Investigation of oxide systems by aero-acoustic levitation

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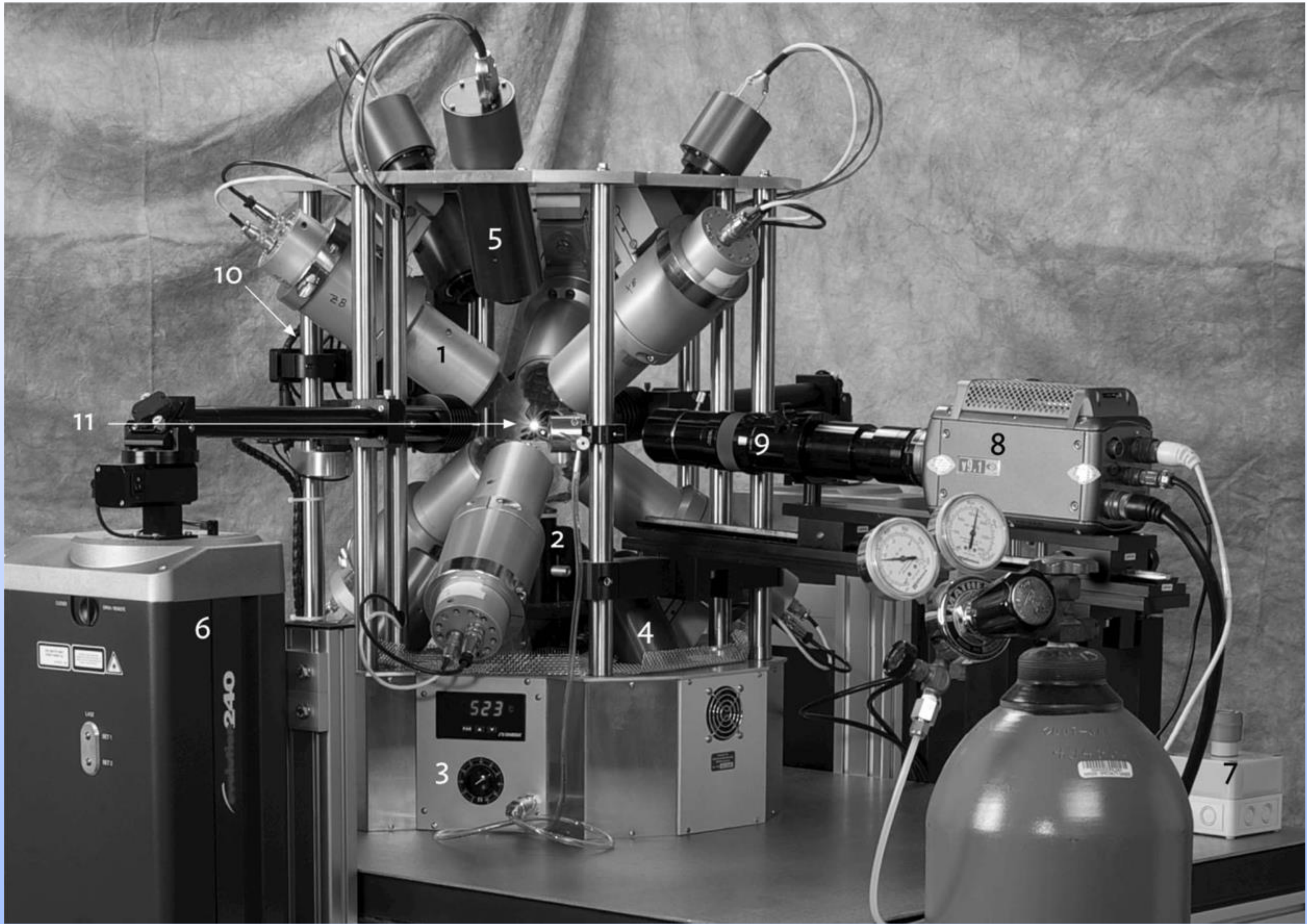
- Single phase: Al_2O_3
- Binary system: ZrO_2–SiO_2
- Ternary system: ZrO_2–SiO_2–Al_2O_3
- Conclusions and Outlook

Motivation

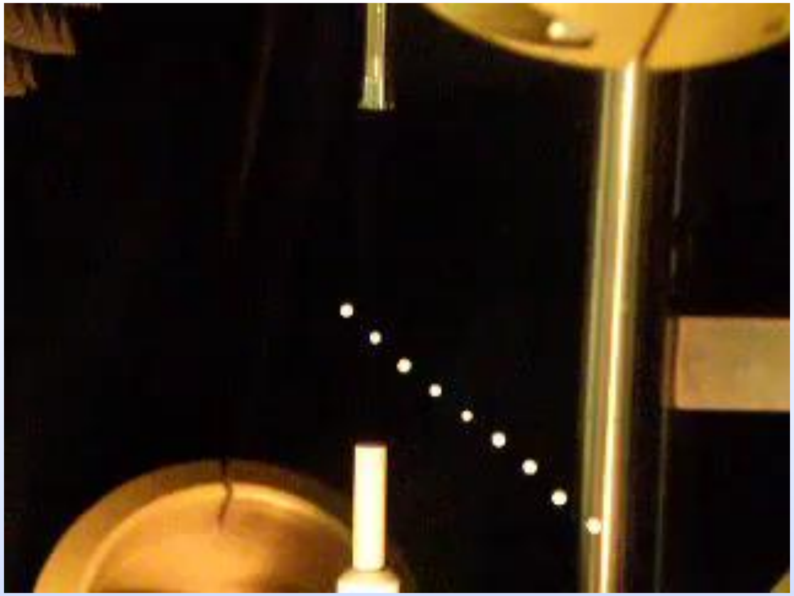
- Ternary AZS system
  - Fundament for technically important ceramics and refractories
- Advantages of levitation experiments:
  - Containerless → No Contamination
  - Contactless Laser Heating and Temperature Measurement by Pyrometry → Highest Temperatures
  - High-Speed Temperature and High-Speed/High-Resolution Camera data in Real-Time

Experimental setup



1. Supersonic transducers
2. Gas exit nozzle
3. Gas heating
4. Position sensing lasers
5. Position sensing receivers
6. CO_2 lasers
7. Emergency stop
8. High speed camera system
9. Microscopic lense
10. Pyrometer
11. Sample

