KEP Technologies

METALS
CHARACTERIZATION

THERMAL ANALYSIS , CALORIMETRY & GAS SORPTION SOLUTIONS

• OXIDATION & CORROSION •
• PHASE DIAGRAM •
• SINTERING •
• THERMOPHYSICAL PROPERTIES •
• HEAT OF FORMATION •

setaram
KEP TECHNOLOGIES

YOUR CHALLENGES

The improvement of metals materials is a prime concern for developers and manufacturers. In a competitive and rapidly developing environment, you need technically reliable and economically profitable characterization solutions.

If you are involved in investigating the structure or reactivity of metals and alloys, we have the solution to help. Assess stability against corrosion. Determine phase diagrams, data for heat transfer simulation in metals-based systems. Understand powders sintering.

COMMON CATALYSTS - STUDIES & SOLUTIONS

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

The stability against corrosion and oxidation is an important criterion. You can study high temperature oxidation of metals and alloys using TGA. You can even simulate harsh conditions involving humidity, pressure, acidic vapors, etc.

Oxidation & Corrosion

Improvement of metal and alloy properties may come from a better control over their structure. For this you can benefit from phase diagrams. DTA or DSC directly measure characteristic temperatures of a phase diagram. Drop calorimetry, together with modelling, is an alternative indirect method.

Phase Diagram

You can make some metallic parts like those manufactured by 3D printing using powder sintering. During sintering, the dimensions of the part changes. You can use TMA to measure powder expansion, shrinkage, and the final part's density.

Sintering

A metals' Coefficient of thermal expansion (CTE) and heat capacity (Cp) are important technical specifications. It is especially true for heat transfer and mechanical stress simulation. You can use TMA and calorimetry to accurately measure these parameters.

Thermophysical Properties

Interested in the stability of complex alloys? You can benefit from thermodynamics measurement. Heat of formation measured by drop calorimetry helps predict the alloys' reactivity. It is also used for phase diagrams calculation.

Heat of Formation

CUSTOMER TESTIMONIAL

"Setaram's THEMYS FLASH enables the simultaneous and continuous evaluation of solid-gas reaction rates for 5 samples, without external manipulation and under thermal cycling conditions. This device makes it possible to multiply the number of samples tested under the same conditions, and to make the results of a study more reliable. Aspects relating to stress development during thermal cycling can be taken into account quantitatively.

The first study carried out compared nickel-based alloys for high-temperature applications, produced by either conventional or additive manufacturing processes. Under the conditions tested, some alloys produced by additive manufacturing showed scaling effects already after 400 cycles."

LIONEL ARANDA, research engineer, Surface and Interface, Chemical Reactivity of Materials Team
"Institut Jean Lamour - Université de Lorraine" - Nancy, France

THE KEP TECHNOLOGIES ADVANTAGE

KEP Technologies is addressing it's 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

REIMAGINE MATERIAL CHARACTERIZATION

OXIDATION & CORROSION

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	

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

APPLICATION

Long term high temperature corrosion measurements

INTRODUCTION

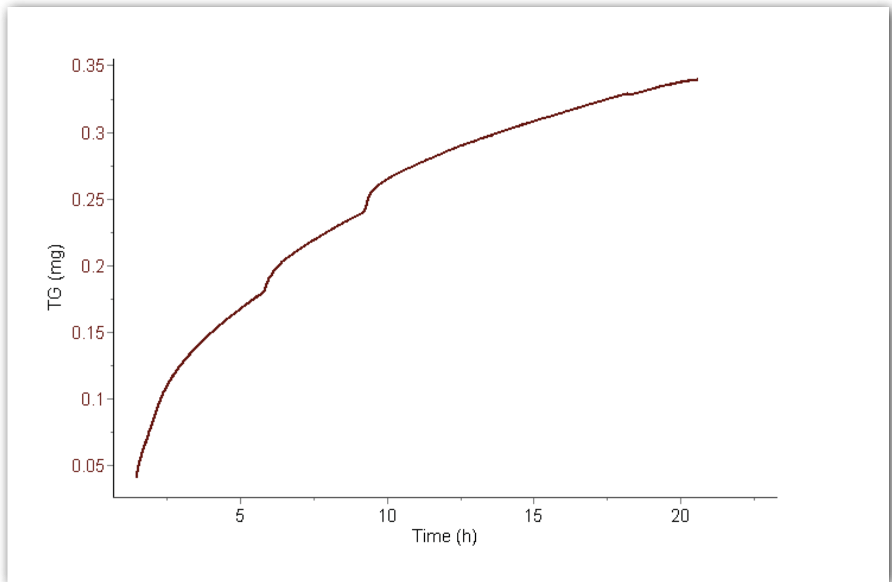
High temperature, corrosion resistant superalloys need to be characterized in terms of their oxidation kinetics.
Thermogravimetric analysis (TGA) is the perfect tool for tool to get the full picture of the oxidation behavior.
These characterizations require the TGA instrument to provide a good gas-solid interface, a high mass signal stability over long periods of time and a efficient control of the sample atmosphere. These requirements are met by THEMYS.

EXPERIMENT

A 53.43 mg and 75.6 mm2 INCONEL 600 sample was directly hung at the TGA suspensions so that it was centered in the furnace. The THEMYS was equipped with the High Sensitivity balance.
The sample was heated up to 900 °C at 10 °C/min under a flow of nitrogen. At 900 °C, nitrogen was switched to oxygen at a flow rate of 20 ml/min during 20 hours.
The TG signal, or mass uptake due to oxidation was measured from that point.

