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TEMPERATURE CALIBRATION OF THERMO-BALANCES WITH NICKEL–IRON–NICKEL-BIMETAL

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Abstract

The behaviour of Ni–Fe–Ni sandwich material was investigated in air atmosphere up to 850°C with the NETZSCH microbalance TG 209. The aim was to determine if this substance is suitable for calibration.

The results show that nickel–iron–nickel-bimetal makes it possible to calibrate thermobalances whilst expending a minimum of time and effort.

Keywords: calibration, Ni–Fe–Ni sandwich material, thermobalance

Introduction

The only accepted method [1] for temperature calibration of thermobalances is the measurement of the transition from the ferromagnetic to the paramagnetic state of metallic specimens in an inhomogeneous magnetic field. These specimens are either a part of ICTA standard set 761 [2] or are in a known relation to it.

The measurable transition is a characteristic property for the pure substances only, not for their oxides.

However, since materials with a high content of iron, such as Trafoperm (96.9% iron, 2.8% silicon) or pure iron are very sensitive to oxidation, the appropriate experiments had to be carried out up to now in an inert gas atmosphere with the lowest possible oxygen content. An adequately low partial oxygen pressure could only be reached when the instrument had a vacuum tight casing.

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Experimental

The samples with a mass of 100 mg were made from a sheet of roll-bonded Ni–Fe–Ni. The original materials were a nickel and an iron sheet with the following composition:
impurity content in ppm:

Ni (Co 58, Fe 29, C 35, O_2 25)

Fe (Mn 1400, Cr 1200, Al 640, S 410, V 200, Ni 130, W 36, P 33, Co 27, Sn 23)

A NETZSCH thermobalance TG 209 was used, with a resolution of ±1 μg and a temperature range from room temperature to 1000°C, which was calibrated in advance with pure iron and nickel material in nitrogen atmosphere.

A frame of magnets was attached to the top of the instrument furnace (Fig. 1) for generating the necessary inhomogeneous magnetic field.

As long as a single component of bimetal, e.g. nickel, is in the ferromagnetic state, an additional force acts on the substance and pulls it towards the magnetic field. But when the substance passes its specific Curie temperature, the additional force disappears and the sample moves in the opposite direction.

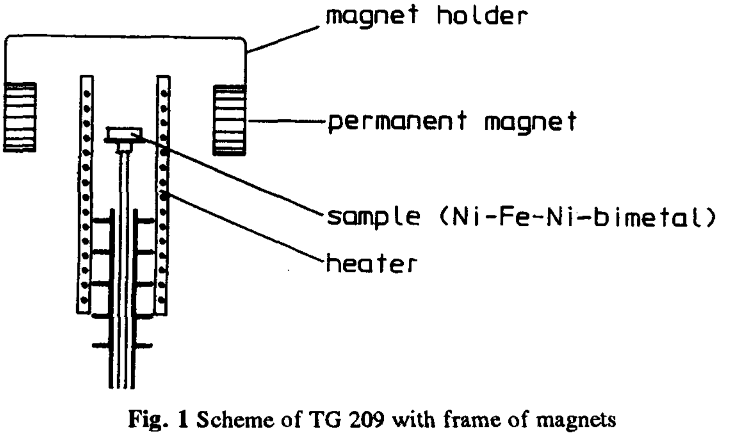

Fig. 1 Scheme of TG 209 with frame of magnets

If the magnet is now positioned somewhat above the sample the corresponding measuring curve shows a sharp weight increase (Fig. 2).

Results and discussion

The small step in the middle of the temperature range of Fig. 2 is presented again in a larger scale in Fig. 3. The increase of the step height is obviously visible during the sample's further thermal stress (see also Table 1).