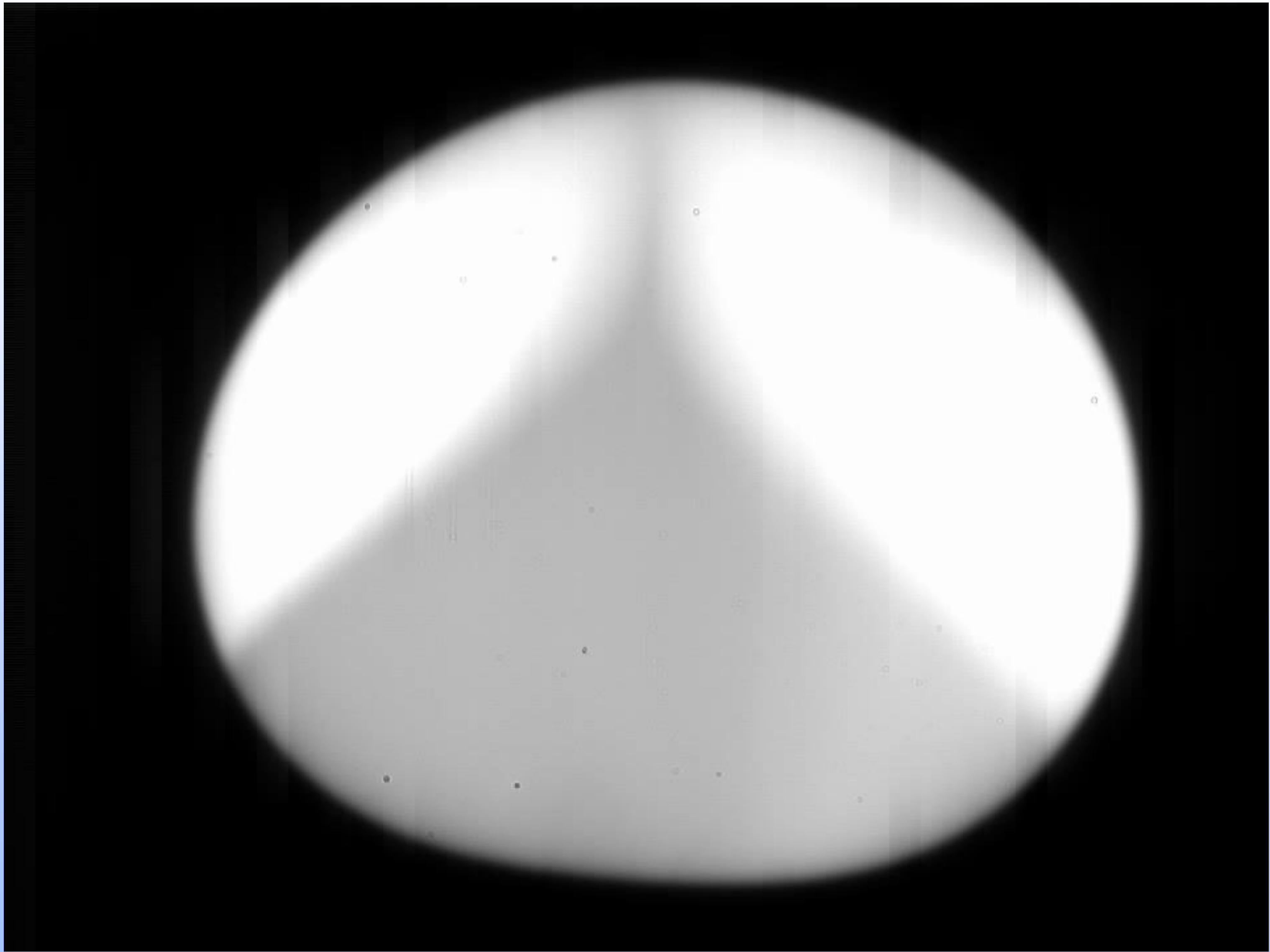
Experimental setup



Acoustic wave positioning

Copper coquille hearth for sample preparation