RESULTS AND CONCLUSION

Figure 1 shows the mass uptake signal over time during the 20 hours isothermal sequence. The total mass gain of the sample was no more than 298.5 µg, i.e. 0.395 mg/cm2. It demonstrate the ability of the TGA instrument to measure small effect over time thanks to the combination of its high sensitivity and stability.
The bumps on the mass uptake curve at about 6 and 10 hours are linked with the formation of cracks in the oxide layer resulting in the exposure of new metallic surface and leading to sudden increase of the oxidation rate. A continuous signal as it is provided by TGA is required to detect such subtle changes in the material behavior.
The excellent signal stability and the possibility to hang samples directly in the furnace without using crucibles as it is possible with THEMYS is beneficial for such applications.



INSTRUMENT

THEMYS FLASH

MULTIPLE SIMULTANEOUS MEASUREMENTS
with flexible balance integrating up to 5 weighing modules

HIGH ACCURACY & VERSATILE
hang-down symmetrical beam balance specifically designed for TGA applications

FAST HEATING AND COOLING
thanks to its unique design of image furnace

A VARIETY OF ATMOSPHERE CONDITIONS
with the possibility of operating under oxidative gas, inert gas, or vacuum

SPECIFICATIONS

Temperature range (°C)	Ambient to 1200
Programmable heating rate (°C/min)	up to 600
Cooling time	<1 min from 1200 to 1000°C 15 min from 1000 to 200°C 15 min from 200 to 70°C
Maximum sample size	15x12 mm (rectangular samples) 12 mm diameter (disks)
Multi-modules balance	1 to 5 weighing modules, to be defined when ordere with further upgrade possible
Maximum loading capacity (g)	35

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

APPLICATION

High temperature oxidation resistance of thermal barrier coating systems

INTRODUCTION

The protection of certain metal parts against the effects of temperature and corrosion may require the development of complex strategies such as thermal barrier coating (TBC). These oxide coatings aim at slowing down the oxidation phenomenon but do not prevent it completely and cause other effects such as cracking of the coatings or spalling.
The study of the oxidation kinetics of metallic alloys at high temperature is frequently performed using thermogravimetric analysis (TGA), which follows the mass increase of samples exposed to the desired temperature and atmosphere conditions.
THEMYS FLASH is a TGA equipment designed to best approximate the rapid and cyclic heating and cooling conditions of parts requiring TBC protection.

EXPERIMENT

- Instrument : THEMYS FLASH
- Sample: AM3 nickel-based superalloy + Thermal Barrier
Coating composed of a NiPtAl bound coating and a ZrO_2 top coating.
Preoxidized during 20 h at 900 °C.
- Atmosphere: Air at a flow rate of 1l/h.
- The temperature profile was composed of a series of heating and cooling cycles with four phases as follows:
1. Heating at 140 °C/min up to 1100 °C,
2. A high temperature dwell time of 1 h at 1100 °C,
3. Cooling down to 200 °C at 300 °C/min,
4. A 15 min hold time at 200 °C.

RESULTS AND CONCLUSION

The mass change due to oxidation is very small, of the order of magnitude of 30µg/cm2/cycle during the first cycles. It is still measurable thanks to the high sensitivity of the equipment. Starting at cycle 202, spalling effects were detected at various stages of the process (marked by *), but they corresponded to small percentages of the total mass of zirconia top coating (0.01–0.3%).

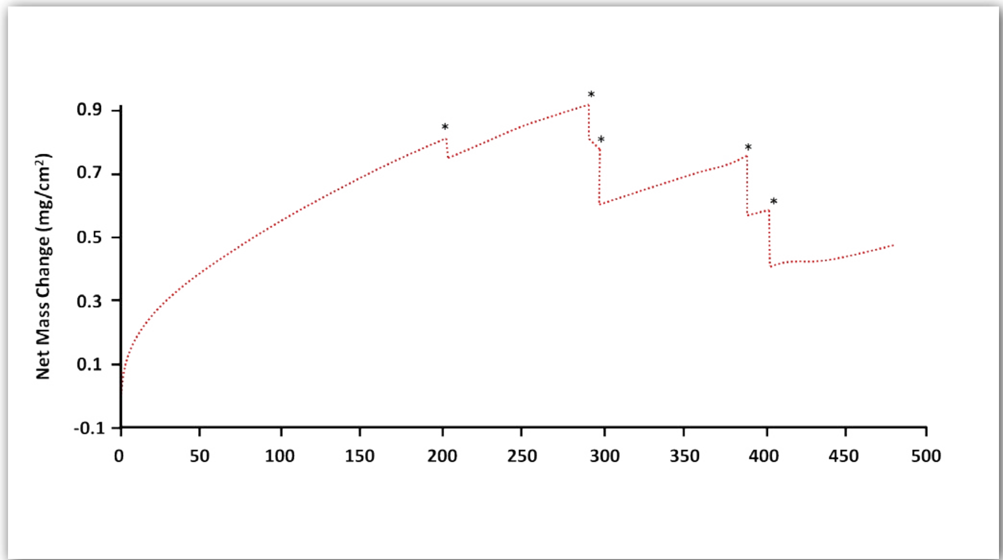


Figure 1 – Net mass change as a function of the cycle number. Stars (*) mark the cycles during which spalling effects occurred.

Reference : A. Vande Put et al., Cyclic thermogravimetry of TBC systems, Surface & Coatings Technology 202 (2007) 665–669.

