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A NEW ADVANCED THERMO-MICROBALANCE

P. Davies, W.-D. Emmerich** and S. Knappe***

*NETZSCH MASTERMIX LTD., VIGO PLACE, ALDRIDGE, WALSALL, WS9 8UG, UK
**NETZSCH-GERÄTEBAU GMBH, WITTELSBACHERSTR.42, D-8672 SELB, FRG

The capabilities of the new NETZSCH thermo-microbalance TG 209 are illustrated by four applications from the polymer industry.

Keywords: new thermo-microbalance, polymers

Introduction

Thermogravimetry (TG) is playing an increasingly important role in the polymer industry. As a supplement to DSC 200 and DMA 242, the new NETZSCH thermo-microbalance TG 209 is ideal for optimization and reproduction of processing procedures, as well as for quantitative analysis in quality control, failure analysis and environmental protection.

In this paper, four different problems were investigated with the TG 209:
1) Thermal degradation of a friction ring made of glass-fibre-reinforced polybutyleneterephthalate-polytetrafluoroethylene (PBT/PTFE) blend. Here the content of PTFE is important for the selected anti-friction properties of the finished part. 2) The distribution of glass fibre in an exterior automotive thermoplastic polyurethane (TPU) part. 3) Comparison of a new an aged cable insulating material of thermoplastic elastomer (TPE). 4) A rapid quality control test of a blend of natural rubber/ethylene propylene diene monomer (NR/EPDM) rubber mixture.

Experimental

Apparatus

The unilaterial microbalance TG 209 (Fig.1) works according to the principle of electromagnetic power compensation. The resolution is 1 μg within a weighing range of 32 mg.

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The micro furnace with the sample holder is situated above the weighing chamber. The temperature range of 20° to 1000°C is ideal for polymer analyses. The stainless steel, water-cooled micro furnace ensures short measuring cycles with heating rates of 0.1 deg·min^{-1} to 80 deg·min^{-1} and a cooling time of 10 min from 1000° to 100°C.

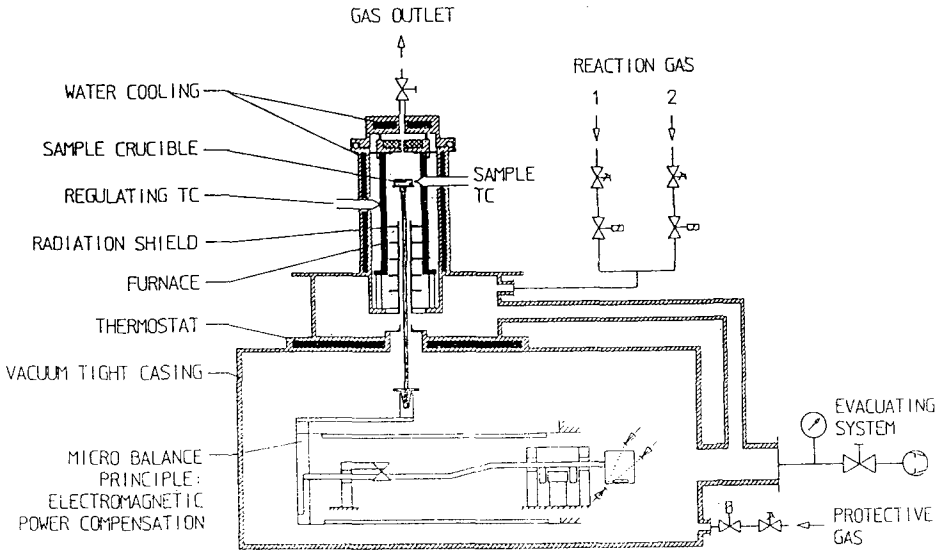

Fig. 1 Schematic diagram of TG 209

The weighing chamber can be purged with inert gas in order to protect the microbalance from corrosive gases which could evolve from the sample.

A gas change can be made at the sample chamber either manually or controlled by the computer system, depending on temperature or time.

The TG 209 is combined with the TA System Controller TASC 414/2 for temperature control and data acquisition.

The TG 209 can be coupled with a mass spectrometer, gas chromatograph or Fourier transform infrared spectrometer for gas analysis.

Materials

The following polymers were investigated:

1) glass-fibre-reinforced PBT/PTFE blend (6.60 mg),
2) glass-fibre-reinforced TPU (10.16, 8.22, 6.00 mg),
3) TPE, aged and new (2.47, 2.39 mg),
4) NR/EPDM rubber mixture (4.51 mg).

Procedure

1) The glass-fibre-reinforced PBT/PTFE blend was studied in a platinum (Pt) pan in nitrogen at a heating rate of 20 deg·min^{-1}. A gas change from N_2 to air was made at 650°C.