Single Phase: Al_2O_3



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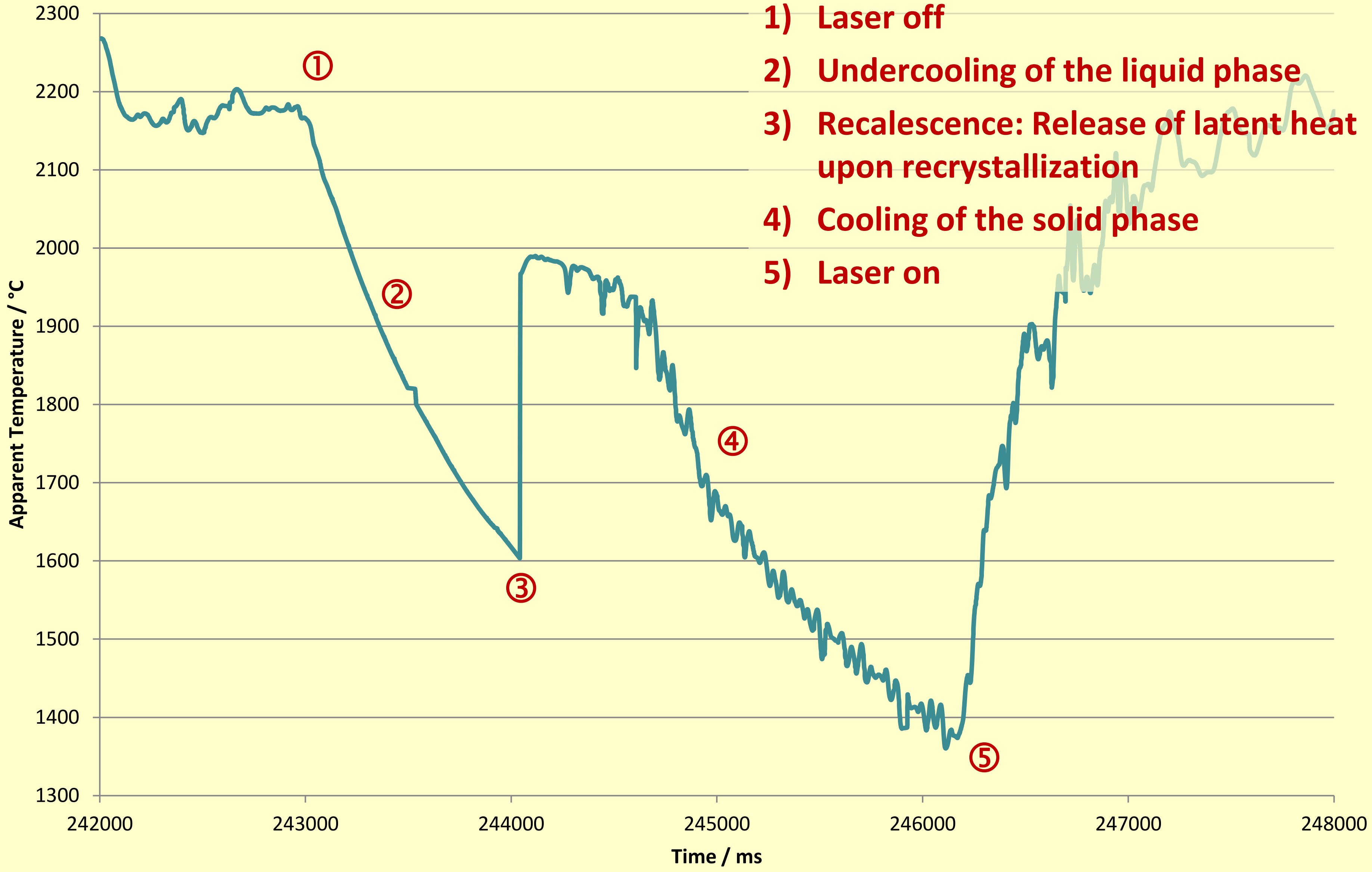
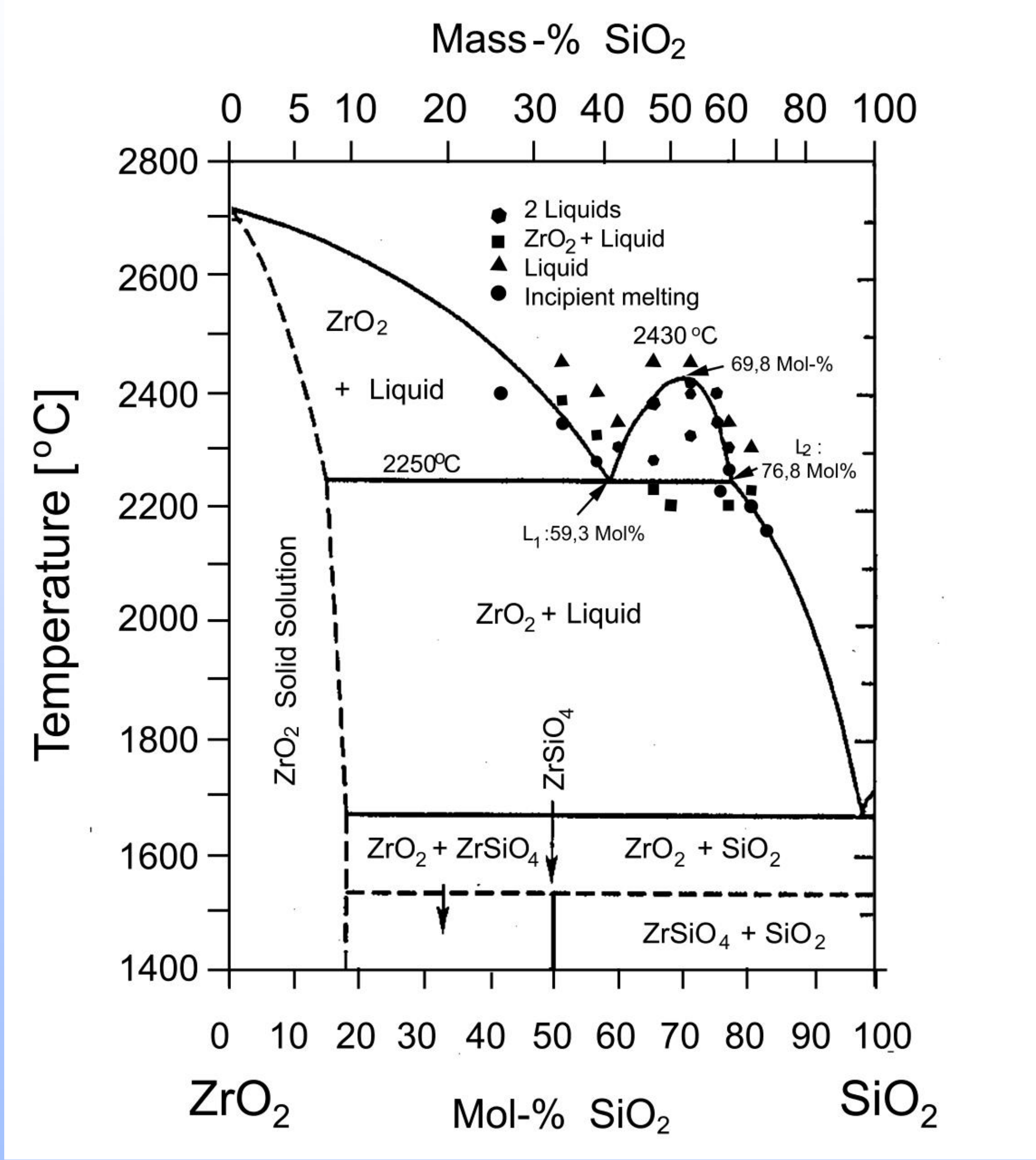