INSTRUMENT

THEMYS DUO

- **ULTRA-HIGH TEMPERATURE CAPABILITY**
  to 1750 °C with the same dual furnace
- **HIGHEST ACCURACY WITH ITS HANG-DOWN SYMMETRICAL BEAM BALANCE**
  eliminate drift & buoyancy effect, improve gas/ sample interaction
- **MODULAR ADAPTATIONS ALLOWING**
  up to 1750 °C: TGA, DTA, TG-DTA
  up to 1600 °C: DSC, TG-DSC
- **ACCURATE AND SENSITIVE**
  Tri-couple DTA technology
- **VARIETY OF ATMOSPHERE CONDITIONS**
  multiple carrier and reactive gas options
- **EXTERNAL COUPLING CAPABILITY**
  with evolved gas analyzers

SPECIFICATIONS

	TGA	DTA, TG-DTA	DSC, TG-DSC
Temperature range (°C)	Ambient to 1750	Ambient to 1750	Ambient to 1600
Programmable heating rate (°C/min)	0.01 to 100		
Crucibles volumes and aximum sample size	55 to 1 500 µl or Height: 20 Diam: 14 mm without crucible	20 to 300 µl	75 to 110 µl
Vacuum	Primary (< 1 mbar), forced primary (< 5.10-2 mbar) options		

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

APPLICATION

Cyclic oxidation of metallic alloys by symmetrical TGA

INTRODUCTION

Metallic alloys are frequently subjected to combined environmental attack and mechanical stresses during their actual service life. Their resistance to this complex loading relies partly on their ability to form a protective oxide scale, i.e. an oxide layer with low growth kinetics and high adherence to the alloy. The nature and kinetics of the growth of the oxide layer is , often studied using isothermal laboratory tests. When testing the resistance to oxidation of hightemperature materials, the cyclic-oxidation test is used as a reference because it integrates isothermal oxidation kinetics, oxide-scale adherence, mechanical stresses, metallic alloy and oxide creep and the evolution of these properties with time, for conditions close to the actual conditions of use.

EXPERIMENT

A NiAl single crystal was oxidized in a THEMYS DUO. The experiment consisted of 168 cycles including dwell times of 5 min at 1150°C and 30 min dwell times at 150°C. Heating was at a constant rate of 60°C/min and cooling was controlled at 20°C/min down to 150°C.
The experiment was conduced in flowing oxygen. The obtained Net Mass Gain recording, without any data processing, is given in Fig. 2. This curve includes 12,000 data points. The usual shape of cyclic oxidation data combining mass gain due to oxidation and mass loss due to spalling is obtained.

RESULTS AND CONCLUSION

The recorded signal is detailed in Fig. 3 which is an enlargement of Fig. 2 during two cycles (148th and 149th) selected at random for illustration. On this enlargement, it is seen that the mass gain during the high-temperature dwell is about 10 mg at 1150°C.

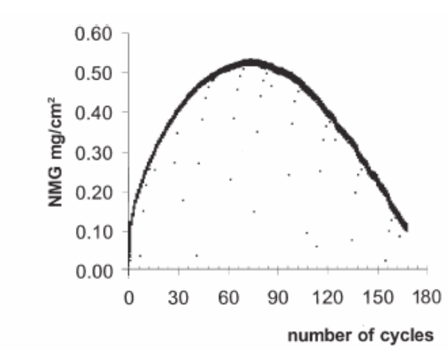
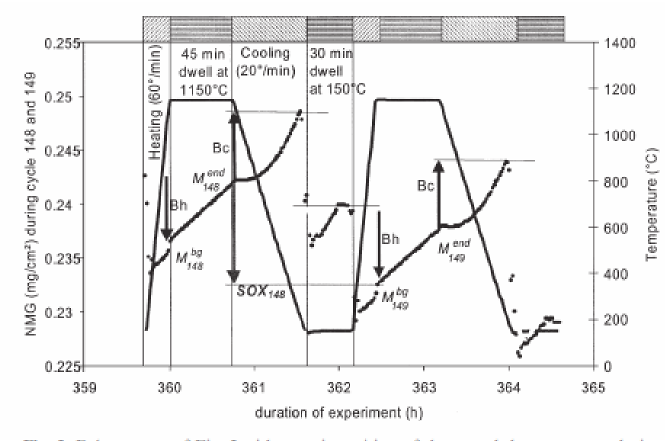


Fig. 2. Experimental Net Mass Gain of a NiAl single crystal (100) oxidized during 168 cycles of 45 min at 1150°C in flowing oxygen. Data set contains 12,000 points recorded with a SETARAM TAG24s thermobalance.



Reference : D. Monceau and D. Poquillon, Oxidation of Metals, Vol. 61, Nos. 1/2, Feb. 2004

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

Fast and accurate heat capacity measurement at high temperature (Cp of tungsten)

