

CALORIMETRIC STUDIES OF ALLOYS AT HIGH TEMPERATURE

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French Industrial group created in 1996

SETARAM > 70 years (Lyon)



Offers industrial and technological solutions



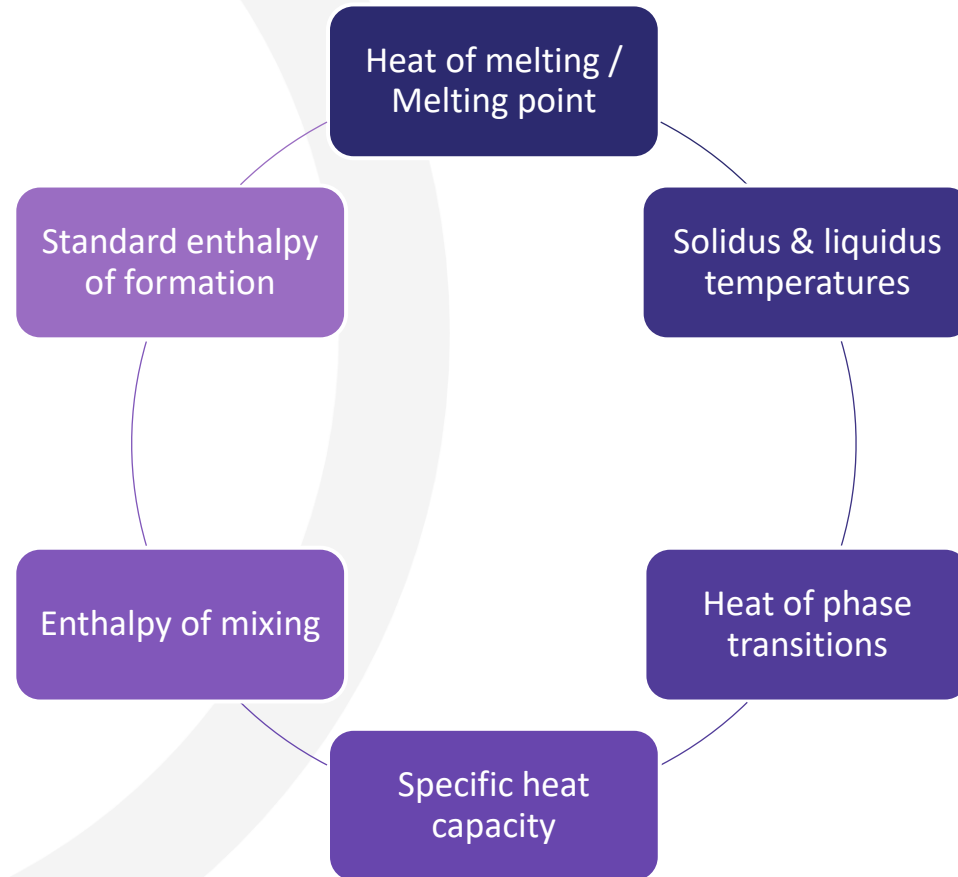
Industrial and commercial presence on 5 continents



Diversity of markets: energy, environment, aeronautics, space, health, defense, research, ...