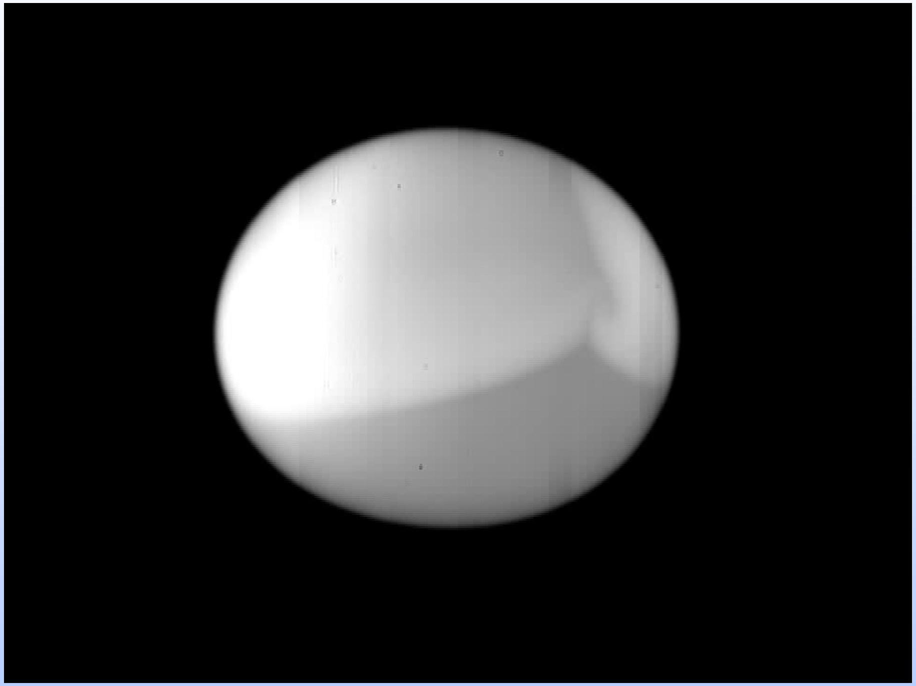
1) Laser off
2) Undercooling of the liquid phase
3) Recalescence: Release of latent heat upon recrystallization
4) Cooling of the solid phase
5) Laser on

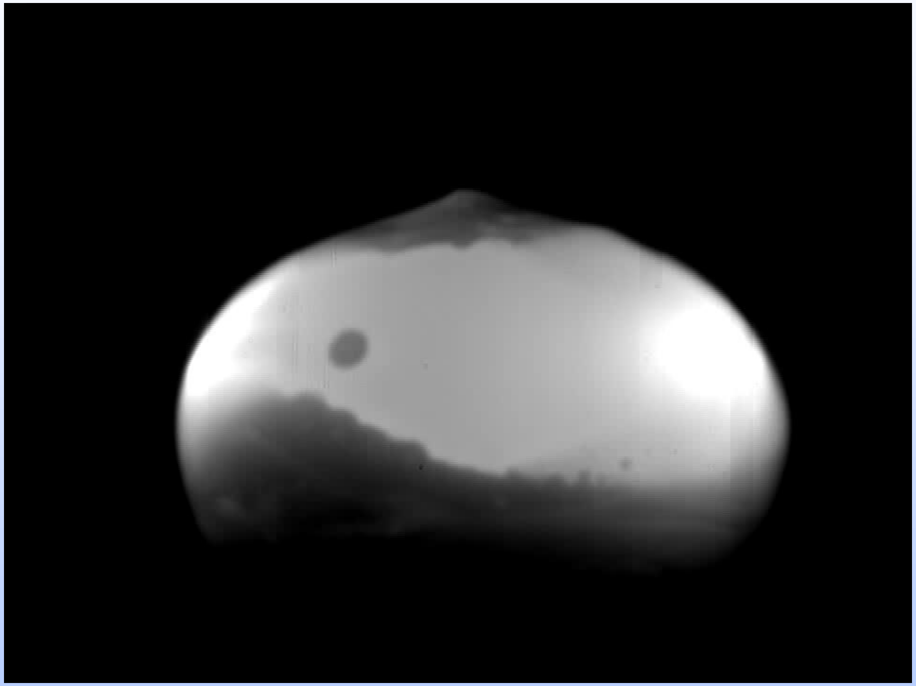
Binary System ZrO_2–SiO_2



Early phase diagram
[Toropov & Galakhov 1956]

Binary System ZrO_2–SiO_2

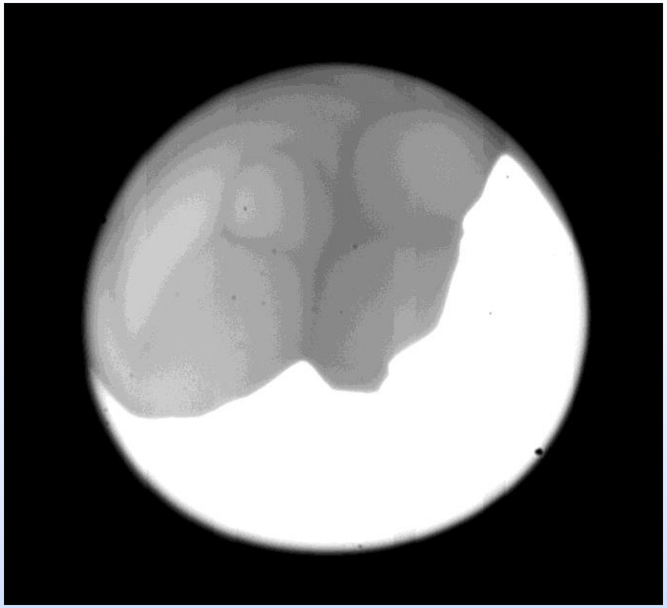




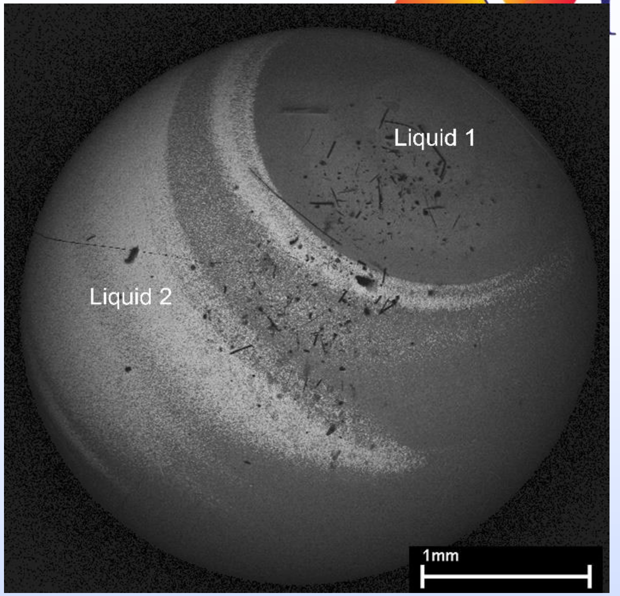
$T \approx T_{crit}$:
Marangoni Convection