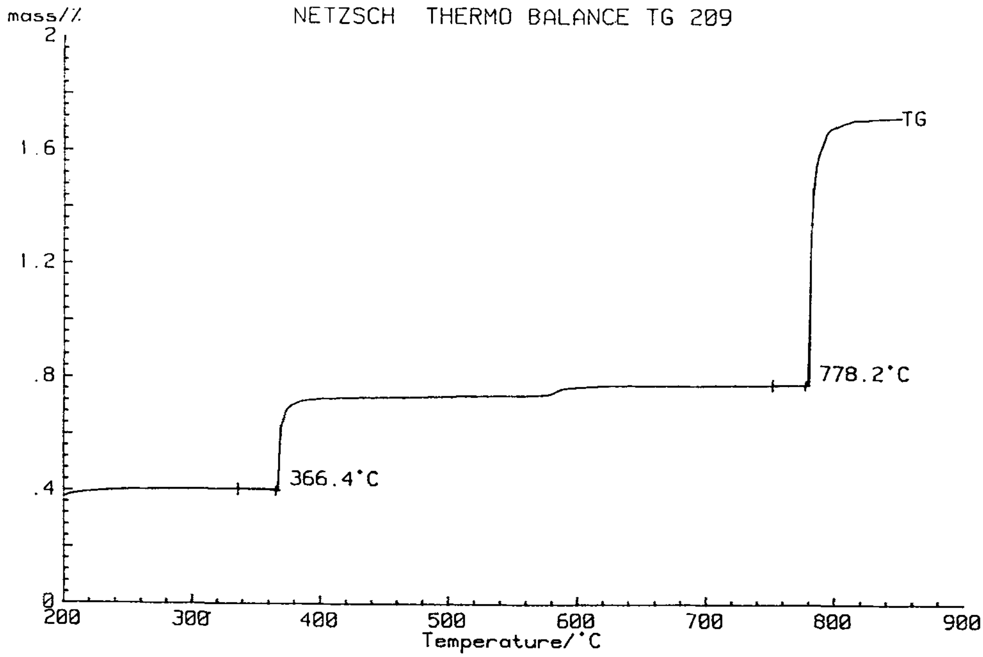

Fig. 2 TG curve of Ni–Fe–Ni sandwich; atmosphere: air; heating rate: 50 deg·min^{-1}

This behaviour is most probably connected to the slow expansion of a ferromagnetic compound at the interface of the sandwich material. The temperature range of the transition indicates that, for example, different defect spinels of the general formula $NiOFe_2O_3$ [3] could possibly be responsible for this.

Table 1 Increase in height of intermediate step in relation to the thermal stress

Number of heating / cooling cycles	Weight change / 10^{-2} mg
2	2.1
8	4.5
18	8.1

The measuring curves for both nickel and iron show clear steps, the height of which can still be easily evaluated after 40 measurements. These steps as well as the weight increase of only 0.6 mg, indicate the extremely low oxidation tendency of the specimens.

Table 2 Measured transition temperatures of nickel and iron

Material	Heating rate / $deg \cdot min^{-1}$	Transition temperature /°C (average of 6 measurements)	Standard deviation/ K
Ni	50	366.6	0.7
	20	358.8	0.3
	5	355.1	0.1
Fe	50	778.7	0.7
	20	774.4	0.3
	5	772.1	0.2

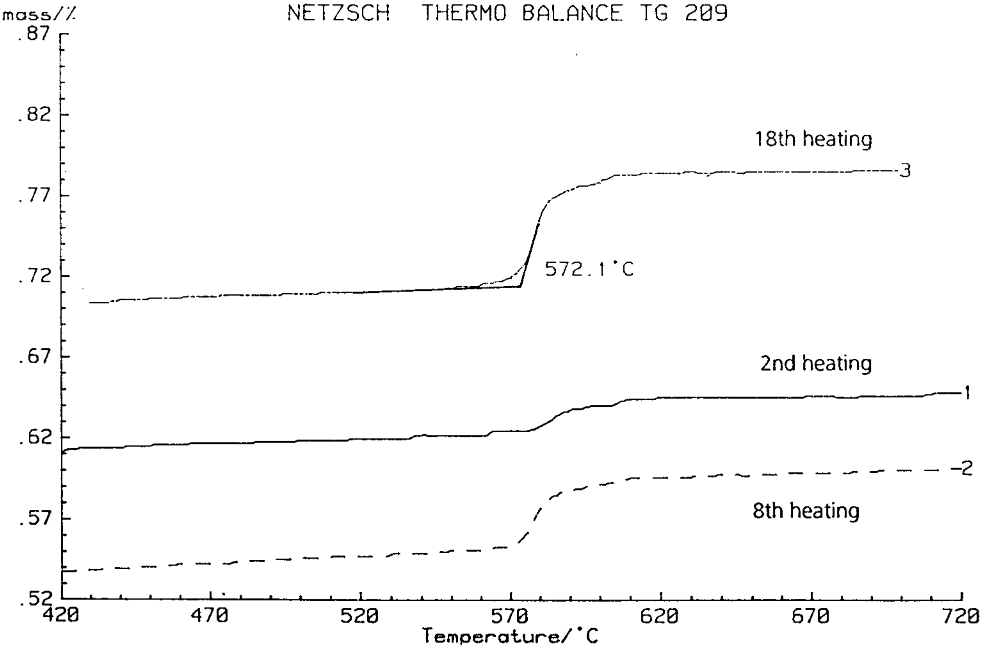

Fig. 3 Magnetic transition of the additional ferromagnetic phase; comparison of the TG steps with the number of heatings; atmosphere: air; heating rates: 50 $deg \cdot min^{-1}$ (1) and 20 $deg \cdot min^{-1}$ (2 and 3)