High-temperature thermodynamic properties of alloys

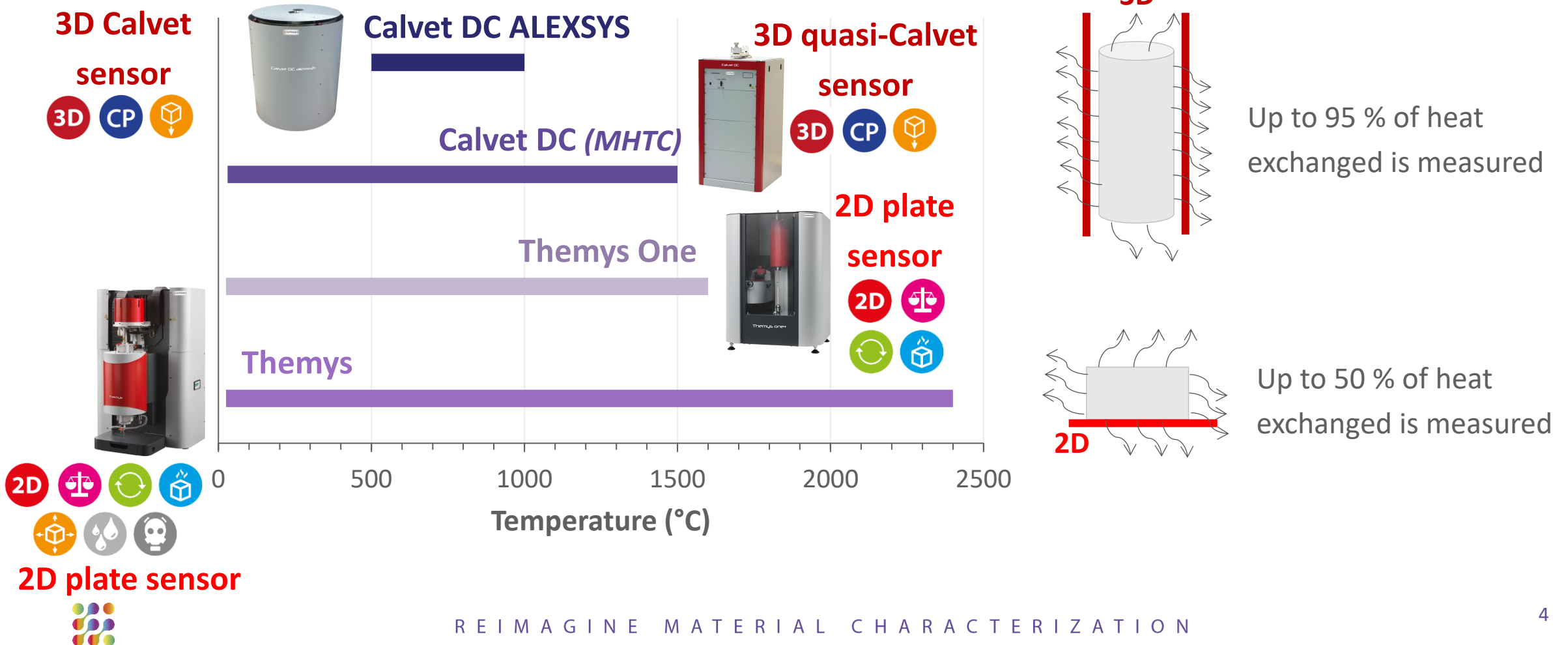


Experimental measurements are useful for:

- Evaluating phase stability to calculate phase-diagrams,
- Predicting the physical, mechanical and chemical properties of alloys,
- Providing data for ab-initio calculations,
- Improving technical processes, such as material synthesis and design or heat transfer.



For high temperature applications



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MELTING POINT DETERMINATION



INSTRUMENT PRESENTATION

TG-DSC/TG-DTA

1



THEMYS ONE

Temperature range: RT – 1600 °C

Heating rate range: 0.01 – 100 °C/min

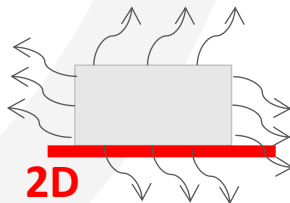
Crucibles: aluminum, alumina, platinum

Crucible volume: 80 – 380 µL

Atmosphere: Air, O₂, N₂, Ar, He, CO₂ (gas panel)

Calibration method: melting of standard materials

Add-ons: coupling with gas analysis techniques
(FTIR, MS, GC-MS)



