

CERAMICS CHARACTERIZATION



THERMAL ANALYSIS, CALORIMETRY & GAS SORPTION SOLUTIONS

- OXYGEN RATIO & STOICHIOMETRY •
- PHASE DIAGRAM •
- SINTERING •
- THERMOPHYSICAL PROPERTIES •
- HEAT OF FORMATION •

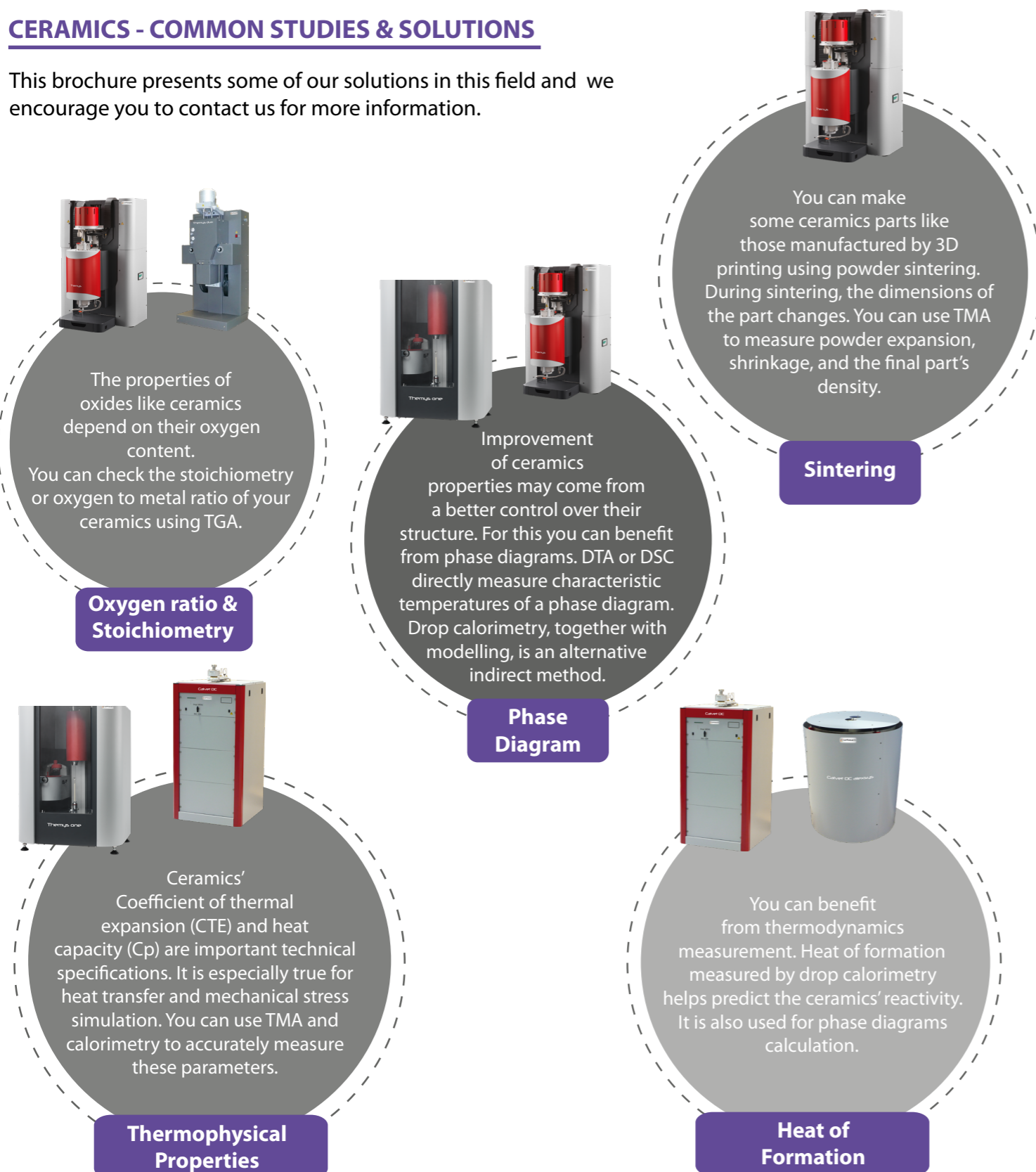
YOUR CHALLENGES

Improving ceramic materials and their production processes is a major concern for their developers and manufacturers, especially in today's competitive and constantly changing environment. This requires technically reliable and cost-effective characterization solutions.

If you are involved in studying the structure or reactivity of ceramics, particularly if they are technical or refractory ceramics, we have solutions to help you with : determining phase diagrams, oxygen ratios, measuring data for heat transfer simulation and understanding powders sintering.

CERAMICS - COMMON STUDIES & SOLUTIONS

This brochure presents some of our solutions in this field and we encourage you to contact us for more information.



CUSTOMER TESTIMONIAL

"The complementary calorimetric approach proved to be a useful tool in addition to dilatometry in order to provide further information, including transformation energies besides it is a technique which is faster than the traditional ones."

