

Micro scale investigations of agglomeration and deagglomeration due to single collisions between wetted particles

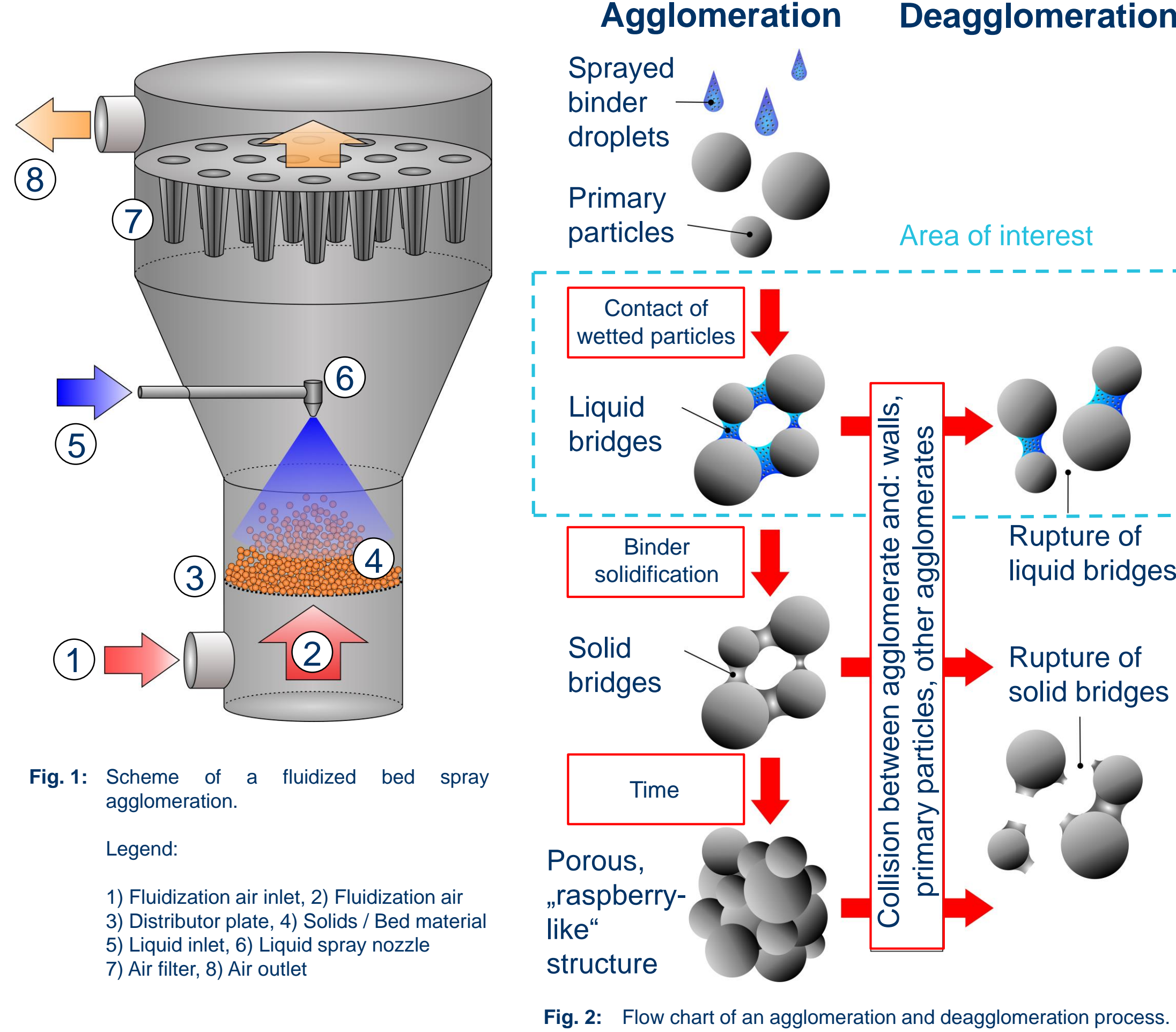
F. Bunke, S. Heinrich

Institute of Solids Process Engineering and Particle Technology, Hamburg University of Technology, Hamburg/Germany

Introduction and motivation

Fluidized beds are widely applied in several industrial fields, as e.g. chemical engineering, pharmaceutical and food industry. They are often used for gas-solid processes that include an additional liquid phase, e.g. agglomeration processes. In the mentioned process a liquid is injected as solution, suspension or melt by installed nozzles. During fluidized beds spray agglomeration wetted particles stick together, resulting in black-berry shaped and porous particles. The extend of agglomeration of the bed material will determine its further transport within the system as well as the product quality and morphology.