2D-DSC rod

Flat shape DSC sensor provides heat measurements



Impact on Mo content on the properties of high-entropy alloys

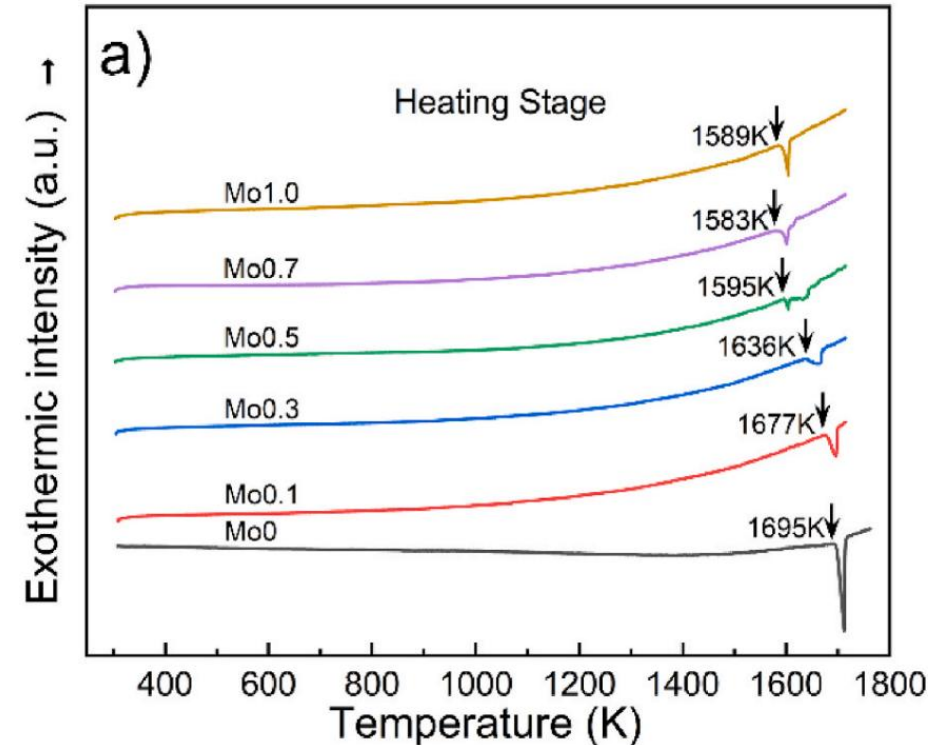
Instrument: Labsys Evo (previous model of Themys One),
TG-DSC rod

Samples: CoCrFeNiMo_x

Crucibles: alumina

Thermal profile:

- RT to 1450 °C at 10 °C/min



With an increase in the Mo content, the melting temperature decreases (destabilization of the material structure due to the large radius of Mo atoms)

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HEAT CAPACITY EVALUATION





CALVET DC HF-DSC (MHTC)

Temperature range: RT – 1600 °C

C_p: around

Heating rate range: 0.001 – 99 °C/min

5°C/min

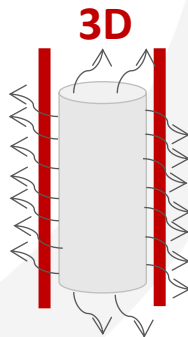
Crucibles: alumina, Pt-Rh

Crucible volume: up to 450 µL, > 1 g

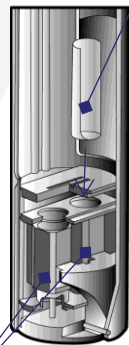
Atmosphere: Air, N₂, Ar, He

Calibration method: melting of standard materials

C_p measurement method: continuous mostly



Ceramic chambers



Crucible being inserted

3D Heat Flux-DSC (HF-DSC) sensor

3D Quasi-Calvet sensor: Based on two ceramic chambers (sample and reference) linked together by 2 conducting plates with 20 thermocouples type S or B