Thomaz Augusto Guisard Restivo, University of Sorocaba, Brazil.
Nuclear Engineering and Design, Volume 265, 2013, Pages 619-624

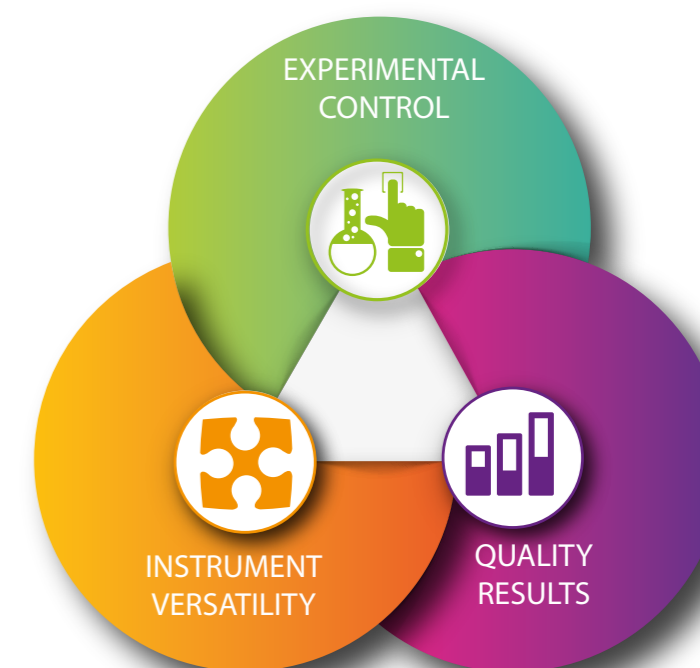
THE KEP TECHNOLOGIES ADVANTAGE

KEP Technologies is addressing its offerings to the metals market by making available the widest and most versatile choice of solutions. Now you can consult with one company, KEP Technologies, to address your challenges across the broadest number of battery studies on the market.

Each solution embodies our "Reimagine Material Characterization" value proposition by delivering the three core customer benefits of **Experimental Control, Instrument Versatility and Quality Results.**

We believe solutions that provide these benefits will deliver the highest value to our customers.

In addition to our core customer benefits, we are able to provide **customized solutions** by harnessing the engineering and project management of our highly skilled organization.




CUSTOMIZED SOLUTIONS

Modular design allows for upgraded and tailored functionality
Access to all previous non-proprietary custom requests
Open access to our engineering development team

PHASE DIAGRAM

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 ADAPPTIONS ALLOWING**
TGA only, DTA only, TG-DTA, and TMA up to 2400°C, DSC only and TG-DSC up to 1750°C all in one instrument
- HIGH ACCURACY & VERSATILITY**
hang-down symmetrical beam balance, specifically designed for TGA applications
- EXTERNAL COUPLING CAPABILITY**
designed for evolved gas analyzers (FTIR, MS, GCMS, MS-FTIR, or FTIR-GCMS)

SPECIFICATIONS

Temperature range (°C)	room temperature to 1750 or to 2400
Isothermal and temperature scanning (°C/min)	0.01 to 100
Sample volume (µl)	up to 2500 in TGA
Optional protected DTA rods for enhanced corrosion resistance, tricouple DTA rods for enhanced sensitivity, protected tricouple for combined advantages	

For more information on specifications please consult the product information and brochures available on our website : www.setaramsolutions.com

APPLICATION

Characterization of Phase Transitions in Yttria and Zirconia in the 2000°C to 2400°C range.

INTRODUCTION

High-temperature differential thermal analysis provided data on phase transitions in zirconia and yttria. The tetragonal form of ZrO_2 transforms to the cubic fluorite structure at $2311 \pm 15^\circ C$ with an enthalpy of 3.4 ± 2 kJ/mol. Cubic C-type Y_2O_3 transforms, probably to the fluorite structure, at $2308 \pm 15^\circ C$ with $\Delta H = 47.7 \pm 3.0$ kJ/mol. This high-temperature polymorph melts at $2382 \pm 15^\circ C$ with an enthalpy of fusion of 35.6 ± 3.0 kJ/mol.