INTRODUCTION

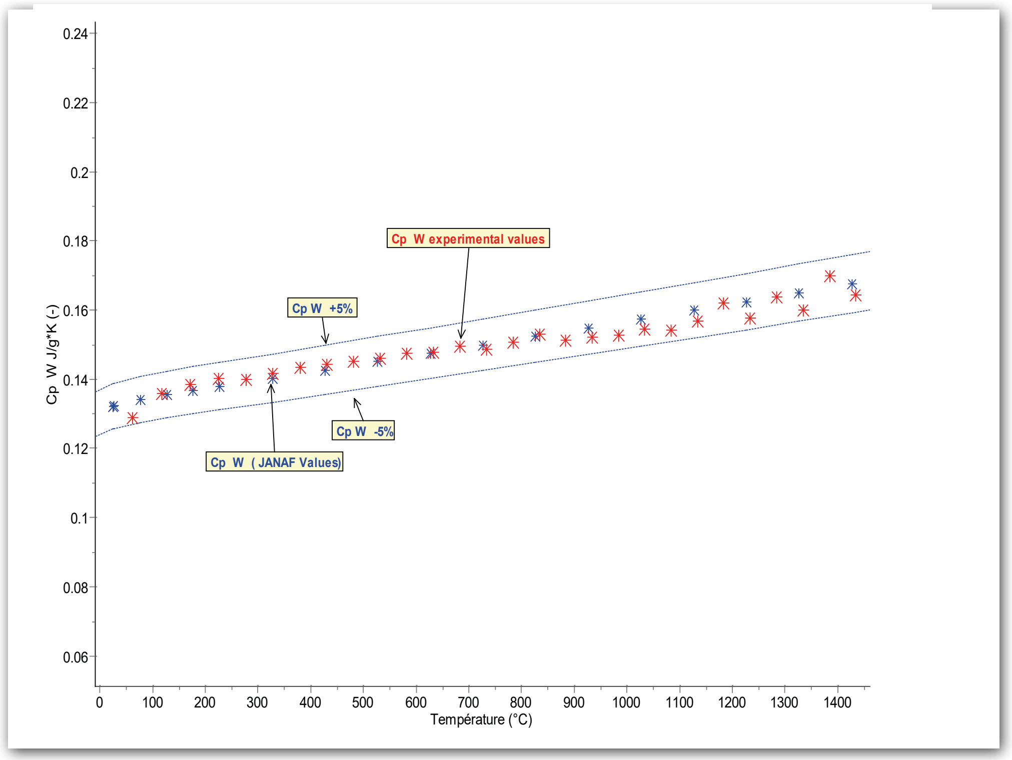
Modern industry is requiring more and more materials to be resistant to very high temperatures. Good characterization
of these materials is necessary in order to measure their precise properties to know the limits of their use and which applications they can be best applied to. Properties that are needed include heat capacity data at high temperatures as well as thermal conductivity and thermal diffusivity data

EXPERIMENT

4213 mg of tungsten is used for the Cp determination from room temperature to 1500°C using the step heating mode under argon. The selected scanning rate for the ramp is 8°C/min
and an isothermal step of 1200 seconds is applied after each ramp. A calibration using sapphire was applied for this determination.

RESULTS AND CONCLUSION

The obtained Cp values of tungsten are compared with the JANAF values. A confidence interval of ±5% is drawn on figure 5. It shows that the accuracy of the Cp determination for tungsten using the 3D Cp rod is better than ±5%.



REIMAGINE MATERIAL CHARACTERIZATION

INSTRUMENT

CALVET DC

IMPROVED HEAT CAPACITY AND HEAT MEASUREMENTS WITH THE CALVET DC

- heat flux DSC up to 1600°C – for accurate heat capacity, heat, and glass transition measurements
- drop calorimetry up to 1500°C – for accurate heat capacity, heat of dissolution and heat of formation measurements

VARIETY OF ATMOSPHERE CONDITIONS
possible with multiple carrier and reactive gas options

CONVENIENCE AND ECONOMY
with one instrument and furnace for TGA, TG-DSC, TGDTA, DSC, DTA, and TMA up to 1600°C

MODULAR ADAPTATION ALLOWING
TGA only, DTA only, TG-DTA up to 2000°C all in one instrument

SPECIFICATIONS

	CALVET DC	THEMYS LV
Temperature range (°C)	room temperature to 1600	
Isothermal and temperature scanning (°C/min)	up to 20	
Sample volume (µl)	up to 450 for heat flux DSC and 5700 for drop calorimetry	up to 18100 in TGA
Sample drop system	Manual or automated (optional)	

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

APPLICATION

Heat capacity of a steel

INTRODUCTION

specifications. It is especially true for heat transfer simulation, whether it concerns the metal's production, transformation or use. You can use high temperature calorimetry to accurately measure the variations of Cp with temperature.

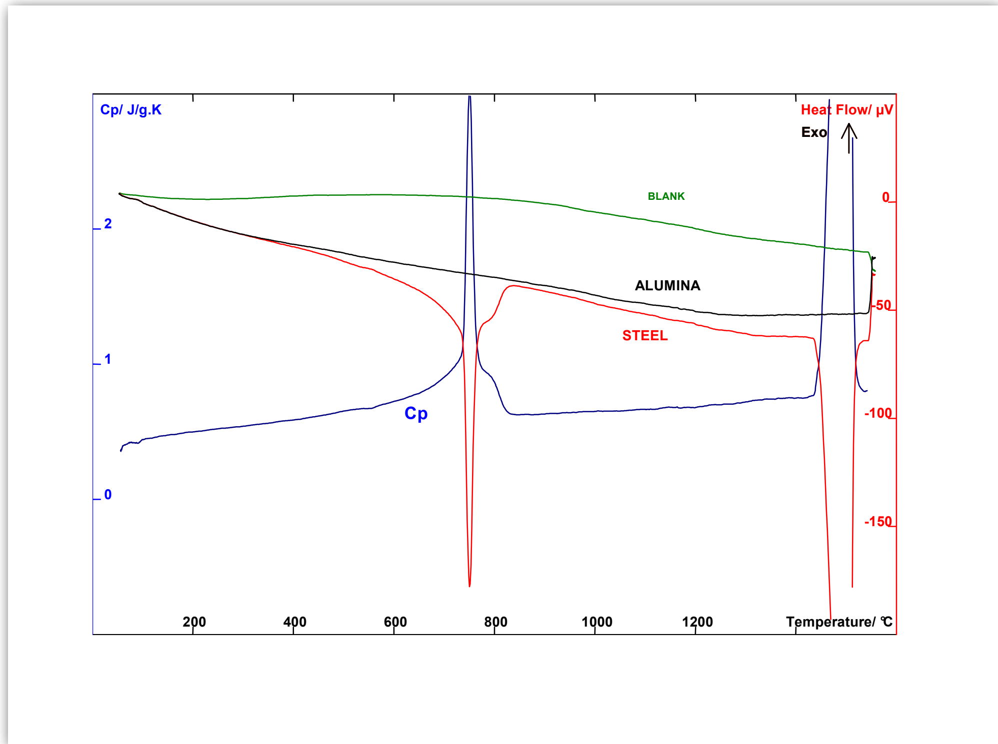
EXPERIMENT

CALVET DC was used with its heat flux DSC module.
Sample : steel
Atmosphere: helium
Sample size : diameter 5,0 mm, height =15 mm.
The sample was placed in a platinum crucible + alumina sleeve. The alumina sleeve protects the sample from alloying with the platinum crucible. The platinum crucible is used to catch the sample's radiations at high temperature. Indeed, alumina is transparent to radiations at high temperature.
The determination of Cp requires three successive heating :
- without any sample
- with a piece of alumina, used as a standard reference material
- with the steel sample