In the heating segments the extrapolated onset temperatures were evaluated. The standard deviation of measurements carried out under identical conditions (repeatability) is maximum 0.7 K, as can be seen in Table 2. This is much more exact than the value 2.1 K as given in the ASTM standard.

The plot of the tabulated values results in a linear dependence between the average extrapolated onset temperature (Figs 4 and 5) and the heating rate. Extrapolation of the curve to heating rate 0 gives a temperature of 354°C for nickel and 771°C for iron. The difference between the transition data in literature [4] 358°C and 770°C and those determined above is caused by the high sensitivity of the magnetic transition compared to that of the compound.

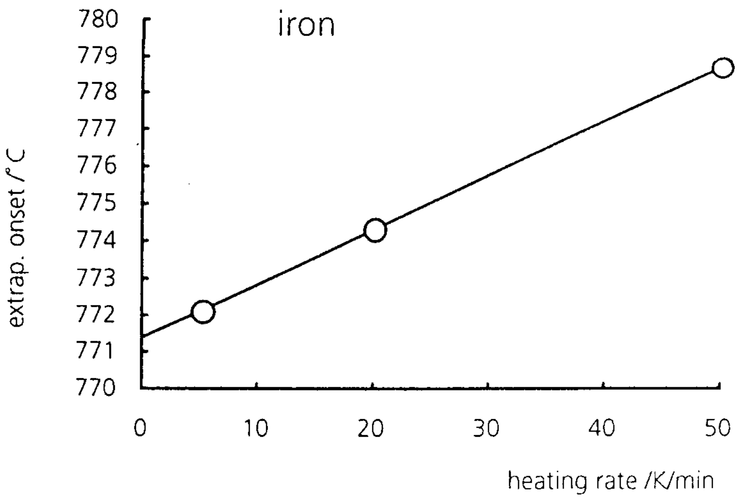

Fig. 4 Iron plot of the average extrapolated onset temperature *vs*. heating rate

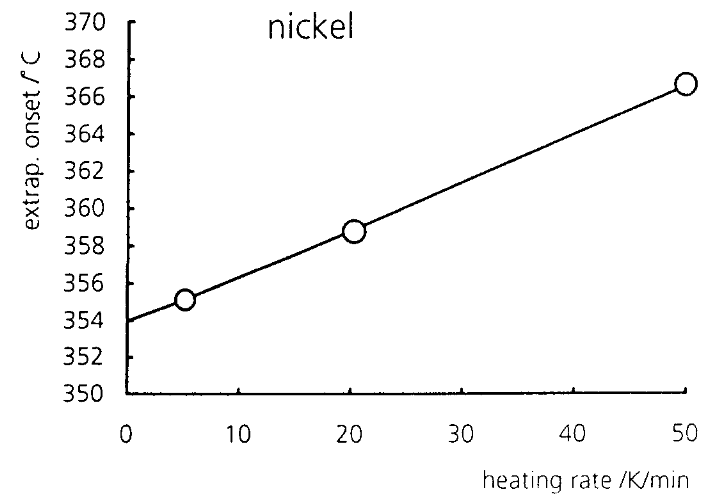

Fig. 5 Nickel plot of the average extrapolated onset temperature *vs*. heating rate

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2 J. O. Hill, For Better Thermal Analysis and Calorimetry, ICTA Edition III, 1991.
3 Ch. Kittel, Einführung in die Festkörperphysik, 5. verbesserte Auflage, R. Oldenbourg Verlag München/Wien, 1980.
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Zusammenfassung — Um herauszufinden, ob sich Nickel–Eisen–Nickel-Bimetall zur Kalibrierung von Thermowaagen in Luftatmosphäre eignet, wurde sein Verhalten im Temperaturbereich bis 850°C mit einer NETZSCH TG 209 untersucht.
Die Ergebnisse zeigen, dass dieses Material neue Möglichkeiten eröffnet um die Temperaturkalibrierung mit minimalem Messaufwand durchzuführen.