Therefore, this project aims to provide a fundamental understanding of the micro-mechanisms of agglomeration as well as deagglomeration of agglomerates. To investigate the mechanisms, single particle experiments are performed by means of a newly developed setup to realize binary collisions of two particles under dry and wet conditions. The collisions are recorded with two high-speed cameras to enable a three-dimensional measurement.



Materials

Yttrium stabilized zirconium oxide (ZrO₂):

Diameter x _{50,3} [μm]	1496.0	1023.5	749.3
Young's modulus [GPa]	215*		
Density [kg/m ³]	6050*		
Microhardness HV 10	1400*		
Surface roughness Ra [μm]	0.113	0.244	0.296
Sphericity [-]	0.979	0.973	0.941

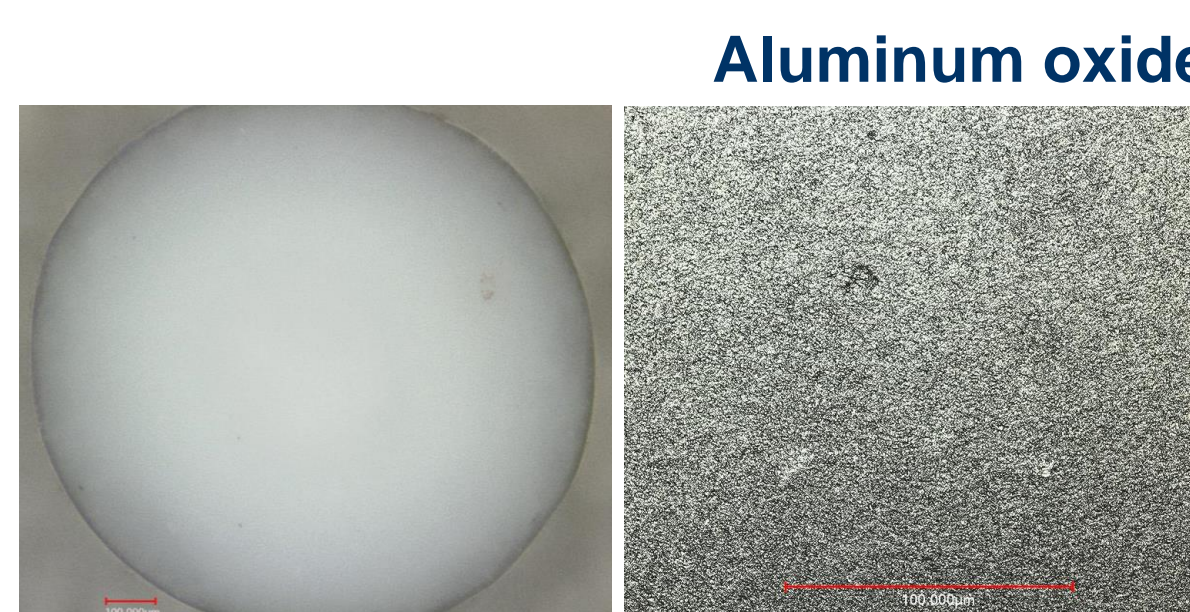


Fig. 3: Optical microscopy image of a ZrO₂ particle of size class x_{50,3} = 1023.5 μm with 10x magnification (left) and laser optical microscopy image of the particle surface with 50x magnification (right).

Aluminum oxide (Al₂O₃):

Diameter x _{50,3} [μm]	1494.0	1087.7	509.2
Young's modulus [GPa]	406*		
Density [kg/m ³]	3800*		
Microhardness HV 10	2000*		
Surface roughness Ra [μm]	0.645	0.698	1.019
Sphericity [-]	0.984	0.963	0.952

Liquids: Silicon oil with different viscosities between 50 cSt and 300 cSt (at 20 °C) and distilled water.
*) Manufacturer's data