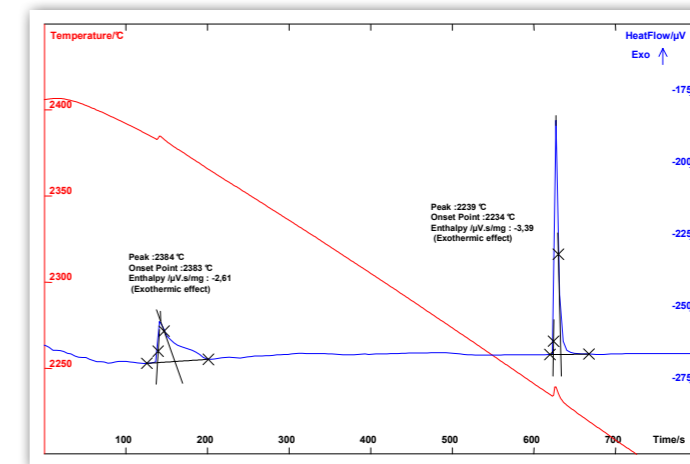


Figure 1 - Cooling curve for Y_2O_3

EXPERIMENT

Crucibles made of tungsten were used. Temperature and sensitivity were calibrated before each experiment by performing the melting of alpha alumina in a tungsten crucible at a heating rate of $10^\circ C/min$ under helium (melting temperature of $2052^\circ C$ and enthalpy of fusion of 1092 J/g). For zirconia analysis, a calibration was also performed afterwards. It was then observed that the temperature correction increased by $50^\circ C$. This variation is mainly because of the aging of the thermocouple during the heating at $2400^\circ C$. Each sample was scanned upwards in temperature from 1600° to $2400^\circ C$ at a rate of $10^\circ C/min$ (heating run), and then the scanning direction was reversed (cooling run).

RESULTS AND CONCLUSION


In general, the cooling runs gave clearer signals than the initial heating runs, probably because the sample, after heating, made better thermal contact with the pan. As an example, Fig. 1 shows a cooling scan for yttria. For zirconia, one transition, namely that of the tetragonal to the cubic phase, could be clearly seen, three runs showing peaks at $2265^\circ C$, $2278^\circ C$ and $2311^\circ C$.

Recommended Values of Thermodynamic parameters for Transitions in ZrO_2 and Y_2O_3

Material	Transition	T (°C)	ΔH (kJ/mol)	ΔS (J/mol/K)
ZrO_2	m-t	1199	5.43 ± 0.31	3.69 ± 0.21
ZrO_2	t-c	2311	3.4 ± 2.1	1.3 ± 0.8
Y_2O_3	c-c	2308	47.7 ± 3.0	18.5 ± 1.2
Y_2O_3	c-m	2382	35.6 ± 3.2	13.9 ± 1.2

Reference: Direct Calorimetric Measurement of Enthalpies of Phase Transitions at $2000^\circ C$ – $2400^\circ C$ in Yttria and Zirconia. Alexandra Navrotsky, Luc Benoist and Herve Lefebvre. J. Am. Ceram. Soc., 88 [10] 2942–2944 (2005)

INSTRUMENT



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APPLICATION

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: Zirconia powder
- Mass: 83mg (DTA) 87mg (DSC)
- Reference: Empty Al₂O₃ 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

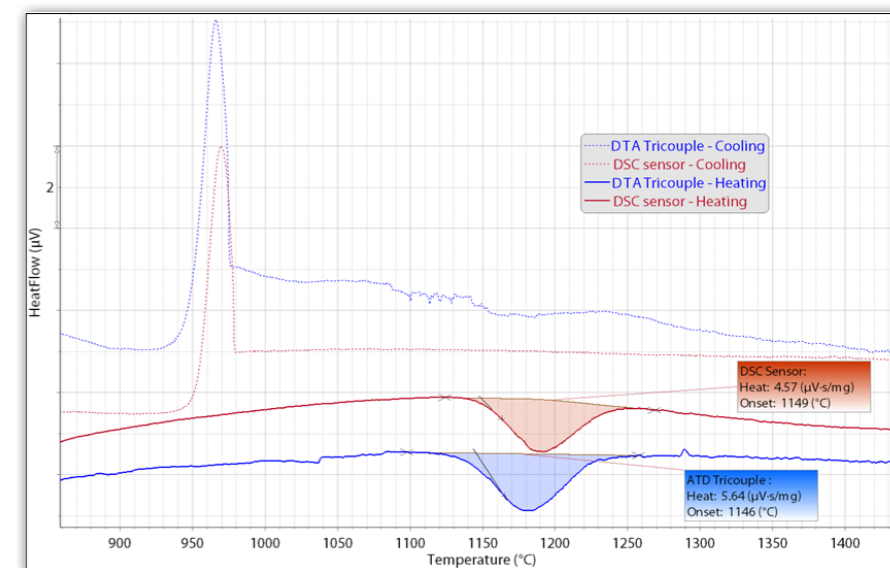


Figure 1 : DTA curve of zirconia sample between 850°C and 1450°C, measured with DSC and DTA Tri-couple sensor

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 TMA



PRESERVATION OF SAMPLES
due to low load vertical TMA system

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

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

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

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

SPECIFICATIONS

Temperature range (°C)	Ambient to 2400
Programmable heating rate (°C/min)	0.01 to 100
Maximum sample size (mm)	Height : 20 Diam : 10
Resolution (nm)	0.2
Measuring range (mm)	+/- 2

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APPLICATION

Sintering of Al₂O₃ with control of the shrinking rate

INTRODUCTION

Because during sintering the dimension of the ceramic material changes drastically, it is important to control this variation to have a final product with the required dimensions and properties. TMA provides information about volume shrinkage, powder grain growth, and their interactions. The THEMYS TMA enables the control of the sintering rate during the experiment, thus providing valuable information about the temperature profile to be applied to the industrial sintering furnace.

