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COMBINATION OF DSC 111 AND THERMOBALANCE : NEW SETARAM SIMULTANEOUS TG-DSC 111

P. LE PARLOUËR, SETARAM, 7, rue de l'Oratoire 69300 Caluire, France

ABSTRACT

The new SETARAM simultaneous TG-DSC 111 combining the well known capacities of the DSC 111 with the qualities of a symmetrical balance of very high precision is presented. The new instrument is especially designed for all investigations relative to interactions between gases and solid or liquid samples and to materials stability.

INTRODUCTION

Most of the thermogravimetric instruments, available on the market, are combined with a DTA detector, making possible two different measurements on a single sample. The DTA trace gives a better understanding of the thermogravimetric experiment by associating the sample mass changes to corresponding thermal effects. In this case, DTA is only used in a qualitative way, the detector consisting in one or some thermocouples, and is not able to associate precise heat variations to mass changes. A real improvement of the combination supposes the use of a quantitative detection unit, like a DSC. The original structure of SETARAM DSC 111 (open experimental tubes) (1) is particularly attractive for a combination with a thermobalance. This gives today the new SETARAM simultaneous TG-DSC 111.

DESCRIPTION OF THE SIMULTANEOUS TG-DSC 111

The DSC 111 (2) is built around two open refractory tubes, crossing the heating furnace. The detection unit, designed according to the Calvet principle, is located in the medium part of the tubes. In order to adapt the balance, the calorimeter is used vertically and fixed on a stand (Figure 1). A symmetrical balance is set above the calorimetric block. The beam of the balance has been designed in such a way that the suspensions are brought into line with the axis of each experimental tubes. The balance is moved by means of a sliding carrier along a screwed axis. The crucibles, hooked at the ends of the suspensions, are driven in the detection zone of the DSC by sliding the balance. The crucibles are centered without mechanical contact with the DSC tubes, and are fully surrounded by the detectors, making possible a good integration of the heat exchanged by the sample with the surrounding. The joining between the balance and the DSC is tight, making easy the

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work under gas flow. A vacuum purge of the instrument is possible before any investigation under gas flow control. Gas circulation and vacuum purge are controlled by means of external devices.

CHARACTERISTICS

Though the crucibles have no mechanical contact with the DSC tubes, the quantitativity of the detector is practically not affected.

The diameter of the crucibles is 5 mm (tube diameter : 7 mm), with 10 mm length. Different types of open crucibles are available : aluminium, platinum, alumina. The temperature range of the TG-DSC 111 is identical to the DSC 111 one : from - 123°C up to 827°C. Different heating functions are available : isothermal, step-heating, scanning (from $1°C.h^{-1}$ up to $30°C.mn^{-1}$). The limit of detection of the DSC 111 is unchanged, even when combined with a thermobalance : 15 µW in isothermal mode and 30 µW in scanning mode.

The background noise of the thermobalance is about 1 µg, that allows very sensitive thermogravimetric measurements. Due to the symmetrical set-up of the balance, the buoyancy effects in each tube are compensated, and the drift of the balance is very low when scanning. With identical empty crucibles, the value of the drift is around 10 µg, from ambient up to the upper limit of temperature.

The TG-DSC 111 is connected with a high performance computer for data acquisition and treatment. Temperature programmer can also be piloted by the computer, giving many different possibilities of scanning. Thermogravimetric and calorimetric data, after storage, are simultaneously treated according to different softwares. Heats and mass changes are correlated. The DTG curve can be numerically calculated.

Though the main interest of the instrument is the simultaneous thermogravimetric and calorimetric measures on the same sample, the thermobalance can be used alone, the DSC 111 block acting as a furnace. The DSC 111 can also be disconnected and set on a special horizontal stand. In such a situation, the classical applications of the DSC 111 can be run : investigations in tight cells, under high pressure, with pressure control... and so on (3).

APPLICATIONS

The TG-DSC 111 is especially designed for all investigations relative to interactions between gases and solid or liquid samples. The fields of applications are very wide, depending on the type of samples, the range of temperature, the nature of inert or reactive gases. Two main types of applications can be distinguished : analysis with evolved gas and analysis with gas interaction.