Experimental setup

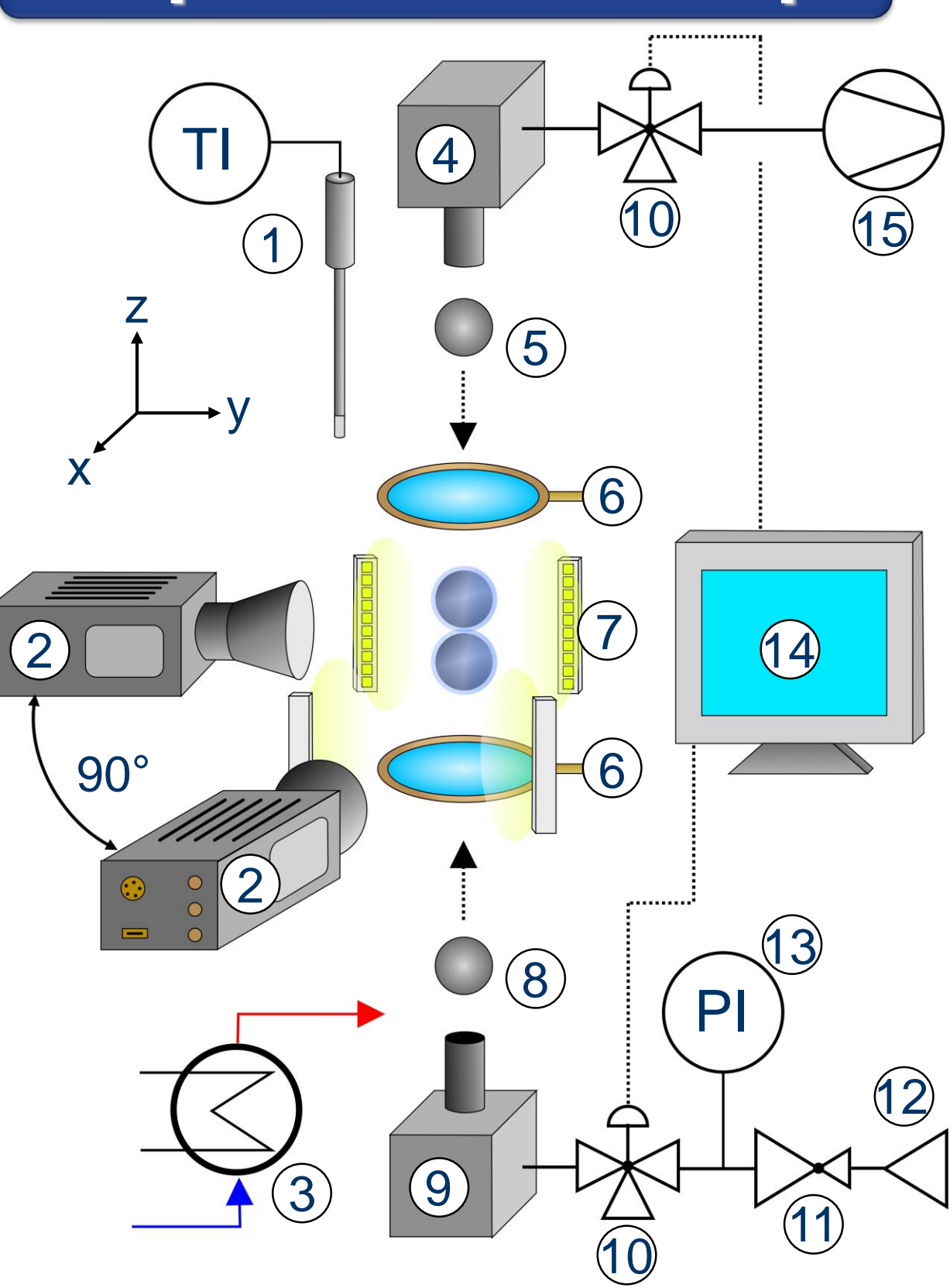


Fig. 5: Basic scheme of the experimental setup (left). View inside the experimental setup to the collision zone (middle). Exterior view of the experimental setup (right).
Legend: 1) Temperature sensor, 2) Highspeed cameras, 3) Heating system, 4) Vacuum tweezer, 5) 1st Particle, 6) Wetting ring (optional), 7) Flicker free LED light sources, 8) 2nd Particle, 9) Particle accelerator, 10) Electromagnetic 3/2-way valve, 11) Pressure regulator, 12) Compressed air supply, 13) Barometer, 14) Control system, 15) Vacuum pump, 16) Positioning system, 17) reflective inner walls of the casing, 18) Exchangeable camera background, 19) Camera positioning system, 20) Casing, 21) Sliding door

