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SIMULTANEOUS DSC-TG UP TO 1700 K

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ABSTRACT

A DSC-TG-sample carrier up to 1700 K employs plate-type thermocouples as heat flux sensors. Flat bottomed sample pans ensure an almost ideal thermal contact with these sensors, resulting in high sensitivity and peak resolution. Its performance was tested with standard materials and some polymeric samples.

The construction of the sample carrier permits quantitative determinations of specific heat at high temperatures. Results are shown with sapphire, aluminium oxide, graphite and glass samples.

INTRODUCTION

The need for DSC results above 1000 K is well known and has now resulted in the development of this new DSC-TG sample carrier with max. temperature 1700 K.

The DSC sample carrier employs Pt10%Rh-Pt or NiCr-CuNi plate thermocouples as heat flux sensors, which are mounted on an alumina support.

The flat bottomed sample crucibles (Pt, Al, Al_2O_3) have a large contact area with the sensors, which together with the design of the measuring head ensures an almost ideal thermal contact.

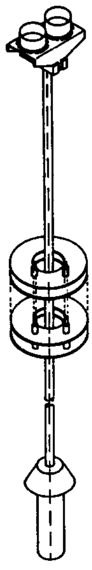

Fig. 1. DSC-TG sample carrier

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EXPERIMENTAL

Its performance with standard materials showed good agreement with literature values.

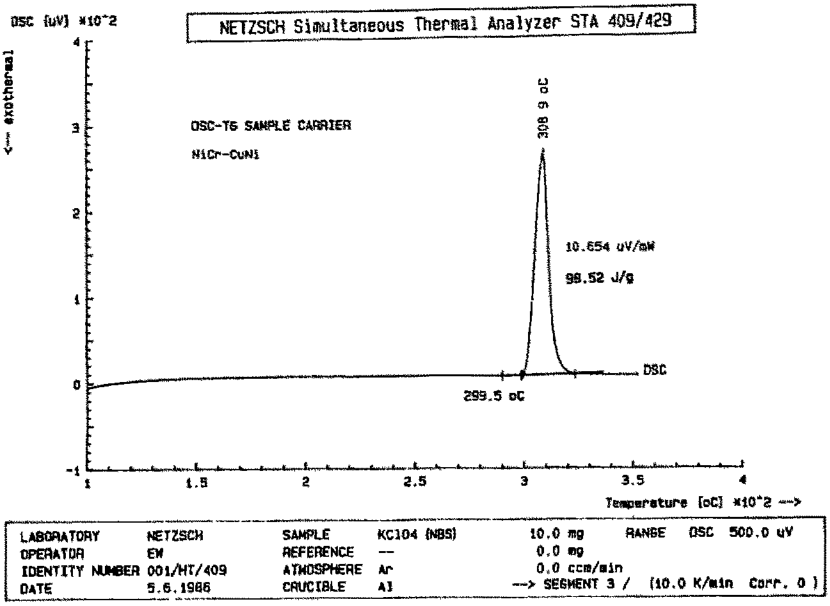

Fig. 2. shows the phase transisition of $KClO_4$ (NBS material)