2) Three different glass-fibre-reinforced samples of TPU were investigated:

A) granulate as a reference,

B) sample taken from a finished part near to the runner,

C) sample taken from the same finished part far from the runner.

Each sample was placed in an open Pt crucible. The atmosphere was automatically changed from nitrogen to oxygen at 600°C. The heating rate was 30 deg·min^{-1}.

3) A cable insulating material of TPE was aged at 120°C for 4510 hours. Comparison tests of aged TPE and a new TPE sample were carried out in Pt crucibles in an air atmosphere at a heating rate of 30 deg·min^{-1}.

4) The NR/EPDM rubber mixture was investigated in a Pt pan in nitrogen at a heating rate of 80 deg·min^{-1}.

Results and discussion

1) Glass-fibre-reinforced PBT/PTFE

In nitrogen the blend decomposes in two stages (Fig. 2). The first mass loss is due to the PBT content (51.6%). The second step (11.9%) corresponds to the content of PTFE which has a higher thermal stability. The third step shows the combustion of carbon in air (4.3%). The residue (32.2%) comprises glass fibre.

2) Distribution of glass fibre in a TPU part

To investigate whether there is a separation between the polymer matrix and the glass fibre, samples were taken from an exterior automotive TPU/GF part with a length of 1 m, which was injected at one end.

Figure 3 shows that there are no significant differences between the sample taken near to the runner (B), the sample taken far from the runner (C) or the granule used as reference (A). The distribution of glass fibre is equal in all three samples (25%); this is due to optimum processing conditions and good mould design.

3) New and aged TPE cable insulation

Figure 4 shows the comparison of new (1) and aged TPE (2) from an enlargement of the TG curve. While the new material (1) starts decomposing at 194°C, the aged material shows a mass increase of 0.4% from room temperature to

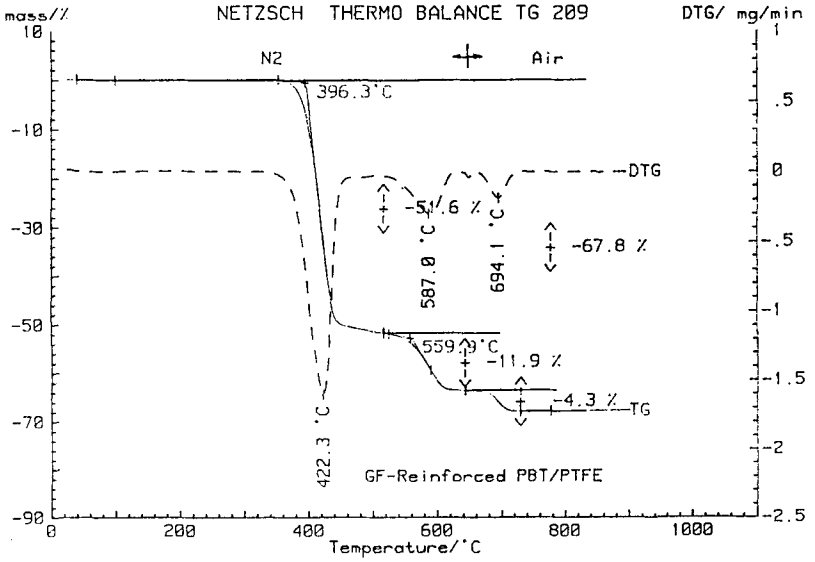

Fig. 2 Thermal degradation of a glass fibre reinforced PBT/PTFE blend

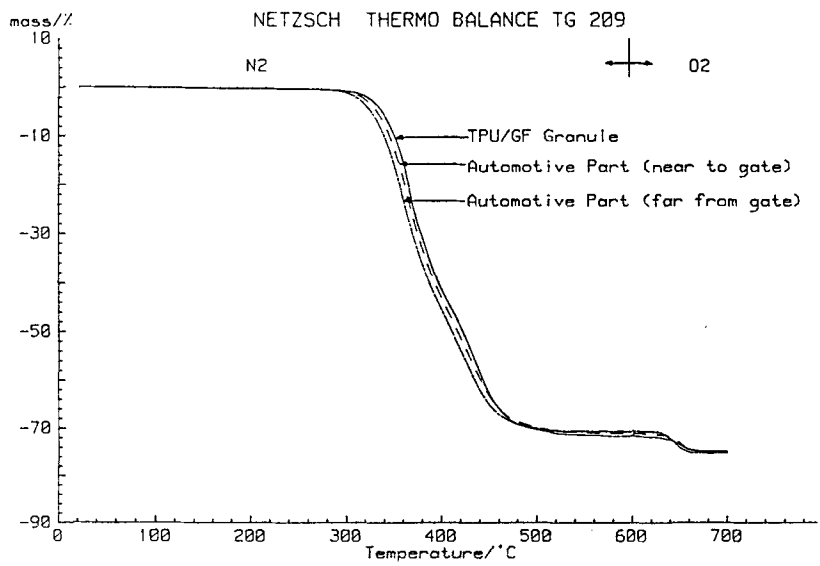

Fig. 3 TG comparison of glass fibre reinforced samples of TPU; A: granulate, B: near to the runner, C: far from the runner