EXPERIMENT

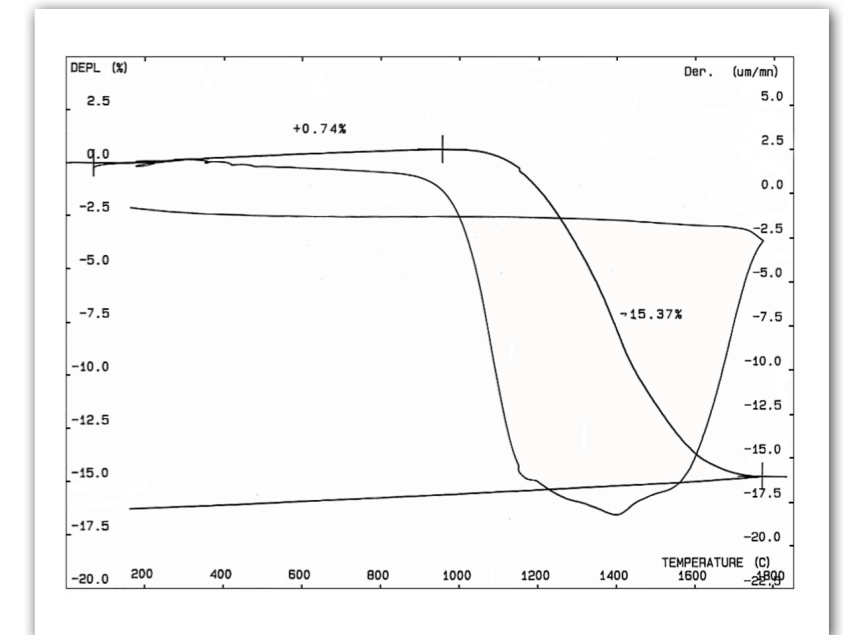
A cylindrical sample of alumina (Diameter=10 mm, height=20 mm) is heated with a heating rate of 5K/min up to 1780°C.

Alumina probe.

Carrier gas : helium.

Maximum shrinking rate = 20 µm/min.

Use of software package to control the shrinking rate.



RESULTS AND CONCLUSION

When the sample of alumina is heated at 5 K/min continuously a shrinkage due to the sintering of about 15% is observed. The maximum of shrinking rate is about 60 µm/min it means that the heating rate (in K/min) will be reduced in order to have a shrinking rate <20 µm/min.

The main interest of the method is to be able to determine in one run the temperature program for sintering at constant shrinking rate.

INSTRUMENT

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technology

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SPECIFICATIONS

Temperature range (°C)	Ambient to 2400
Programmable heating rate (°C/min)	0.01 to 100
Maximum sample size (mm)	Height : 20 Diam : 10
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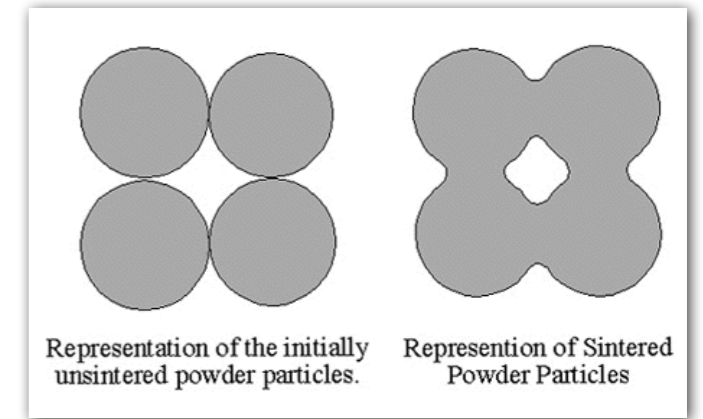
APPLICATION

Sintering of Silicon Carbide (SiC)

INTRODUCTION

Sintering is a process for making objects from powder, increasing the adhesion between particles as they are heated. It is used with ceramic powders and in powder metallurgy.

For example, the most convenient method for producing dense SiC is pressureless sintering. Due to the strongly covalent character of the Si-C chemical bond and hence slow diffusion kinetics, the addition of sintering enhancers is necessary, if a high degree of densification is required. The most frequently used enhancer combinations, B+C or Al+C, allow for sintered densities above $\geq 97\%$ of the theoretical value.



EXPERIMENT

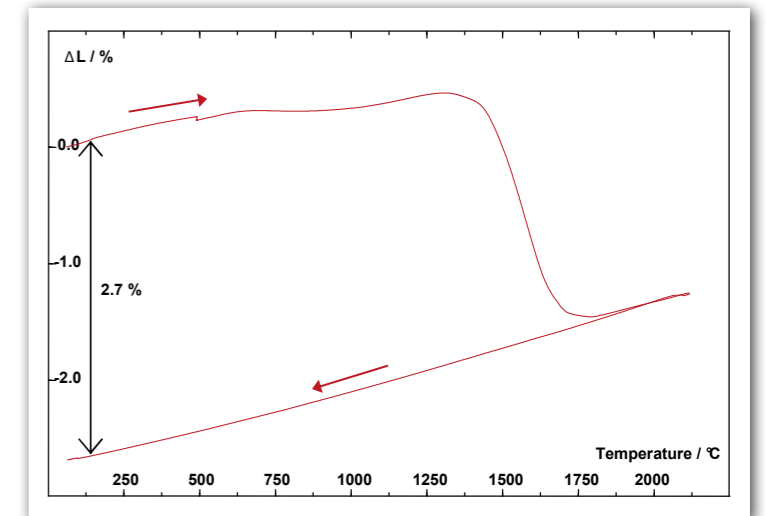
The sample was a precompacted α -SiC powder cube with the following dimensions:
9 mm x 9 mm x 9 mm

It was analyzed under argon and the applied load was 5g.