The three heating have been run from ambient up to 1550°C at 10 K/min.

RESULTS AND CONCLUSION

The heat capacity of the steel sample was calculated from the experimental curves.
The Cp could be calculated also in the liquid state at 1550°C.



REIMAGINE MATERIAL CHARACTERIZATION

PHASE DIAGRAM

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

Fast and accurate heat capacity measurement at high temperature (Cp of tungsten)

INTRODUCTION

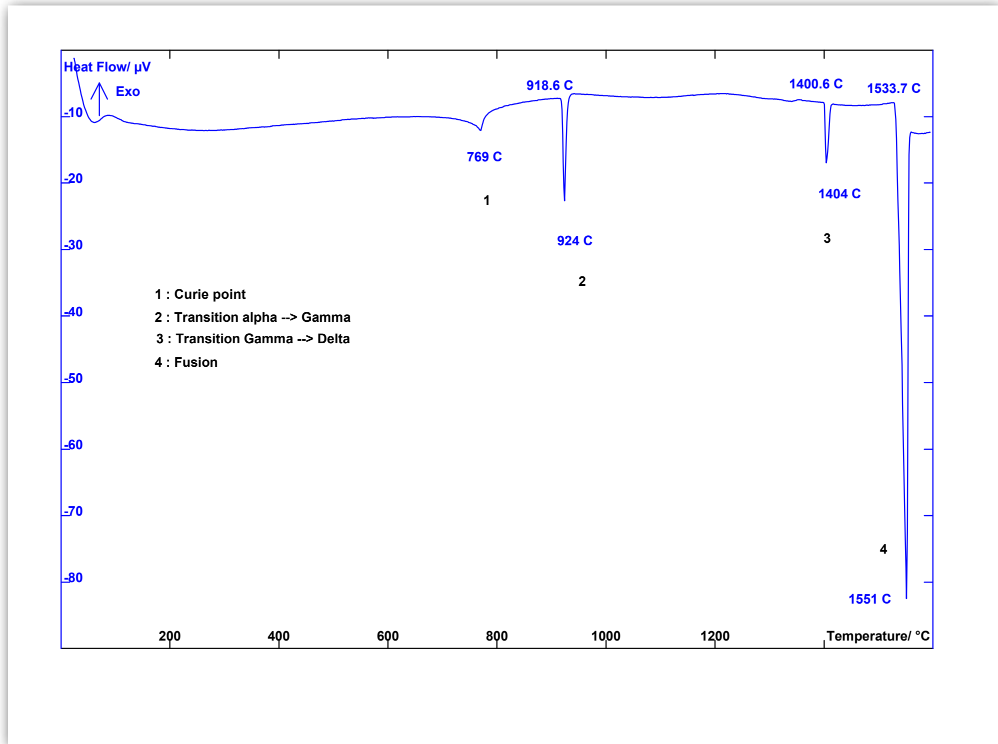
Improvement of metal and alloy properties may come from a better control over their structure. For this you can benefit from phase diagrams. DTA or DSC directly measure characteristic temperatures of a phase diagram.

EXPERIMENT

Instrument : THEMYS ONE
Sample : iron
Sample mass : 229.59 mg
Crucible : alumina
Atmosphere : Argon
The temperature was programmed from 20°C up to 1600°C at 10 K/min.

RESULTS AND CONCLUSION

Different events may be observed during heating :
1. at 769°C : curie point (magnetic transition)
2. at 924°C : α γ transition
3. at 1400.6°C γ δ transition
4. at 1533.7°C : melting of iron



REIMAGINE MATERIAL CHARACTERIZATION

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, 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

Phase diagram of the Gd-Ti binary system

INTRODUCTION

The design and processing of metallic alloys requires a detailed knowledge of their thermodynamic properties.
Differential thermal analysis provides this information and, with the help of complementary techniques, allows the determination of accurate phase diagrams. In the example reported here, the binary Gd-Ti phase diagram has been studied as a subsystem of the intermetallic compound Co-Gd-Ti. This compound is of great interest for its magnetic properties.

EXPERIMENT

- Instrument : THEMYS DTA
- Sample: Gd20TI80
- Atmosphere: helium flow
- Crucible : zirconia
- Temperature : Heating and cooling from room temperature to 1950K at 10K/min

RESULTS AND CONCLUSION

The DTA scan observed in figure 1 showed four significant endothermic peaks. Thanks to extra analytical techniques like XRD, each could be associated to a specific phase transition:
(1) Eutectoid bcc-Ti(Gd) <-> hcp-Gd(Ti) + hcp-Ti(Gd) at 1140 K
(2) Eutectoid bcc-Gd(Ti) <-> hcp-Gd(Ti) + bcc-Ti(Gd) at 1488 K
(3) Eutectic L <-> bcc-Gd(Ti) + bcc-Ti(Gd) at 1508 K
(4) Monotectic L1 <-> L2 + bcc-Ti(Gd) at 1841 K

Several GdxTi100-x samples could be tested the same way, and the experimental data were used to plot the Gd-Ti binary phase diagram as showed on Figure 2.