T = 2250°C~2350°C:
Emulsions of immiscible melts

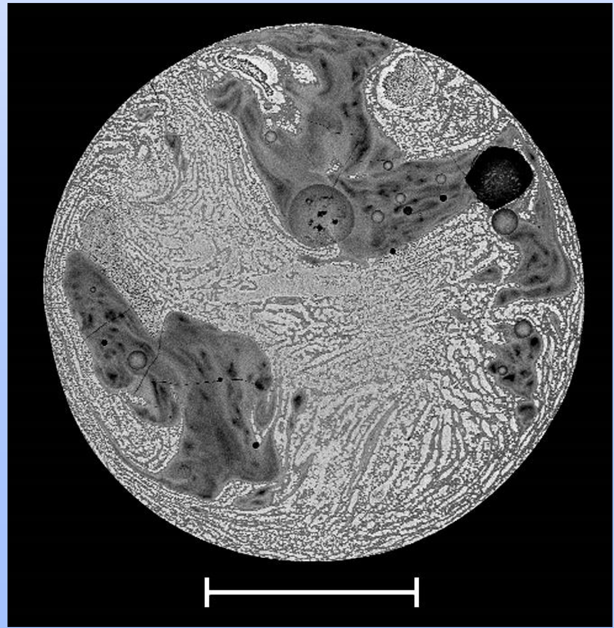
Binary System ZrO_2–SiO_2



Video still of the moment of starting crystallization with light emission and convection cells in the residual liquid