The following temperature program was used:

- 20°C up to 480°C at 10 K/min and maintained during 3 hours at this temperature.
- 480°C up to 2200°C at 15 K/min.
- 2200°C down to ambient temperature at 15K/min.

The experiment curve was corrected from a blank carried without sample and in the same experimental conditions.



RESULTS AND CONCLUSION

The experimental curve presents a global expansion between ambient and 1310°C.

Above 1310°C, shrinkage is noticed : it is due to the sintering of SiC. And it is completed at 1815°C.

Above 1815°C, the expansion of SiC is observed.

During cooling, a shrinkage of sintered SiC is observed. The global shrinkage after cooling back to ambient is 2,7%.

INSTRUMENT

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SPECIFICATIONS

Temperature range (°C)	Ambient to 2400
Programmable heating rate (°C/min)	0.01 to 100
Maximum sample size (mm)	Height : 20 Diam : 10
Resolution (nm)	0.2
Measuring range (mm)	+/- 2

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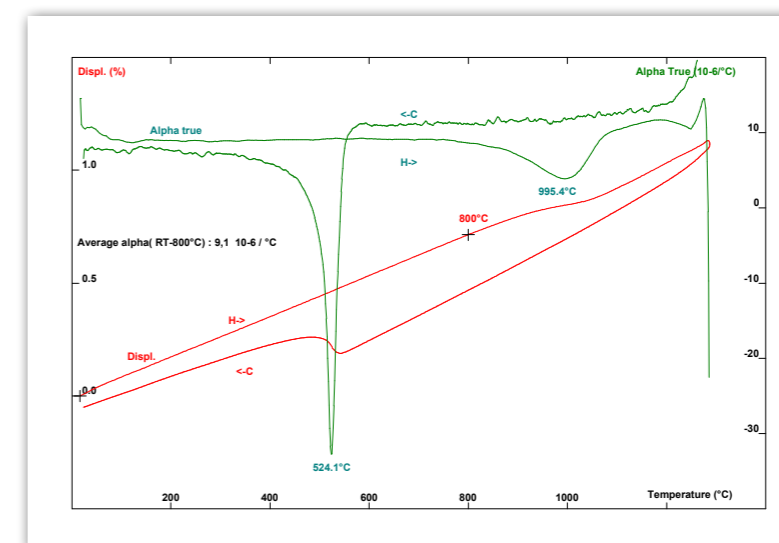
APPLICATION

Thermal expansion of Zirconia(SiC)

INTRODUCTION

Under the effects of increasing temperature any material will expand. This can lead to significant changes in dimensions of ceramic parts when heated. The Coefficient of Thermal Expansion (CTE) is a thermophysical property which characterizes the ability of a material to expand under the effect of temperature elevation. It tells you how much the ceramic will remain dimensionally stable under temperature.

The thermal expansion difference between parts that are in contact in a complex structure may develop internal stresses and stress concentrations, leading to premature failure to occur. Hence, CTE is important for quality and functioning of any structure that may be heated during its cycle life.



EXPERIMENT

Sample : Zirconia.
Sample length : 19.90 mm.
Probe : alumina, flat ended.
Gas : helium.
Heating from 20°C up to 1300°C at 1.5 K/min.
Cooling from 1300°C down to 20°C at 0.5 K/min.
The same experiment without sample was also carried out (blank).

RESULTS AND CONCLUSION

During the heating the average coefficient of expansion between 20°C and 800°C is 9.1.10-6/°C. This result was obtain after applying the standard correction of the probe and sample holder tube expansions, and of the instrumental drift.

THERMOPHYSICAL PROPERTIES

INSTRUMENT

THEMYS ONE



HIGH SENSITIVITY BALANCE FOR THE DETECTION OF SMALL MASS VARIATIONS specifically designed for TGA analysis.

CONVENIENCE OF ONE FURNACE to reach temperatures as high as 1600°C.

PLUG AND PLAY INTERCHANGEABLE RODS to perform TGA only, TG-DSC, TG-DTA, and 3D high sensitivity/Cp measurements.

EXTERNAL COUPLING CAPABILITY including evolved gas analyzers

SPECIFICATIONS

Temperature range (°C)	room temperature to 1600
Isothermal and temperature scanning (°C/min)	0.01 to 100
Sample volume (ml)	up to 1 in TGA
Evolved gas analyzers (FTIR, MS, GCMS, MS-FTIR, or FTIR-GCMS) for performing qualitative and quantitative gas characterization	

For more information on specifications please consult the product information and brochures available on our website : www.setaramsolutions.com

APPLICATION

Heat capacity of ceramics up to high temperatures

INTRODUCTION

Ceramics are generally exposed to high temperatures during their manufacturing process or their applications. Therefore, controlling the heat capacity of ceramic precursor materials and the final ceramics is very important to guarantee the performance of the manufacturing process, and their resistance against thermal ageing. It is measured by DSC, calorimetry or microcalorimetry.