Impact of Ni on the C_p of Fe-C-Ni based alloys

Instrument: Calvet DC, HF-DSC sensor

Samples: 3 Fe-C-Ni alloys

Crucibles: alumina sleeves inside a platinum with lid

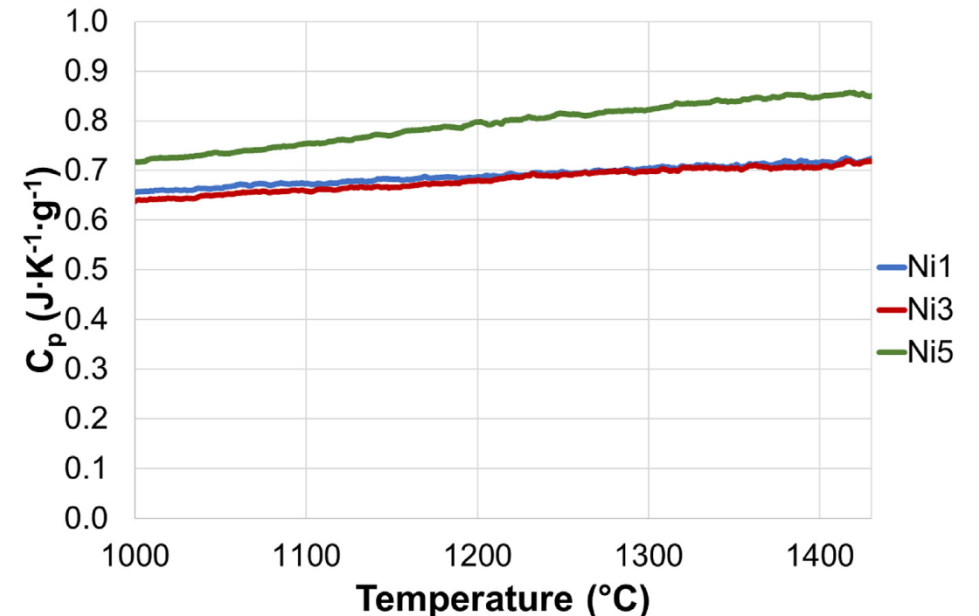
Standard reference: Platinum

Method: continuous

Thermal profile:

- 1000 °C to 1575 °C at 5 °C/min under helium

	C (wt%)	Ni (wt%)
Ni1	0.382	1.084
Ni3	0.375	2.990
Ni5	0.338	4.478



Possible to detect small C_p variations at high temperature (> 1000 °C)

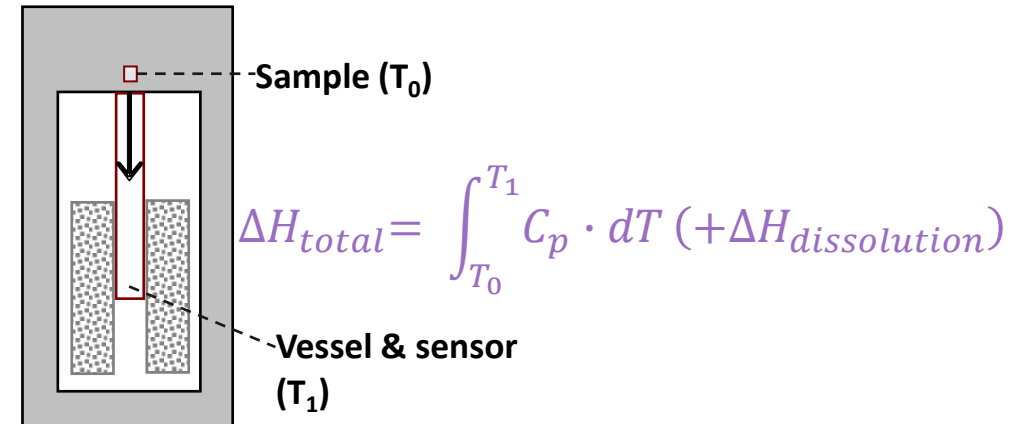
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DETERMINATION OF THE ENTHALPY OF MIXING OF ALLOYS (UP TO 3 ELEMENTS)



Drop calorimetry

Sample at RT is dropped inside a calorimeter set at high temperature: measure the heat necessary to change the sample temperature



Techniques to determine the enthalpy of mixing:

- **Direct:** compounds are dropped simultaneously, and the alloy formation occurs inside the calorimeter (depends on the melting point of the alloy)
- **Solution or solute-solvent:** compound is synthesized beforehand and dropped into a liquid element/solvent in which it can dissolve easily (no precipitation). Successive additions are necessary to get multiple concentrations.