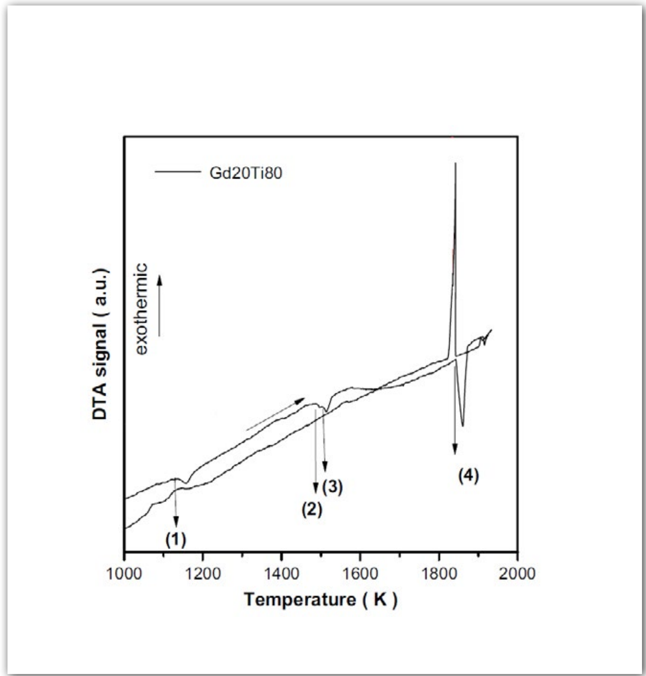


Figure 1 - DTA thermogram of Gd20Ti80. The endotherms observed during heating are indicated by numbers on the graph.

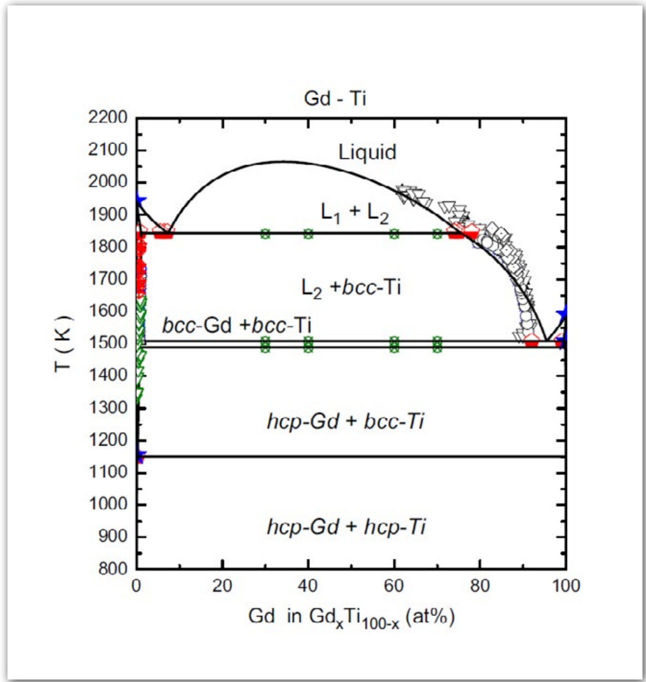


Figure 2 - Phase diagram of the Gd-Ti system as obtained by DTA (green crossed circles) and other techniques like XRD, SEM. The lines are calculated using the CALPHAD method.

Reference : N. Mattern et al, Experimental and thermodynamic assessment of the Gd–Ti system, Calphad, Volume 42, 2013, Pages 19-26

INSTRUMENT

THEMYS H2

ULTRA-HIGH TEMPERATURE CAPABILITY
to 1750°C with a single furnace

MULTIPLE BUILT-IN SAFETY SYSTEMS
for instrument and user's secured operations
• up to 100% Hydrogen capacity

HIGH ACCURACY & VERSATILE
hang-down symmetrical beam balance specifically designed for TGA applications

EXTERNAL COUPLING CAPABILITY
to 1000 °C with Mass Spectrometers

ACCURATE AND SENSITIVE
Tri- Couple DTA and DSC technologies up to 1000°C

VERY HIGH TEMPERATURE
DTA technology up to 1750°C

SPECIFICATIONS

Temperature range (°C)	Ambient to 1 750
Programmable heating rate (°C/min)	0.01 to 100
Vacuum	< 5.10-2 mbar
Measuring range	+/- 20 mg +/- 200 mg
Resolution	0.002 µg 0.02µg