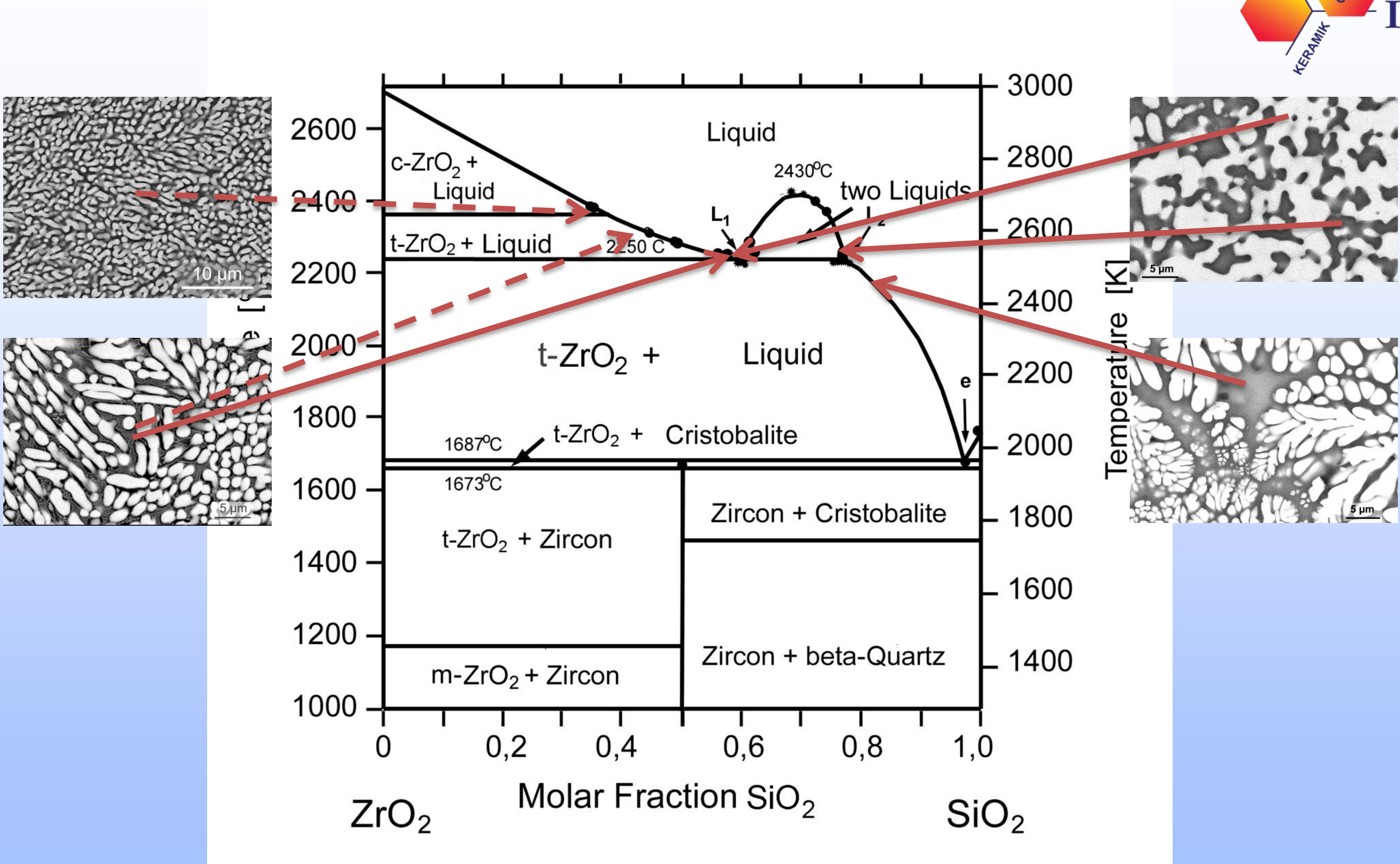


SEM micrograph of solidified sphere with reams



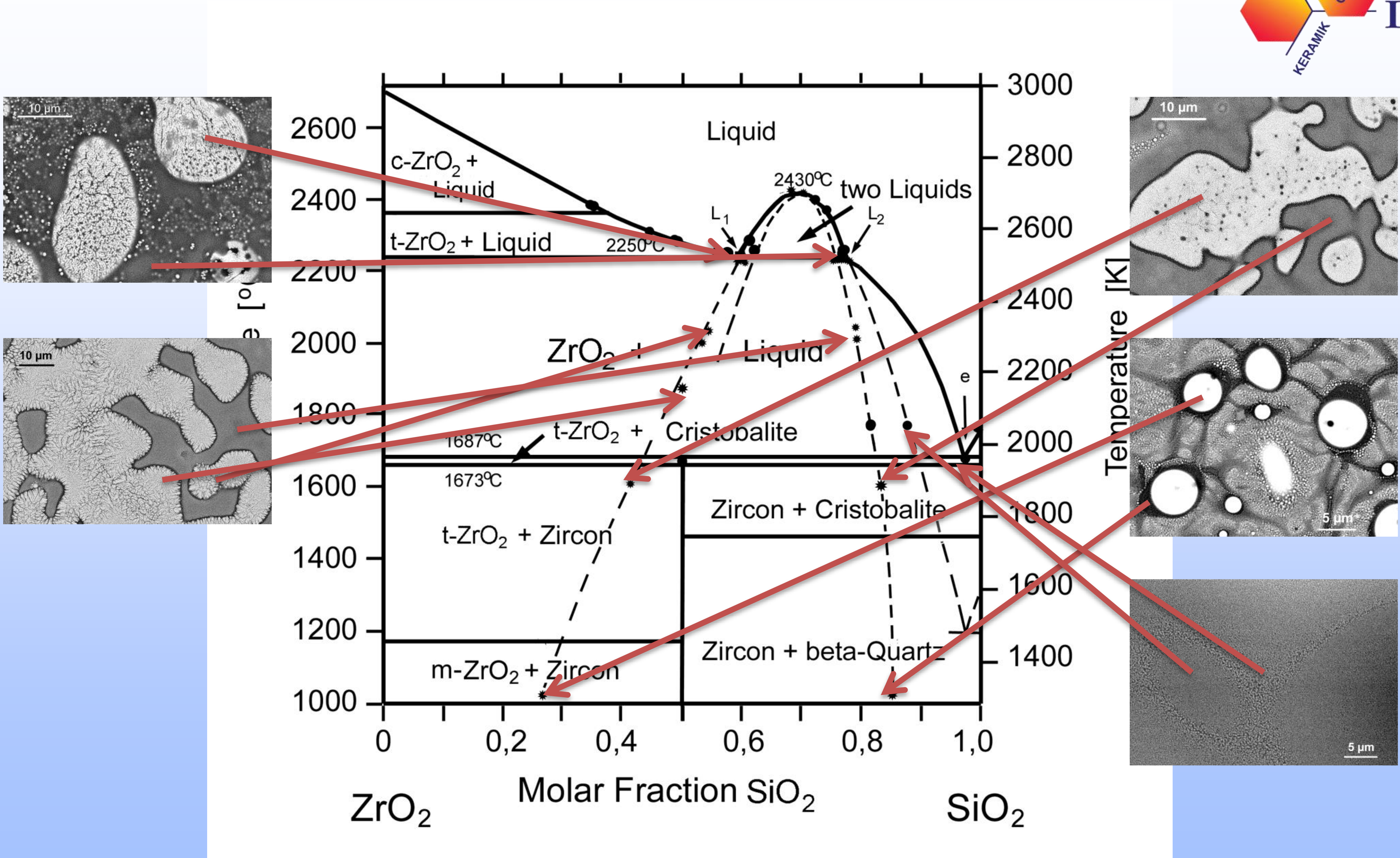
Polished cross-section of emulsified ZrO_2–SiO_2 liquids

Binary System ZrO_2–SiO_2



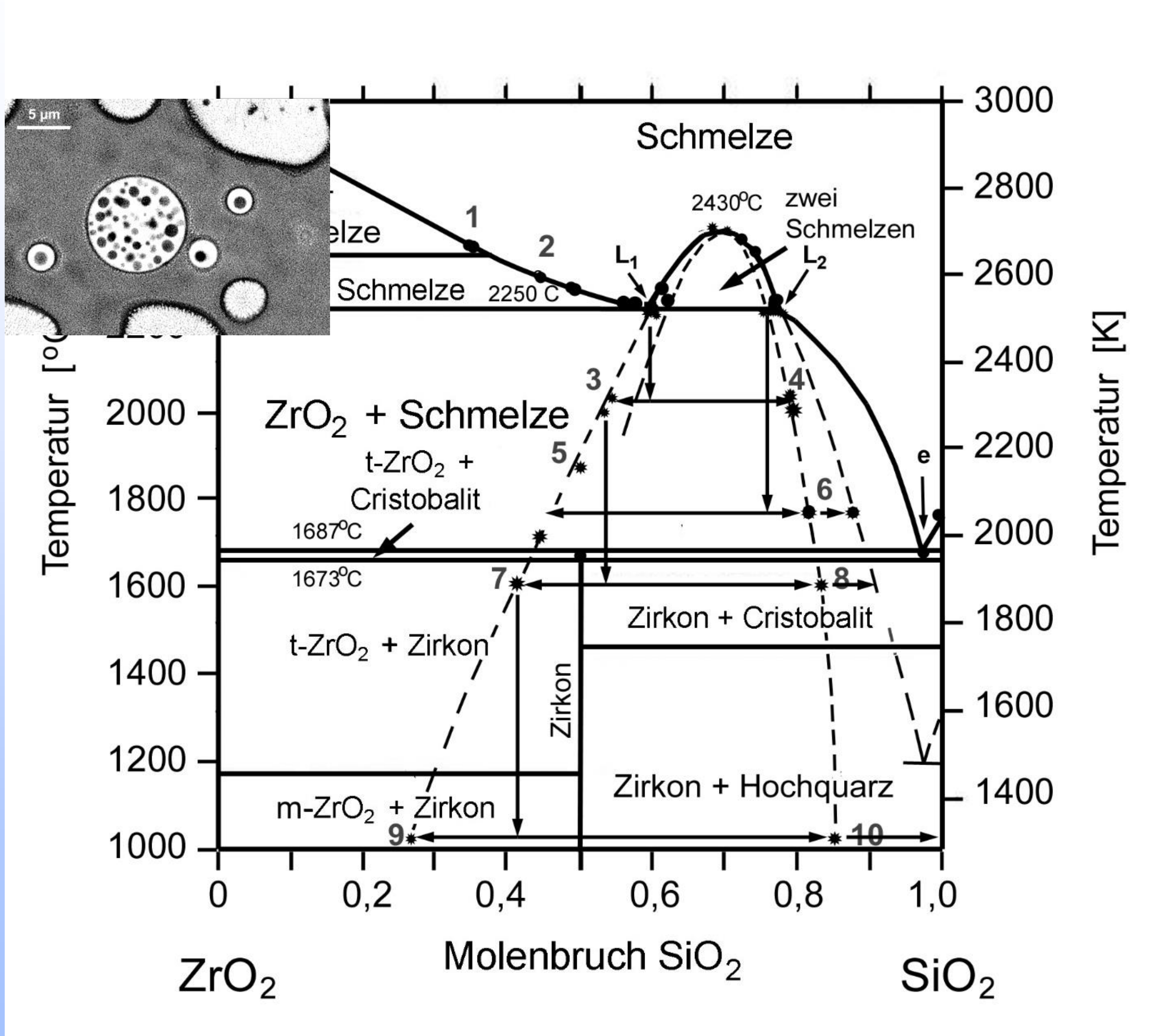
Solidification from stable supersolidus conditions