EXPERIMENT

361 mg of alumina nanopowder is used for the Cp determination from room temperature to 1400°C with the continuous method at 20°C/min. The sapphire calibration is used for this determination.

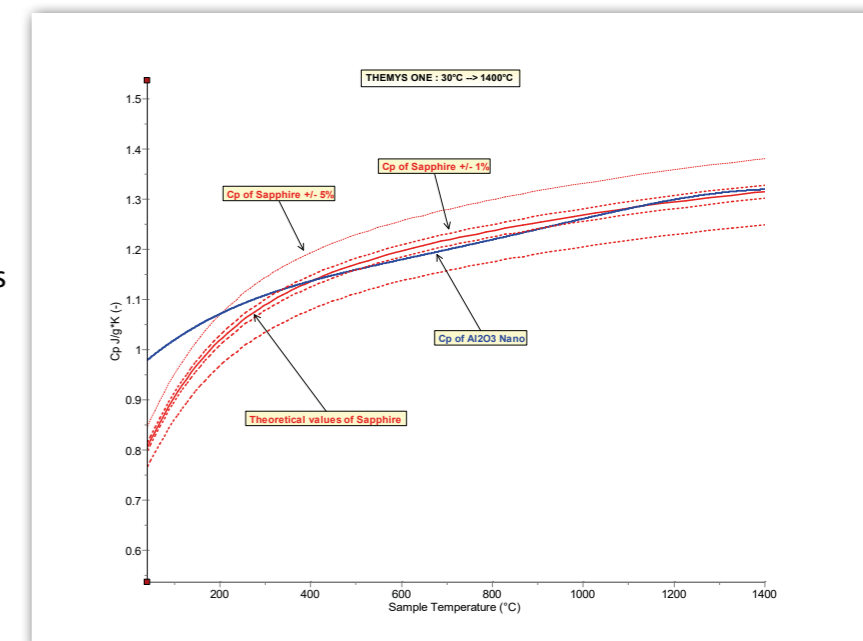



Figure 1: Cp determination of alumina nanopowder using the continuous heating mode

RESULTS AND CONCLUSION

Above 300°C, the Cp of alumina nanopowder stays within ±1% of the sapphire Cp interval up to 1400°C. Two types of confidence intervals are drawn on Figure 1 at ±1% and ±5% of the sapphire Cp value.




OXYGEN RATIO & STOICHIOMETRY

INSTRUMENT



THEMYS TGA

- **VARIETY OF ATMOSPHERE CONDITIONS**
multiple carrier and reactive gas options
- **MODULAR ADAPTIONS ALLOWING**
TGA only, DTA only, TG-DTA, and TMA up to 2400°C, DSC only and TG-DSC up to 1750°C all in one instrument
- **HIGH ACCURACY & VERSATILITY**
hang-down symmetrical beam balance, specifically designed for TGA applications
- **EXTERNAL COUPLING CAPABILITY**
designed for evolved gas analyzers (FTIR, MS, GCMS, MS-FTIR, or FTIR-GCMS)

SPECIFICATIONS

Temperature range (°C)	room temperature to 1750 or to 2400
Isothermal and temperature scanning (°C/min)	0.01 to 100
Sample volume (µl)	up to 2500 in TGA
Optional protected DTA rods for enhanced corrosion resistance, tricouple DTA rods for enhanced sensitivity, protected tricouple for combined advantages	

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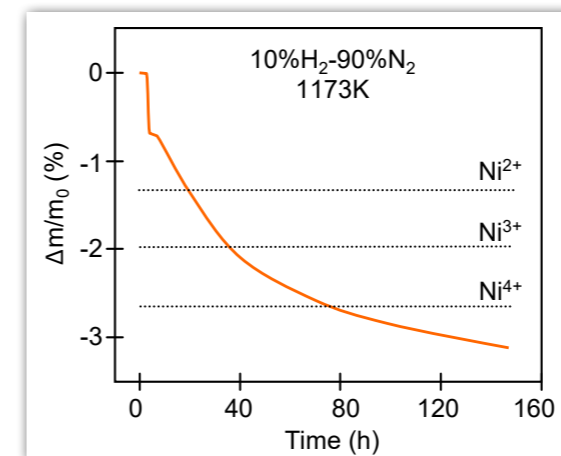
APPLICATION

Oxygen stoichiometry of $\text{LaGa}_{0.65}\text{Mg}_{0.15}\text{Ni}_{0.20}\text{O}_{3-d}$

INTRODUCTION

LaGaO_3 -based solid electrolytes and mixed ionic–electronic conductors attract significant attention during the last decade due to their potential application as materials of intermediate-temperature solid oxide fuel cells (ITSOFCs), electrochemical oxygen sensors, and membrane reactors for conversion of natural gas to synthesis gas.