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

APPLICATION

Reduction of iron ore by hydrogen

INTRODUCTION

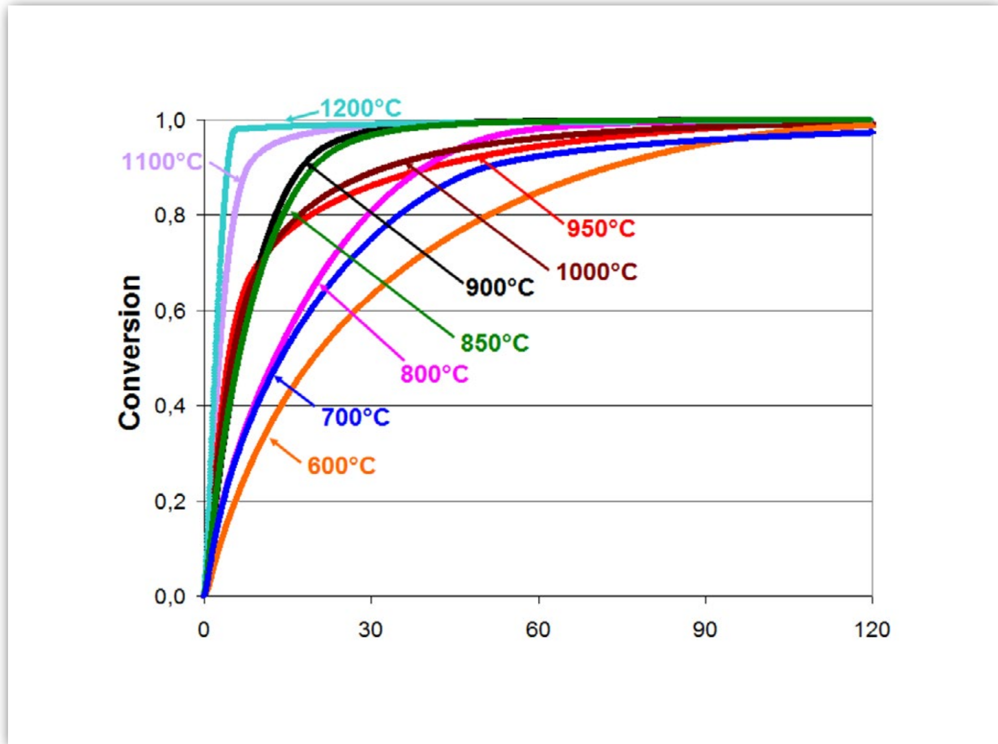
Traditional steelmaking processes generate large amounts of CO2, corresponding to 7% of global human emissions.
To reduce greenhouse gas releases, cleaner processes are being considered, such as the use of hydrogen instead of coal for iron ore reduction. This approach has the advantage of generating H2O instead of CO2 as a by-product.
Thermogravimetric analysis is an ideal tool to study such a gas-solid reaction under realistic conditions, and to characterize its efficiency and speed.

EXPERIMENT

The conversion data were calculated from the mass loss vs. time signals measured by the THEMYS H2 TGA. From 600 to 900 °C, and from 1100 to 1200 °C, increasing temperature accelerates the reaction kinetics. But between 900 and 1100 °C, it was observed that the reaction rate decreases from about 70% conversion. It was found to be due to kinetics-limiting step in the hydrogenation process. This specific behavior may of course have an impact on the performance of the industrial furnaces.

RESULTS AND CONCLUSION

The conversion data were calculated from the mass loss vs. time signals measured by the THEMYS H2 TGA. From 600 to 900 °C, and from 1100 to 1200 °C, increasing temperature accelerates the reaction kinetics. But between 900 and 1100 °C, it was observed that the reaction rate decreases from about 70% conversion. It was found to be due to kinetics-limiting step in the hydrogenation process. This specific behavior may of course have an impact on the performance of the industrial furnaces.



F. Patisson and al, Hydrogen steelmaking. Part 1: Physical chemistry and process metallurgy, Matériaux & Techniques 109, 303 (2021)

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

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

APPLICATION

Sintering of MoSI2

INTRODUCTION

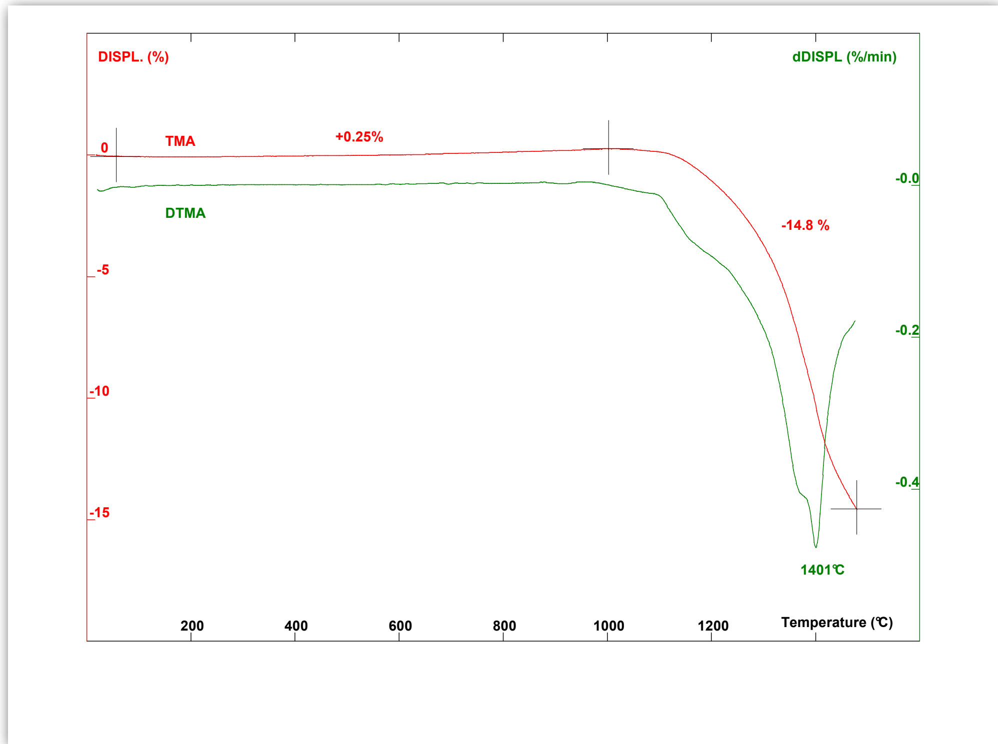
Some metallic parts are made using powder sintering processes, like those manufactured by 3D printing. These processes are preferred when it is necessary to save costly raw materials, or to manufacture lighter and more complex design parts. During sintering, the dimensions of the part change. TMA is used to measure the powder's expansion, shrinkage, and the final part's density. Our vertical TMAs preserve the sample's integrity before the experiment starts, thanks to the application of very low loads.