Binary System ZrO_2–SiO_2



Liquid phase differentiation in the undercooled state

Binary System ZrO_2–SiO_2



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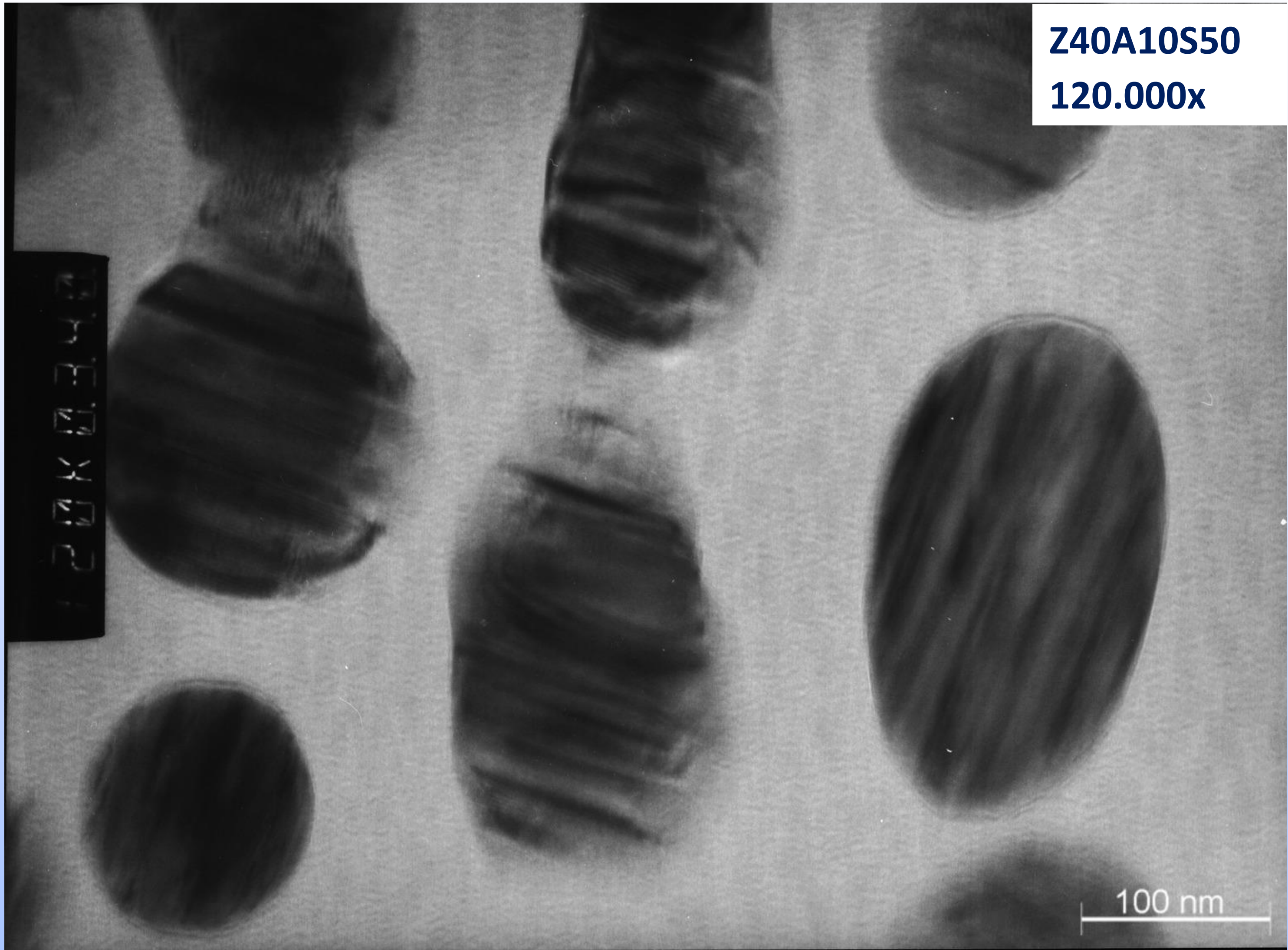
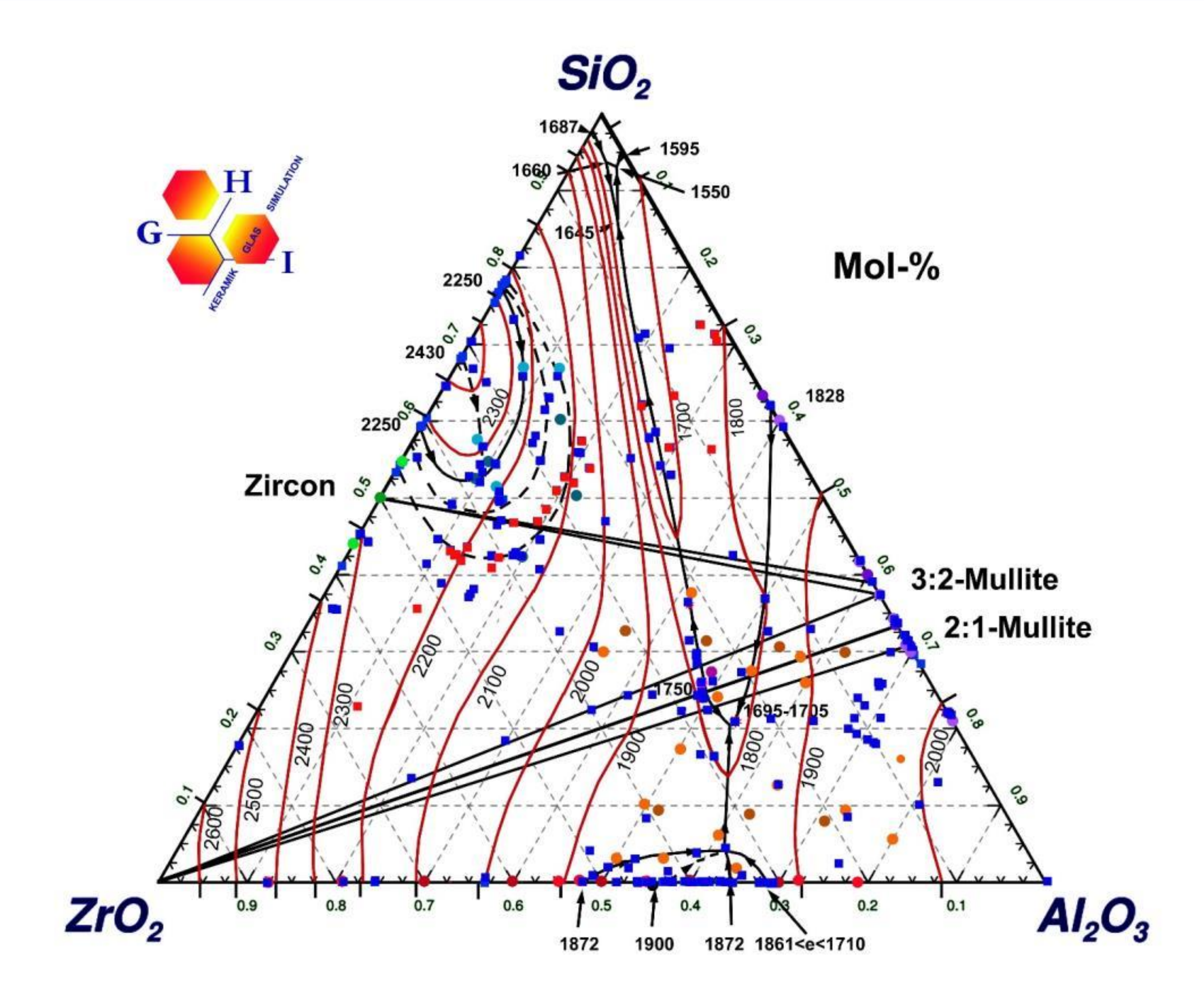


