Solid phase transition in Zirconia

INTRODUCTION

Phase transitions are commonly characterized using DTA or DSC sensors. These techniques are very sensitive at low temperatures, but may face certain limitations above 1000°C. SETARAM's THEMYS thermal analyzers offer an interesting alternative in the form of TRI-COUPLES DTA sensors. These sensors are designed with **3 thermocouple junctions** under each crucible instead of just one for standard DTA. They also use less material than a DSC sensor and are therefore less subject to parasitic radiative effects at high temperatures.
As a result, this design considerably improves the amount of heat collected and the sensitivity of the sensor.

Low energy phenomena at high temperature can be accurately measured with this sensor. For example, **zirconia solid-solid phase transition (Monoclinic - Tetragonal) around 1200°C** has been tested and compared with standard DSC sensor.

EXPERIMENT

The following experimental procedure has to be used:

- Sample: Zircone powder
- Mass: 83mg (DTA) 87mg (DSC)
- Reference: Empty Al_2O_3 crucible
- Heating: 800 – 1500°C at 10K.min^{-1}
- Apparatus: THEMYS with Type S tri-couple DTA or DSC sensor
- Atmosphere: Air at 20ml.min^{-1}

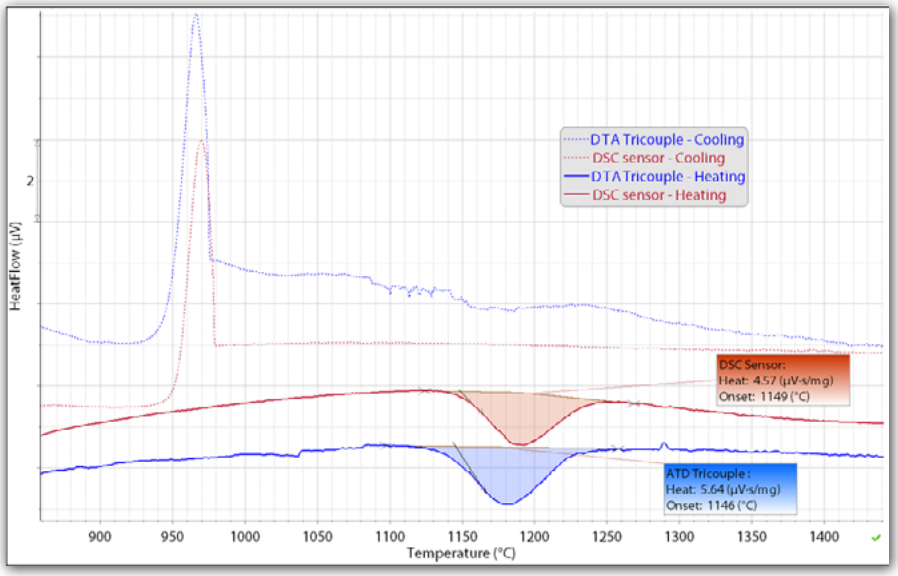

Figure 1 : DTA curve of zirconia sample between 850°C and 1450°C, measured with DSC and DTA Tri-couple sensor

RESULTS AND CONCLUSION

Upon heating, an endothermic effect is measured between 1125°C and 1225°C. It corresponds to the solid-state transition from monoclinic to tetragonal. While it is observed with both DTA Tri-couple and DSC sensor, **the sensitivity is 23% higher with the DTA Tri-couple.** Therefore, this sensor design allows a better measurement of thermal phenomena at high temperature. Recrystallization to monoclinic structure is also measured upon cooling, at much lower temperature.

INSTRUMENT

THEMYS STA

ACCURATE AND SENSITIVE ULTRA-HIGH TEMPERATURE
Heat flow measurement with Tri-Couple DTA technology

ULTRA-HIGH TEMPERATURE CAPABILITY
to *2400°C* with a single furnace.

MODULAR ADAPTIONS ALLOWING
TGA only, DTA only, TG-DTA, andTMA up to 2400°C, DSC only and TG-DSC up to 1600°C all in one instrument

EXTERNAL COUPLING CAPABILITY
designed for evolved gas analyzers (FTIR, MS, GCMS, MSFTIR, or FTIR-GCMS)