Applications with evolved gas are the main experimentations run on the actual TG-DTA instruments in a very wide range of temperature. With the TG-DSC 111, the mass losses are correlated with heat effects precisely measured. Tests are either run in scanning or isothermal mode. Especially in isothermal mode, the stability and the base line reproducibility of the DSC 111 make possible the record of slow reactions, that is difficult with a DTA detector. The experimentations with evolved gas under controlled atmosphere are well known : dehydration, dehydroxylation decomposition, pyrolysis,... The dehydration and dehydroxylation of bentonite under controlled argon atmosphere are an example of the use of the TG-DSC 111 from 0°C up to 820°C (Figure 2).

Interactions between gas and sample are among the most interesting applications of thermogravimetry and calorimetry. For example, it is possible to measure precisely the amount of gas adsorbed on a sample, and the corresponding heat of adsorption, even in the case of slow rate of adsorption. Kinetics parameters can be obtained from both gravimetric and calorimetric data.

The main applications are all types of gas adsorption (oxidation, reduction,) more generally chemisorption including catalysis investigations. With the low temperature capabilities of the TG-DSC 111, physisorption can also be investigated.

A typical application of the TG-DSC 111, in such a field is given by the combustion of coal (Figure 3). The DSC curve shows the classical exothermic effect with two peaks. Looking at the TG curve, it appears that the first exothermic peak is associated with a mass increase corresponding to adsorption of oxygen on coal. It is then followed by an important mass loss corresponding to the combustion of coal (second exothermic effect). This example shows the complementarity of the two methods.

CONCLUSION

With the well known capabilities of the DSC 111 and the high performances of a symmetrical balance, the TG-DSC 111 is an instrument to become a very useful tool for laboratories involved in gas-solid reactions, surface reactivity and materials stability.

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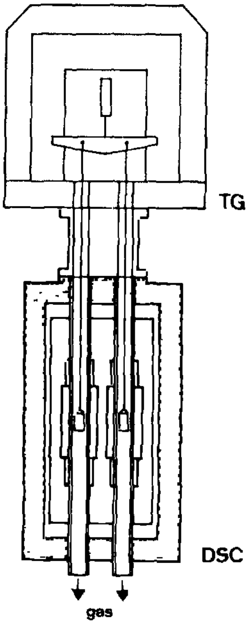

Figure 1 : Schematic cross-section of the TG-DSC 111

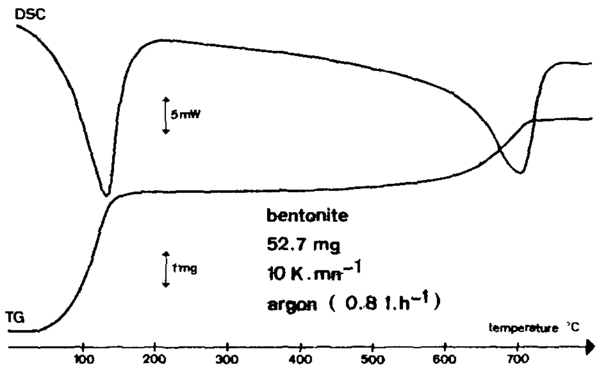

Figure 2 : Dehydration and dehydroxylation of bentonite

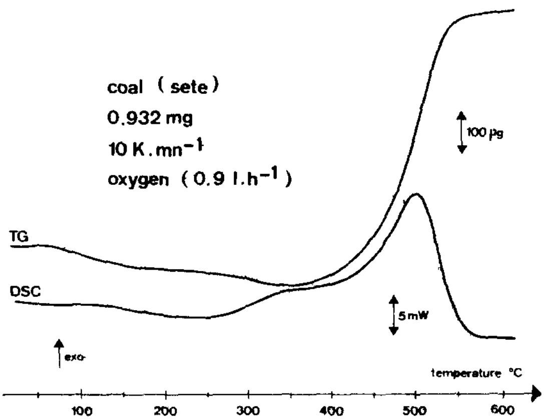

Figure 3 : Combustion of a coal sample