In particular, a very high level of ionic transport is observed for $\text{La}_{1-x}\text{Sr}_x\text{Ga}_{1-y}\text{Mg}_y\text{O}_{3-d}$ (LSGM) solid solutions with $x=0.10-0.20$ and $y=0.15-0.20$. The aim of this paper is to estimate oxygen stoichiometry from the weight changes by thermogravimetric analysis.



Example of reduction kinetics of $\text{LaGa}_{0.65}\text{Mg}_{0.15}\text{Ni}_{0.20}\text{O}_{3-d}$ in flowing 10% H_2 -90% N_2 mixture at 1173 K. The sample was kept for 2h in air, 1h in argon, and then 144h in the H_2 -containing mixture. Dashed lines correspond to the theoretical weight changes upon reduction into metallic nickel and binary oxides, calculated assuming that there is no gallium oxide volatilization and that all nickel cations in air are in 2+, 3+ or 4+ oxidation states.

EXPERIMENT

Estimation of oxygen stoichiometry of $\text{LaGa}_{0.65}\text{Mg}_{0.15}\text{Ni}_{0.20}\text{O}_{3-d}$ ceramic was carried out in a Setsys TGA. The following program is used:

- Heating at 3K/min in flowing air with equilibration steps at 1073, 1123 and 1173K for 2h at each temperature;
- Flushing of the apparatus with argon for 1h;
- Reduction at 1173K in flowing 10% H_2 -90% N_2 mixture (cf. figure);

RESULTS AND CONCLUSION

The values of oxygen nonstoichiometric in air at 1073-1173K, calculated from the TG data, are listed in the table below:

T (K)	δ	Average Ni oxidation state
1173	0.080	2.95
1123	0.070	3.05
1073	0.062	3.13

The average oxidation state of nickel cations is +2.95 at 1173 K, and increases up to +3.13 on cooling down to 1073K. This indicates coexistence of Ni^{2+} , Ni^{3+} and Ni^{4+} states in the lattice of $\text{LaGa}_{0.65}\text{Mg}_{0.15}\text{Ni}_{0.20}\text{O}_{3-d}$ under oxidizing conditions. The formation of tetravalent nickel should still be understood as a hypothesis and requires additional experimental confirmation, particularly to verify exact location of the electron holes formed due to oxygen incorporation.

HEAT OF FORMATION

INSTRUMENT

CALVET DC ALEXSYS



BEST IN CLASS HIGH TEMPERATURE MEASUREMENTS BY DROP CALORIMETRY

improved heat capacity, heat of phase transitions, heat content measurements up to 1000 °C

HIGHLY SENSITIVE HIGH TEMPERATURE CALVET 3D SENSOR

dual drop capability with a measurement drop tube and a reference drop tube

FINE CONTROL OF THE SAMPLE REACTIVITY

preserved sensor lifetime with an Inconel protective tube, a chemically inert silica based drop tube with optional platinum liner, control of the atmosphere above the sample.



SPECIFICATIONS

CALVET DC ALEXSYS	
Temperature range (°C)	500 to 800 or 1000°C
Temperature stability (°C)	+/- 0.2
Calorimetric precision (%)	+/- 1%*
Cells volume (ml)	Up to 28 ml (quartz cell) or 20ml (platinum crucible)

*Based on sapphire drop

For more information on specifications please consult the product information and brochures available on our website : www.setaramsolutions.com

APPLICATION

Heat of formation of an oxide (Bi₂WO₆)

INTRODUCTION

Drop calorimetry is a technique designed for measuring the enthalpy change of a sample of material when it is dropped from room temperature into a calorimeter at a high temperature. If the calorimeter's cell contains a solvent, then the heat of dissolution of the material contributes to the measured enthalpy change. By running a series of dissolution tests, as seen on Figure 1, the heat of formation can be calculated.

- Step 1: Measurements of heats of dissolution of the studied components in a single selected solvent
- Step 2: Calculation of heat of formation by combining the measured heats of dissolution obtained in step 1

EXPERIMENT

- Instrument : CALVET DC ALEXSYS
- Samples : Bi₂WO₆ and its precursors Bi₂O₃ and WO₃
- Drop solution method at 983 K
- Na₂O + MoO₃ as a liquid solvent at the test temperature