Particle wetting process

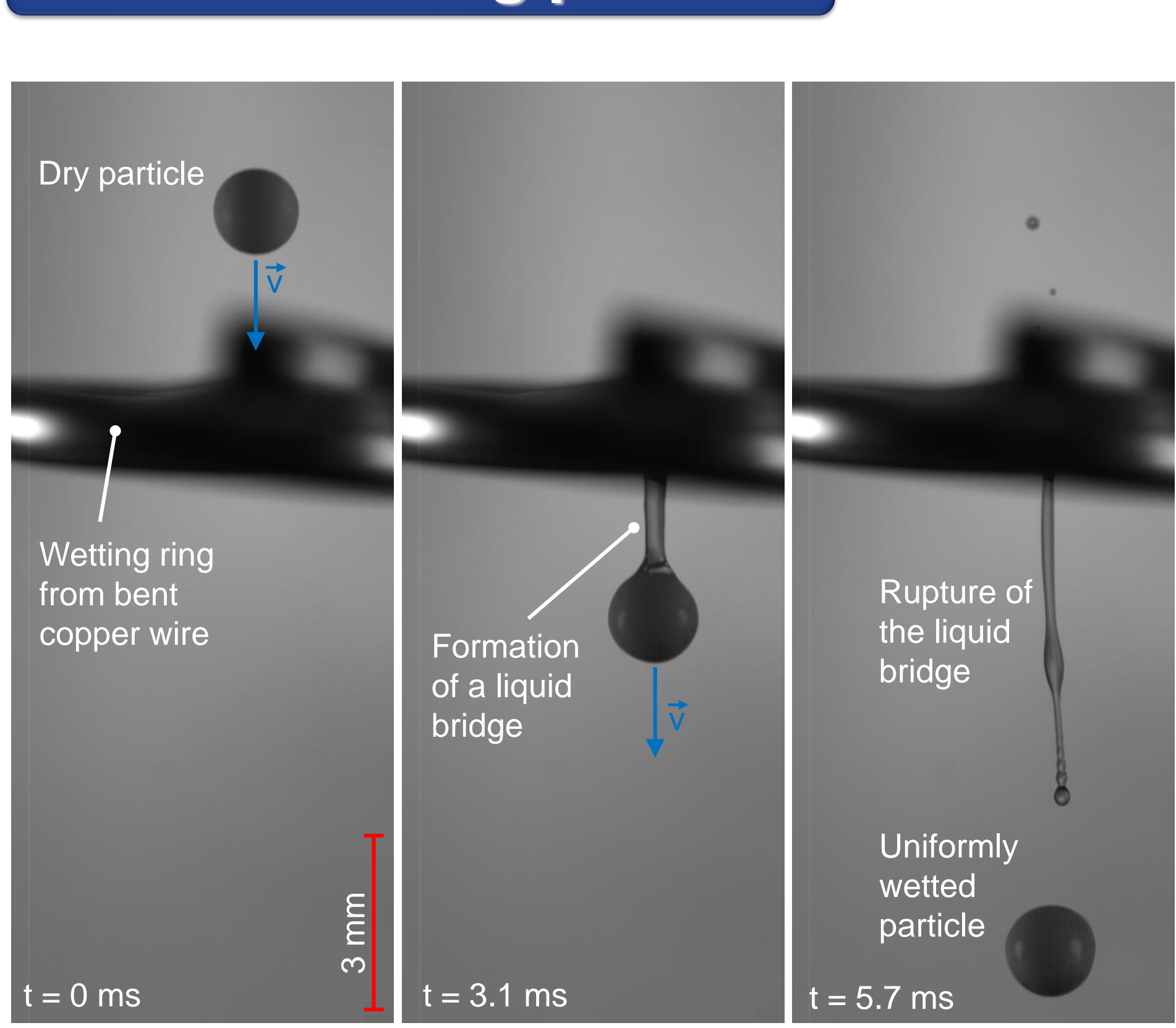


Fig. 6: Wetting process of an aluminum oxide particle. Wetting is provided by a wetting ring, which has already been used by Buck et al. (2018) [1]. Wetting liquid: Distilled water. Diameter of the particle: 1.5 μm.

