

Simultaneous Measurement of Transformation Energetics and Mass Changes of Polymers using the STA 409 PC Luxx®



J. Blumm, S. Lemarchand
NETZSCH-Gerätebau GmbH, Wittelsbacherstr. 42, 95100 Selb/Bavaria, Germany

1. Introduction

Simultaneous thermal analysis refers to the simultaneous application of two or more thermoanalytical methods on one sample at the same time. This term is in most cases used for simultaneous measurement of the mass changes and calorific effects on a sample under thermal treatment. The benefits of such a system are obvious. Frequently the material available for testing is costly or difficult to produce. Using simultaneous thermal analysis one has the chance to get information on the transformation energetics and the mass change on one sample in one run under identical conditions. Of course, the time necessary for the tests is also reduced by a factor of two. Additionally, it can never be excluded that there are influences on the measurement conditions and/or sample preparation if two different instruments or samples are employed for the tests. Using an STA the comparability of characteristic temperatures measured on the TG and DSC runs is ensured. In case of inhomogeneous sample materials, problems resulting from differences in the sample composition for the TG and DSC measurements are excluded.



Figure 1: Simultaneous Thermal Analyzer STA 409 PC Luxx®

The new STA 409 PC Luxx® (figure 1) combines the advantages of a highly sensitive top-loading thermobalance and a high-temperature differential scanning calorimeter. The maximum sample mass as well as the measurement range of the balance are 18 g. The entire measurement range can be analyzed with a resolution of 2 µg (up to 0.00001%). Easily exchangeable sample carriers allow optimum adjustment of the system to different applications (TG, TG-DTA and TG-DSC measurements). The various sensors available are shown in figure 2. Different exchangeable furnaces for low temperatures (-120 ... 750°C) as well as for the high-temperature region (1550°C) are available. The vacuum-tight construction enables tests in defined (e. g. pure inert) atmospheres. Analysis of the measured data can easily be done with a standard PC system and a well-proven MS®-Windows™-based software package.

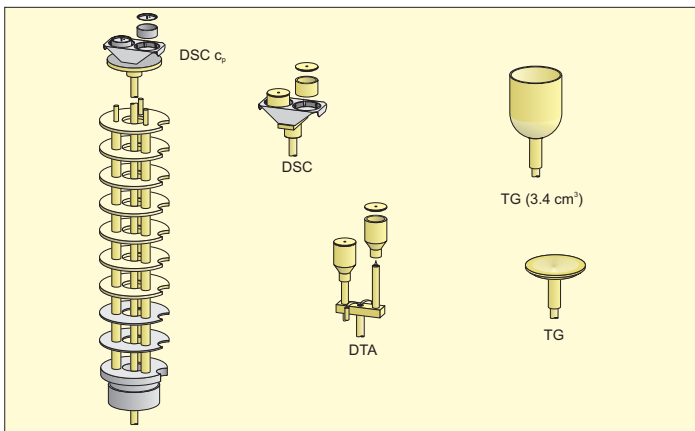


Figure 2: Sensors of the STA 409 PC Luxx®

2. Test results

Presented in figure 3 are the mass change and transformation energetics of a linear low-density polyethylene measured in a pure nitrogen atmosphere. Melting of the sample can be seen between 60 and 130°C. Characteristic peaks were found at 111, 117 and 122°C indicating that the sample is not pure but a blend of polyethylene with different densities. The endothermic decomposition of the material started at 393°C (DSC result). The mass loss of 100% during decomposition clearly shows that the sample contained no filler material.