CALVET DC DROP (MHTC)

Temperature range: RT – 1500 °C

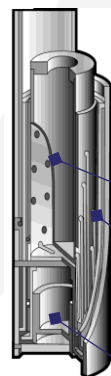
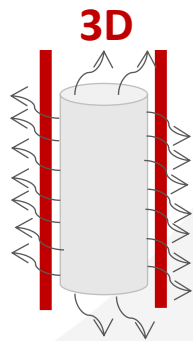
Heating rate range: 0.001 – 99 °C/min (usually used in isothermal mode)

Crucibles: alumina, Pt-Rh

Crucible volume: up to 5.7 mL, > 1 g

Atmosphere: Air, N₂, Ar, He

Calibration method: dropping reference pellets between each experiment



Drop sensor

3D Quasi-Calvet sensor: Composed of 56 thermocouples placed between the reference and sample zones

Chamber where the sample is dropped

Thermocouple network

Reference chamber



Measurements of mixing enthalpy of Ag-Mg alloys

Instrument: Calvet DC, Drop sensor

Samples:

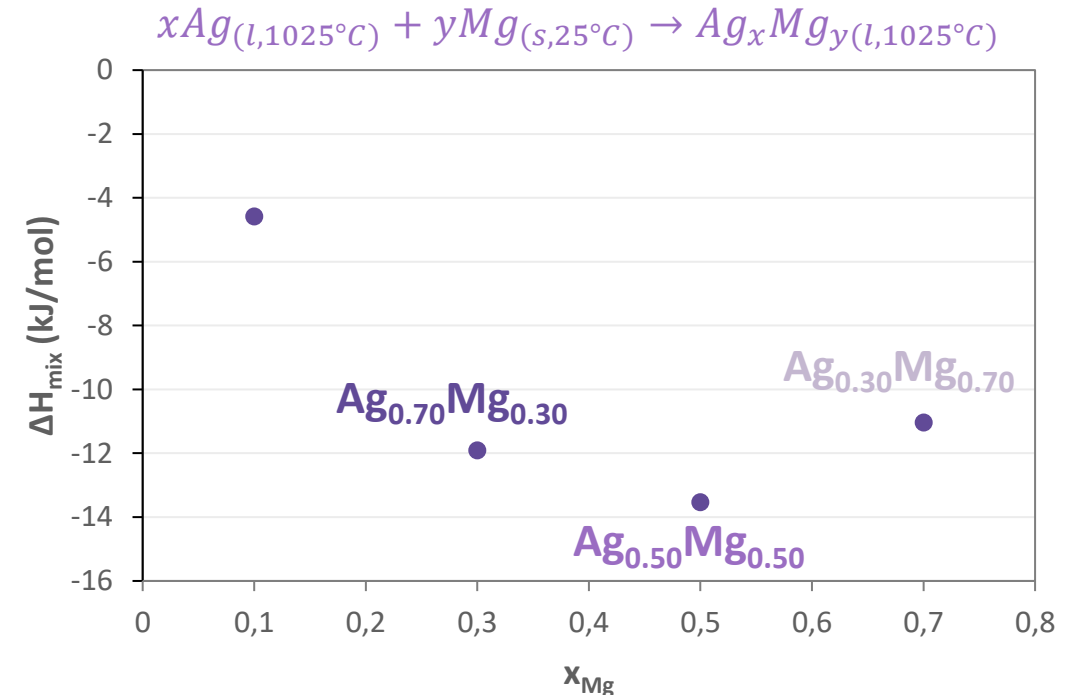
- **Liquid:** Ag
- **Solid dropped:** Mg

Crucible: MgO

Standard reference: Ag samples

Thermal profile:

- Isotherm at 1025 °C



The mixing enthalpy was measured with different ratios of Mg in Ag

Results show negative values across the entire range of concentrations and demonstrate a parabolic shape

THANK YOU



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