Experimental results

Dry collisions

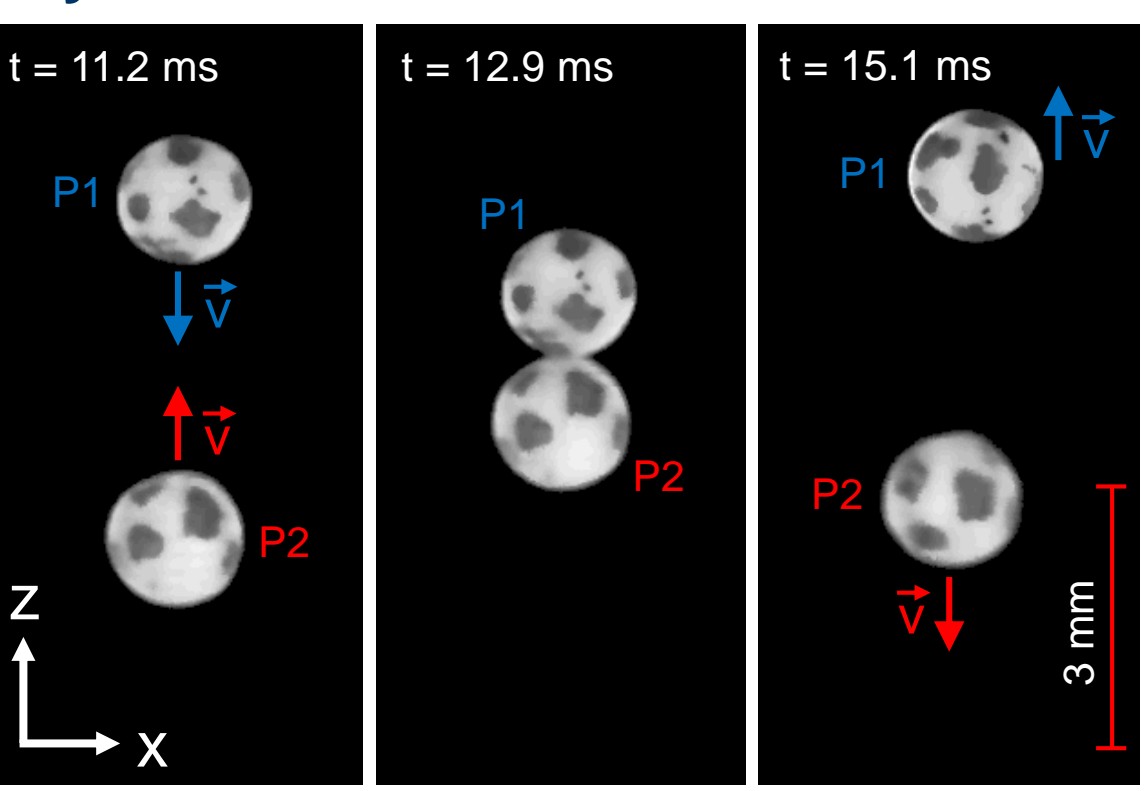


Fig. 7: Dry collision of two aluminum oxide particles, marked with dots (rotation marker) to track the rotation of the particles.