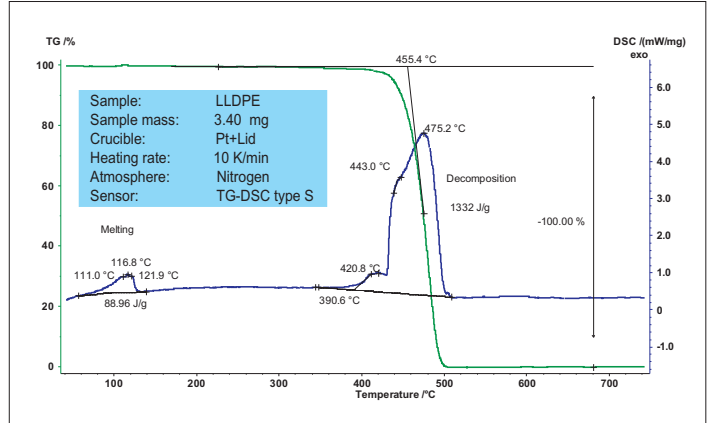


Figure 3: Mass change and transformation energetics of a LLDPE sample

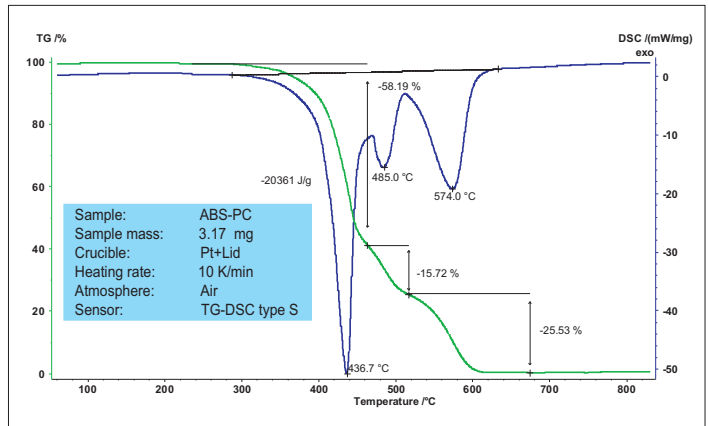


Figure 4: Mass change and transformation energetics of an ABS-PC blend

Presented in figure 4 are the mass change and transformation energetics of an ABS-PC blend measured in dynamic air. The different oxidation steps of various blend components (Acrylonitrile, Butadiene, Styrene and Polycarbonate) can clearly be seen in this presentation. Of course, due to the oxidizing atmosphere the decomposition is dominated by exothermal reactions. The different glass transitions of the blend components (between 100 and 150°C) are not evaluated, here.

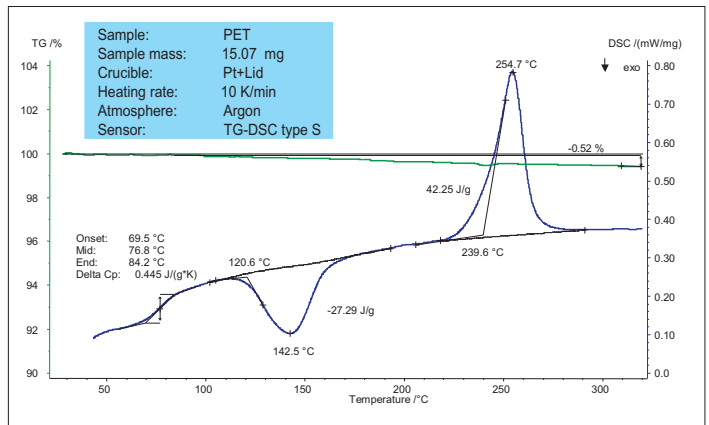


Figure 5: Mass change and transformation energetics of PET

Figure 5 shows the mass change and transformation energetics of PET. The test was carried out under Argon atmosphere. The DSC result clearly shows the glass transition (between 70 and 84°C). The cold crystallization was detected at an extrapolated onset temperature of 121°C. The crystallisation enthalpy was -27.3 J/g. Melting was measured at 240°C. The heat of fusion was 42.2 J/g. The enthalpy values allow, of course, the determination of the degree of crystallinity at room temperature (10.7%, here). The small mass loss of 0.517% above the glass transition is most probably due to the evaporation of processing additives.