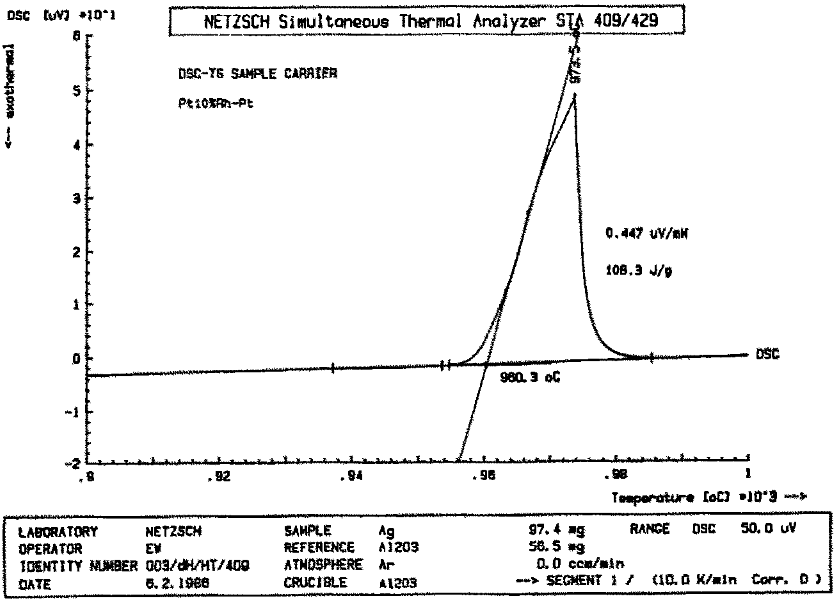

Fig. 3. shows the melting of a silver sample

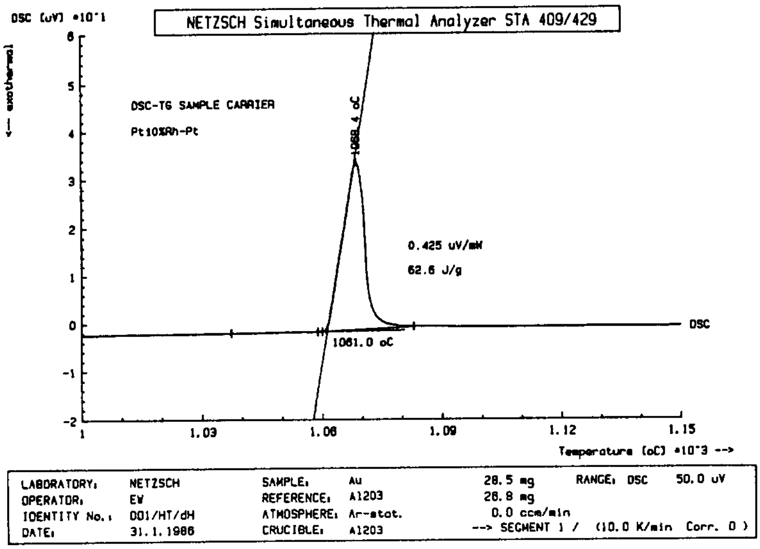

Fig. 4. the melting of a gold sample

Its performance with polymeric samples such as PET and EPDM with inorganic filler was also tested.

