as a function of the normalized magnetic susceptibility. These measurements are repeated at different temperatures.

The composition of the gases evolved by the sample during thermal analysis is of interest.

An EGA module can easily be coupled to the described thermoanalyzer. Mass spectrometry, thermal conductivity or preferably gas chromatography sensors can be applied.

Example of results

Figure 2 presents the results of a simultaneous DTA, TG and TMAG study of a sample of ferromagnetic FeCo60 alloy [3]. Copper was used as standard reference. The temperature was elevated at a rate of 20 deg·min^{-1}. For the initial susceptibility detection, an excitation field of 0.2 Oe with a frequency of 1 kHz was used.

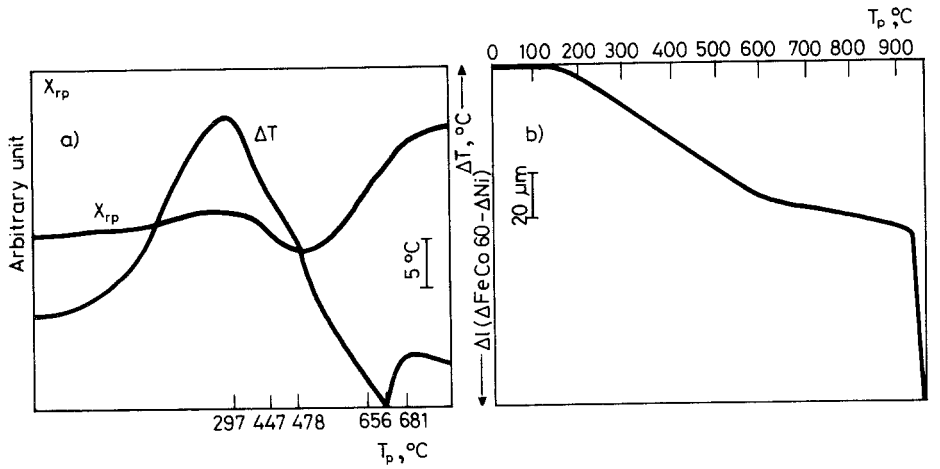

Fig. 2 Results of simultaneous DTA, TD and TMAG analysis of FeCo60 alloy. a) DTA and TMAG, b) TD

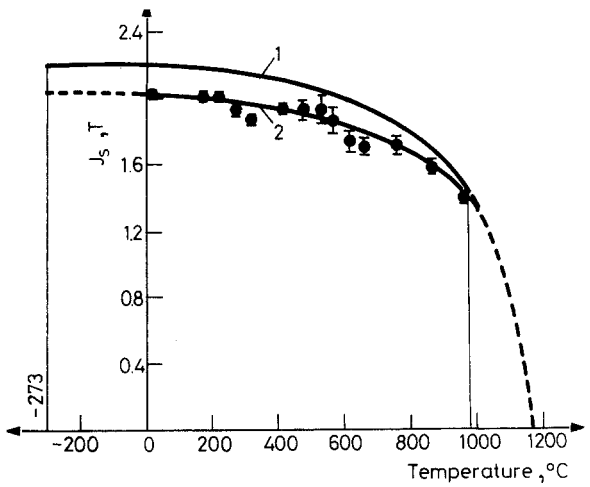

Fig. 3 Temperature-dependence of saturation magnetization of FeCo60 alloy: curve 1 taken from [4], curve 2 from present measurements

Figure 3 compares the results of saturation magnetization measurements with the above method for FeCo60 alloy (curve 2) with those in [4] (curve 1). These two curves confirm the possibility of quantitative TMAG with the described thermoanalyzer.

References

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Zusammenfassung — Es wird ein Thermoanalysator zur simultanen Untersuchung von Metallen und Legierungen durch drei Methoden beschrieben: Differentialthermoanalyse (DTA), Thermodilatometrie (TD) und Thermomagnetometrie (TMAG). Für alle drei Verfahren wurde die gleiche Referenzsubstanz als Standard benutzt. Messungen können im Vakuum oder is statischer bzw. bewegter Atmosphäre von Gasen beliebigen Druckes zwischen Normaldruck und $5 \cdot 10^{-5}$ Torr durchgeführt werden. Die Temperatur der Probe kann zwischen 20 und 1100 °C linear variiert werden. Während DTA und TG die herkömmlichen Verfahren zu Grunde liegen, basiert TMAG auf der Temperatur- und Magnetfeldabhängigkeit der reversiblen magnetischen Suszeptibilität der Probe. Einige Analysenergebnisse wurden dargestellt.

Резюме — Описан термоанализатор для анализа металлов и сплавов одновременно тремя методами: дифференциальным термическим анализом (ДТА), термической дилатометрией (ТД) и термомагнетометрией (ТМАГ). Один образец сравнения был использован в качестве стандарта во всех трех методах анализа. Измерения могут проводится в вакууме, в статической и динамической атмосфере газов при давлении от нормального до $5 \cdot 10^{-5}$ торр. Температура образца может линейно изменяться в интервале 20–1100°. ДТА и ТД измерения проводятся классическим методом, тогда как основой термомагнетометрических измерений является зависимость обратимой магнитной восприимчивости образца от температуры и магнитного поля. Представлены результаты такого анализа.