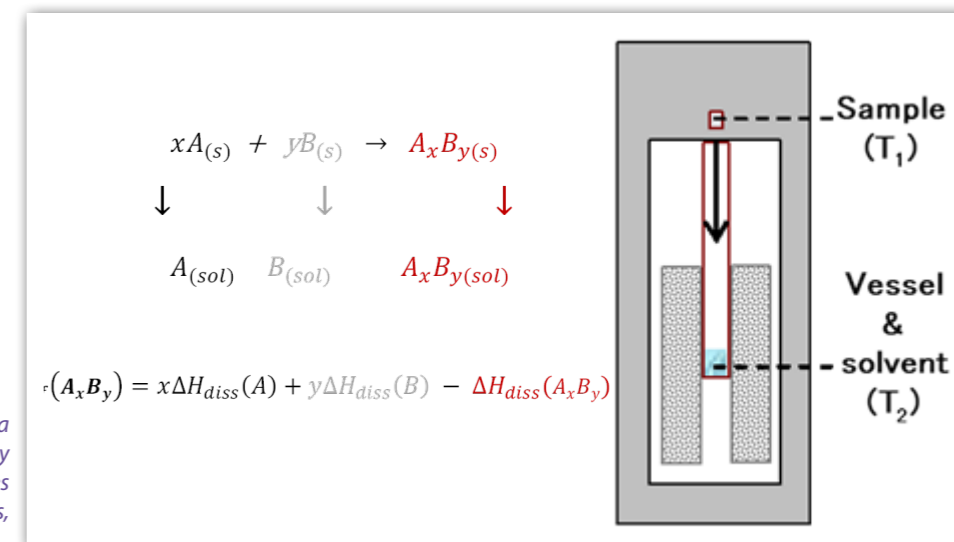


Figure 1 – Calculation of the heat of formation of a material using the drop method. The formula A_xB_y is given as an example but the method applies multiple materials including to intermetallics, oxides, carbides, etc...

RESULTS AND CONCLUSION

As seen on Figure 2, the integrations of the peaks on the heat Flow signal allow for the determination of the heats of dissolution. Additionally, the heat of oxidation of Bi and W into Bi₂O₃ and WO₃ is found in literature.

The reaction scheme showed below in Figure 3, the literature and experimental data allow for the calculation of the enthalpy of formation of Bi₂WO₆ from Bi, W and O.

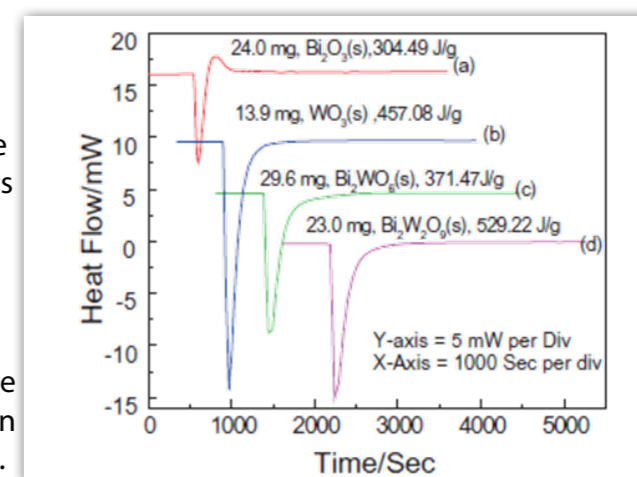


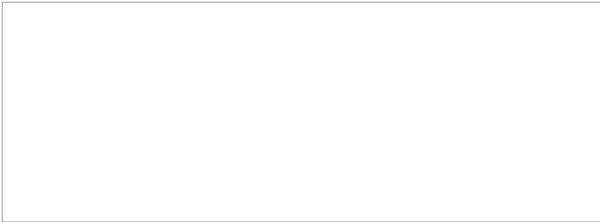
Figure 2 - Drops of Bi and W oxides at 983 K

Reaction scheme for the standard molar enthalpy of formation of Bi₂WO₆(s) (M(sln) = dilute solution of species M in 3 g liquid Na₂O + MoO₃ 3:4 M solvent maintained at 983 K, $\Delta_f H_{298}^\circ = -\Delta H_1 + \Delta H_2 + \Delta H_3 + \Delta H_4 + \Delta H_5$)

Reaction	ΔH_i	ΔH_i (kJ · mol ⁻¹)	Reference
Bi ₂ WO ₆ (s, 298 K) + (sln, 983 K) = Bi ₂ O ₃ (sln, 983 K) + WO ₃ (sln, 983 K)	ΔH_1	118.93 ± 5.83	This work
Bi ₂ O ₃ (s, 298 K) + (sln, 983 K) = Bi ₂ O ₃ (sln, 983 K)	ΔH_2	52.48 ± 4.82	This work
WO ₃ (s, 298 K) + (sln, 983 K) = WO ₃ (sln, 983 K)	ΔH_3	43.07 ± 2.18	This work
2 Bi(s, 298 K) + 3/2 O ₂ (g, 298 K) = Bi ₂ O ₃ (s, 298 K)	ΔH_4	-574.00 ± 0.80	Ref. [12]
W(s, 298 K) + 3/2 O ₂ (g, 298 K) = WO ₃ (s, 298 K)	ΔH_5	-828.60 ± 0.13	Ref. [12]
2Bi(s, 298 K) + W(s, 298 K) + 3O ₂ (g, 298 K) = Bi ₂ WO ₆ (s, 298 K)	$\Delta_f H_{298}^\circ$	-1426.0 ± 7.9	This work

Figure 3 - Reaction scheme for the standard molar enthalpy of formation of Bi₂WO₆(s), $\Delta_f H_{298}^\circ = -\Delta H_1 + \Delta H_2 + \Delta H_3 + \Delta H_4 + \Delta H_5$

Reference: Phapale, Das, Mishra, J. Chem. Thermodynamics 63 (2013) 74–77



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