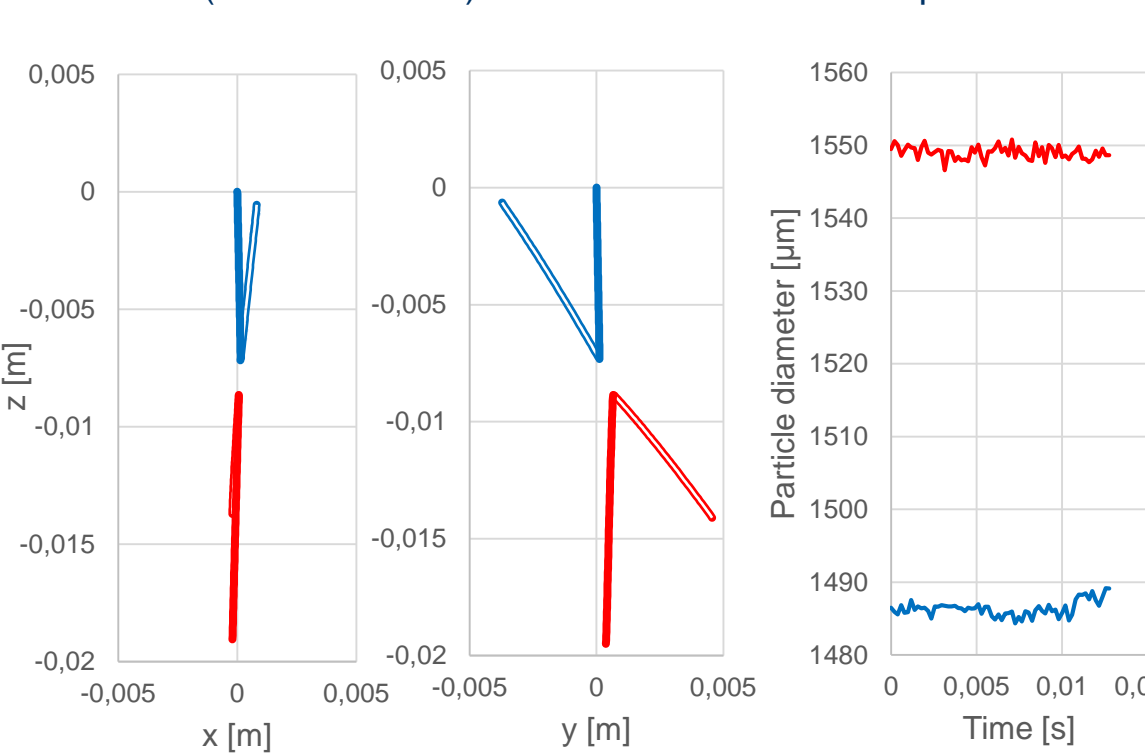


Fig. 8: Position trajectories of the particles center of mass (left and middle). Detected Particle diameter over time (right).

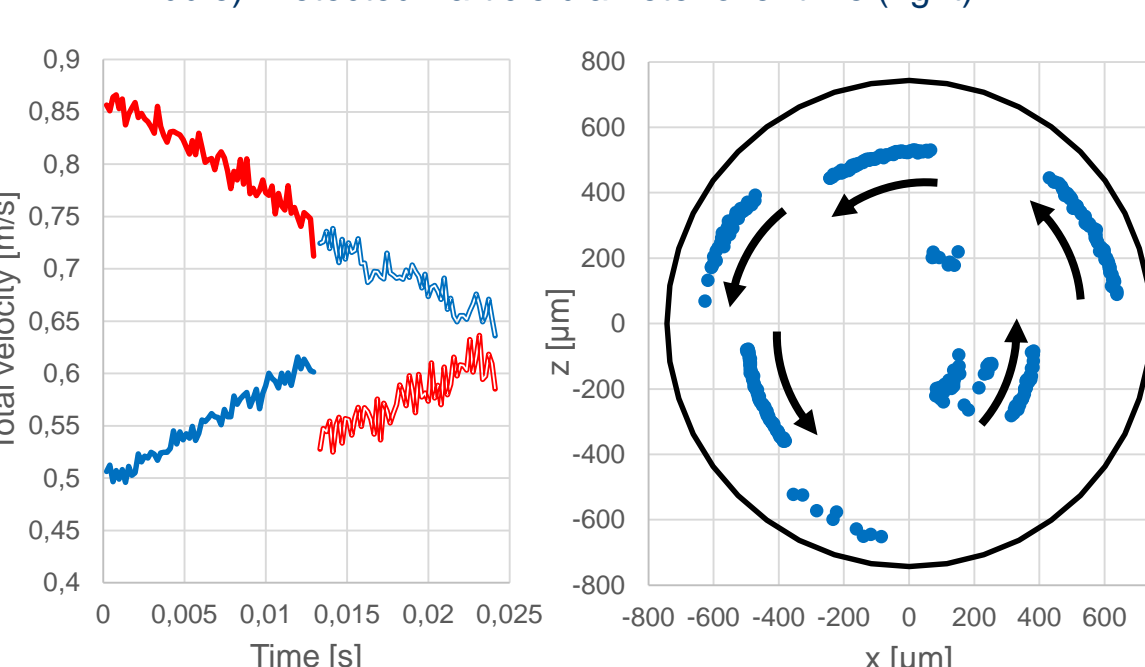


Fig. 9: Total particle velocities over time (left) and rotation marker trajectories of particle 1 before collision (right).