TEM micrograph: ZrO_2 particles in SiO_2–Al_2O_3 glas as matrix phase

Ternary System ZrO_2–SiO_2–Al_2O_3



- **Liquidus surface**
- **2 Eutectics**
- **2 Miscibility gaps**

Conclusions

- Experimental investigation and description of the liquidus surface and the (meta-stable extension of the) miscibility gap of the binary system ZrO_2–SiO_2 (and its extension into the ternary system)
- Experimental confirmation of the liquid miscibility gap 59 years after postulation [Toropov & Galakhov 1956]
- No solid solubility of SiO_2 and ZrO_2 observed

Outlook

- Investigation of further material systems

- Determination of further thermophysical properties, e.g.
  - Radiant heat emittance

$$\varepsilon_S(t) - \frac{\dot{Q}(t) - \dot{q}_{conv}(t)}{A_S\,\varepsilon_\infty\,\sigma\,(T_S^4(t) - T_\infty^4)}$$

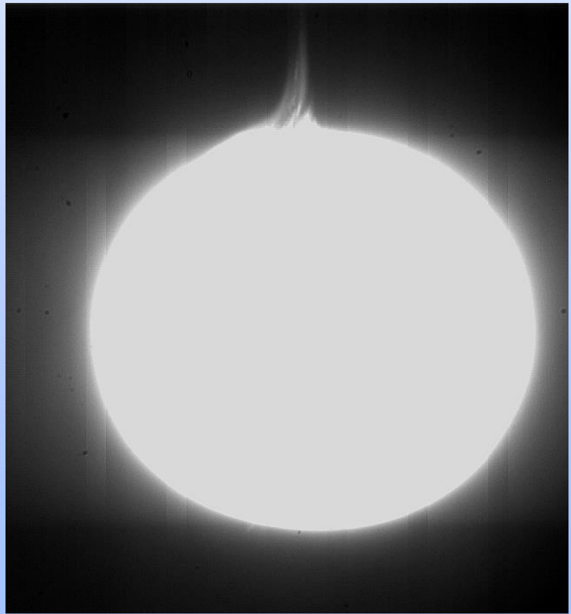
GREFFRATH, F.; PRIELER, R.; TELLE, R.: Infrared Physics & Technology 67 (2014) 333–337.

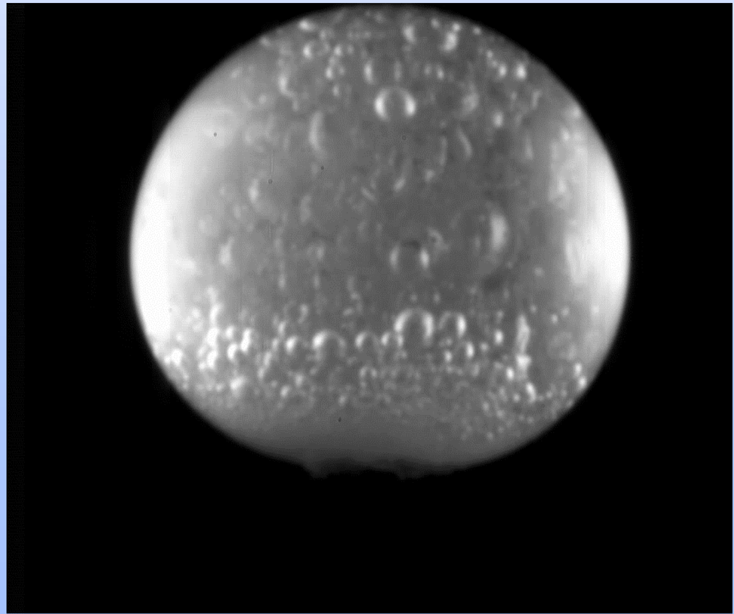
  - Surface tension

$$\sigma = \frac{3\,\pi\,m}{8}\left[\frac{\nu_{20}^2 + 2\nu_{21}^2 + 2\nu_{22}^2}{5} - 2\nu_1^2\right]$$

Limitations

- Measurement of „apparent“ temperature
- Preparation to spherical sample impossible
- High angular momentum during levitation
- Evaporation of SiO_2
- Formation of gas bubbles, glass formation, …





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Thank you very much for your attention!