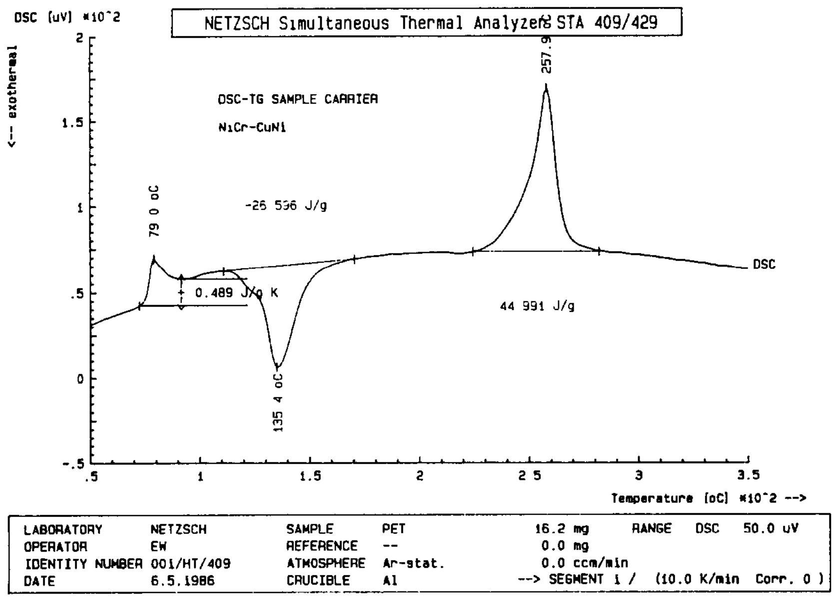

Fig. 5. shows a PET sample

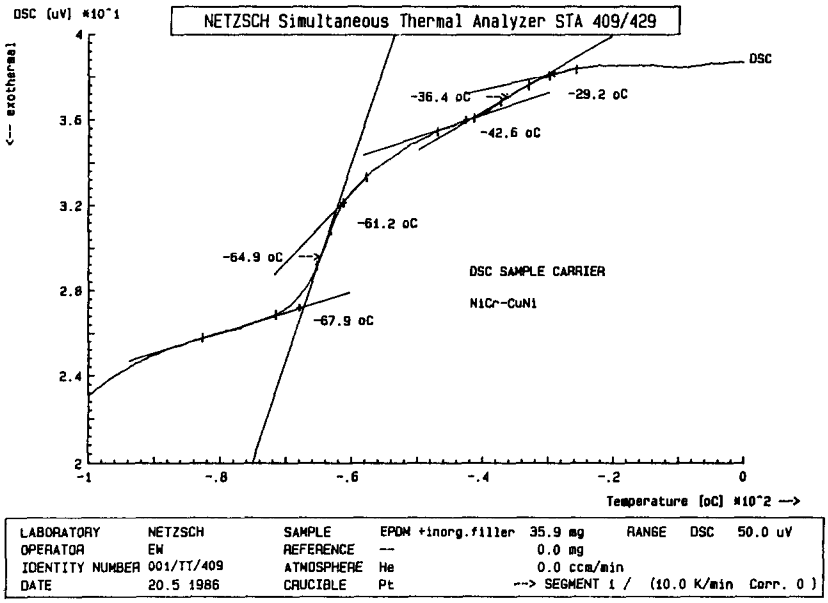

Fig. 6. shows the glass transition of EPDM at low temperatures

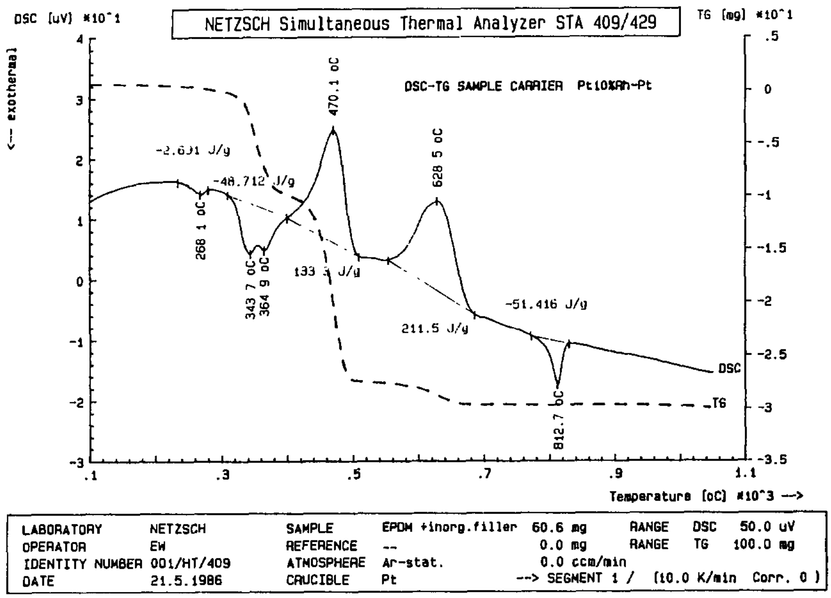

Fig. 7. shows the pyrolytic decomposition of EPDM in argon atmosphere up to 1200 K

The construction of the measuring head permits quantitative measurements of specific heat at high temperatures, a thermophysical property of growing significance.

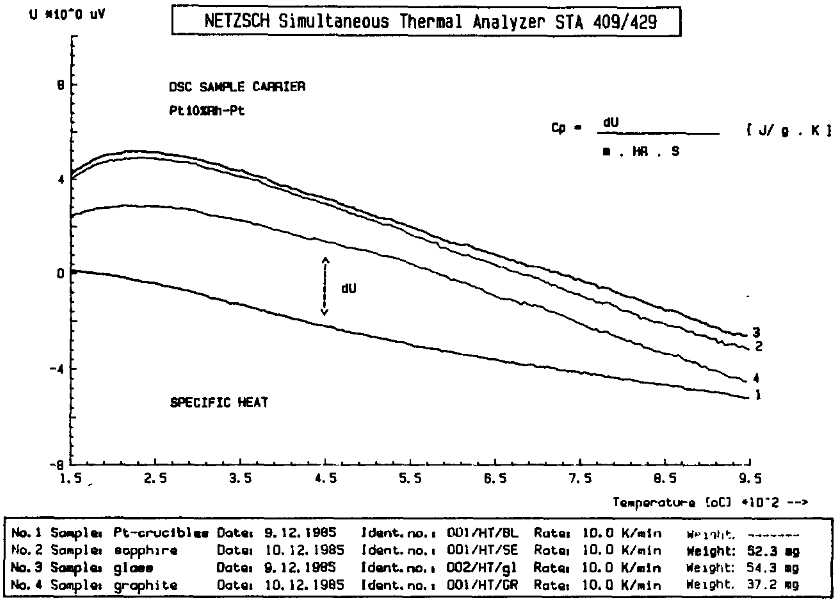

Fig. 8. shows some results obtained with samples such as alumina powder, graphite and glass.

Evaluation

$$c_p = \frac{dU}{m \cdot HR \cdot S} \qquad (J/g.K)$$

dU = voltage difference (μV)

m = sample weight (g)

HR = heating rate, effective (K/s)

S = sensitivity (μV/mW)

RESULTS

To summarize, the new DSC measuring head which can be fitted to DTA and STA (TG-DTA) units developed since 1973, has many advantages over conventional DTA/STA measuring heads, including ultimate detection limit, peak resolution, better base line and higher sensitivity.

Reproducibility: onset temperatures $\pm$0.5 K, enthalpy $\pm$ 1 %

Sensitivity: 1 μV/mW -(Pt10%Rh-Pt)-type, 12 μV/mW -(NiCr-CuNi)-type

Detection limit: for an enthalpy change of 100 J/g sample sizes as low as 30 to 50 μg can be used.

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