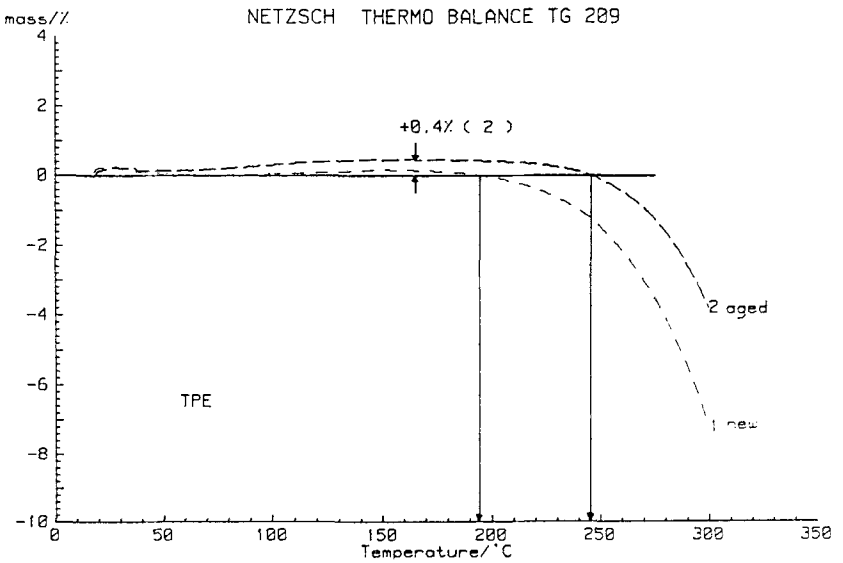

Fig. 4 TG comparison of new (1) and aged TPE (2)

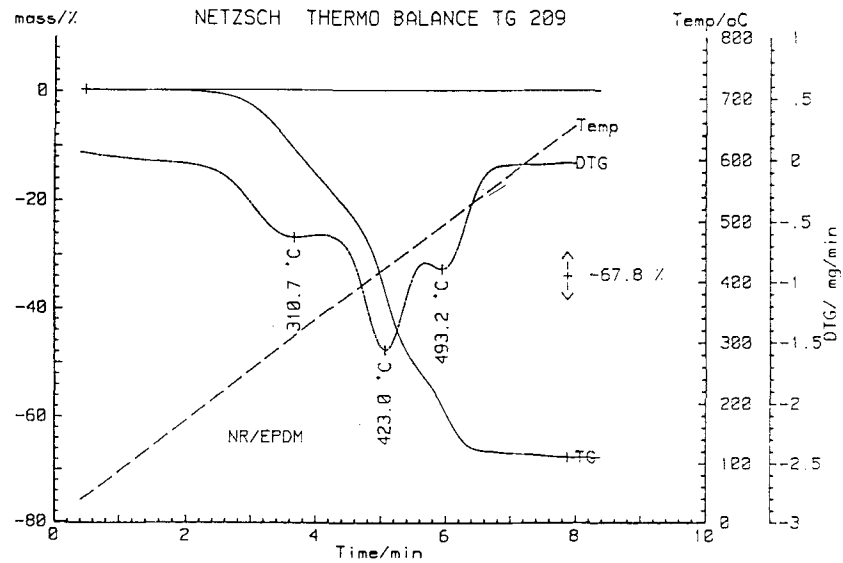

Fig. 5 TG and DTG for an NR/EPDM rubber mixture

165°C. This is due to reaction between the more reactive chemical groups in the aged TPE and oxygen. Because of aging, this material also starts decomposing at a higher temperature (245°C). These two points are useful when characterizing aging.

4) NR/EPDM rubber mixture

Even at high heating rates such as 80 deg·min^{-1} the separation of NR and EPDM is possible (Fig. 5).

The first DTG peak at 311°C is due to evolution of volatile plasticizers and residues of the vulcanization system and of the antioxidant. The second DTG peak (423°C) relates to NR, the third (493°C) to EPDM.

Although the fast heating rates result in DTG peak temperatures which are higher than expected, this procedure is often sufficient for rapid quality control of incoming material.

Zusammenfassung — Anhand von vier Anwendungsbeispielen in der Polymerindustrie werden die Fähigkeiten der neuen NETZSCH Mikrothermowaage TG 209 beschrieben.