EXPERIMENT

A compacted MoSi2 powder sample of 3.94 mm was heated up to 1500°C at 5 K/min.
Gas : helium.
Applied load : 2 g, to avoid any sample deformation before the experiment started.
Probe : alumina.

RESULTS AND CONCLUSION

The sample shows a slight thermal expansion up to 1000°C followed by a shrinkage of 14.8% at 1500°C. This shrinkage is dure to the powder's sintering. The maximum sintering rate is 0.5 %/min at 1401°C.



INSTRUMENT

CALVET DC

IMPROVED HEAT CAPACITY AND HEAT MEASUREMENTS WITH THE CALVET DC

- heat flux DSC up to 1600°C – for accurate heat capacity, heat, and glass transition measurements
- drop calorimetry up to 1500°C – for accurate heat capacity, heat of dissolution and heat of formation measurements

VARIETY OF ATMOSPHERE CONDITIONS
possible with multiple carrier and reactive gas options

CONVENIENCE AND ECONOMY
with one instrument and furnace for TGA, TG-DSC, TGDTA, DSC, DTA, and TMA up to 1600°C

MODULAR ADAPTATION ALLOWING
TGA only, DTA only, TG-DTA up to 2000°C all in one instrument

SPECIFICATIONS

	CALVET DC	THEMYS LV
Temperature range (°C)	room temperature to 1600	
Isothermal and temperature scanning (°C/min)	up to 20	
Sample volume (µl)	up to 450 for heat flux DSC and 5700 for drop calorimetry	up to 18100 in TGA
Sample drop system	Manual or automated (optional)	

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

APPLICATION

Enthalpies of mixing of a lead-free soldering : Ag-Pd

INTRODUCTION

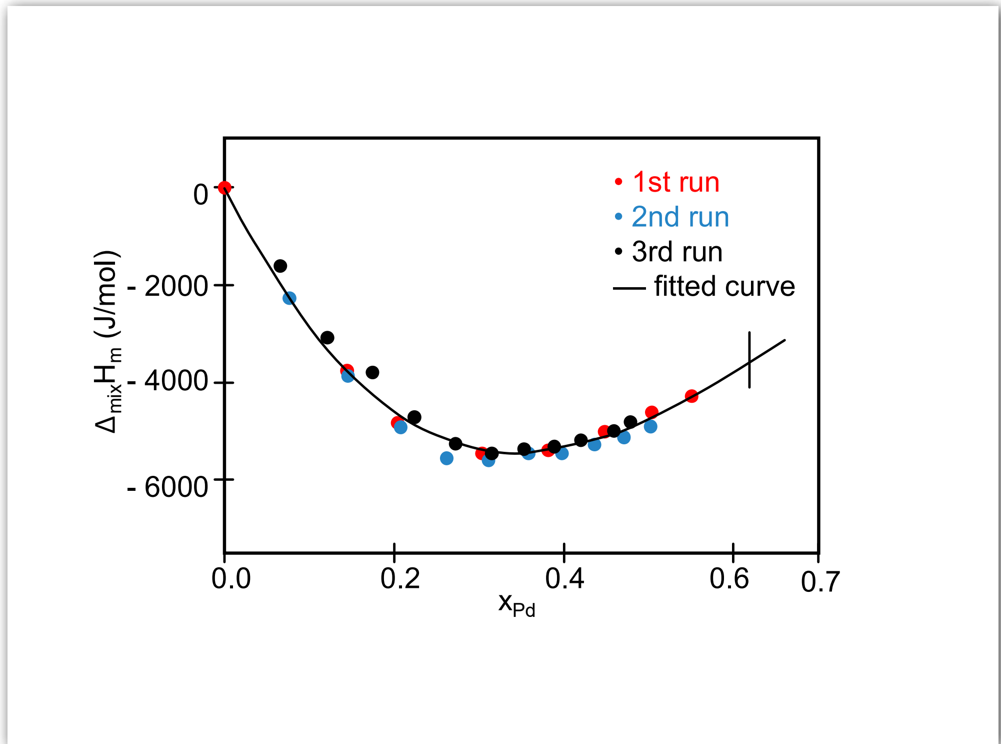
Lead and lead-containing materials are harmful but they are still present in the everyday life. Therefore, many investigations are realized to find alternatives, for example to replace the standard tin-lead solders. One of leadfree alloys being able to be appropriate is Ag-Pd because palladium is used in the production of integrated circuits.
Therefore, it is indispensable to know thermochemical properties of this alloy for the thermodynamic optimization of phase diagram and the estimation of several physical properties.

EXPERIMENT

The drop calorimetric measurements were conducted using a CALVET DC calorimeter. Small pieces of Pd (40-70mg) were dropped in pure Ag (about 600mg) to measure the enthalpy of mixing of the binary bordering Ag-Pd systems. The interval between individual drops was usually 25 min and a relatively high temperature, 1400°C, was chosen to provide a maximum liquid range.
All experiments were carried out in boron nitride crucibles and under argon atmosphere to prevent oxidation. The flow of argon was approximately 30cm3/min.

RESULTS AND CONCLUSION

Experimental data of the integral molar enthalpy of mixing for the Ag-Pd system at 1400°C were measured in three runs. According to the literature, the liquidus line is crossed at 1400°C at 62 at.%Pd. This phase boundary is marked by a vertical solid line in the figure.
All measured enthalpies of mixing are exothermic and the fitted curve shows a minimum at 35at.%Pd and -5440J/mol.
Then, the experimental data can be fitted using a Redlich-Kister polynomial according to the Calphad method.



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