Wetted collisions: Case I - rebound

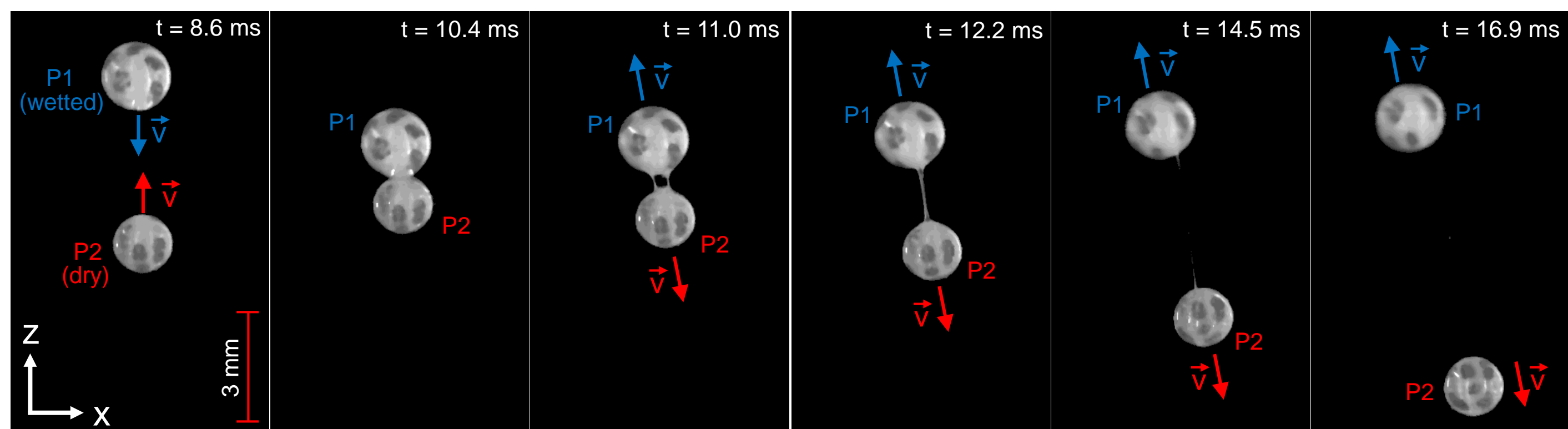


Fig. 10: Wetted collision of two zirconium oxide particles, marked with dots (rotation marker) to track the rotation of the particles. The upper Particle (P1) is wetted with a 116.3 μm thick layer of silicon oil with a viscosity of 150 cSt at 20°C. The lower particle (P2) is dry.

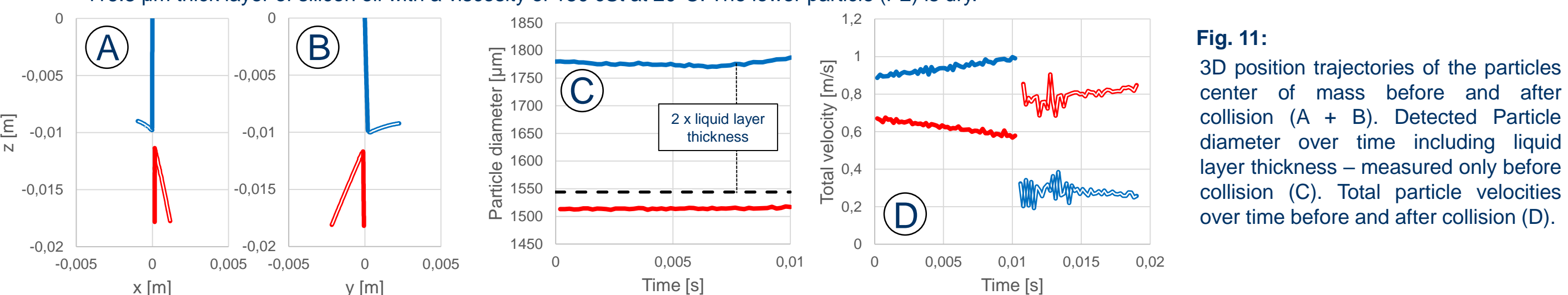


Fig. 11: 3D position trajectories of the particles center of mass before and after collision (A + B). Detected Particle diameter over time including liquid layer thickness - measured only before collision (C). Total particle velocities over time before and after collision (D).

Wetted collisions: Case II - agglomeration

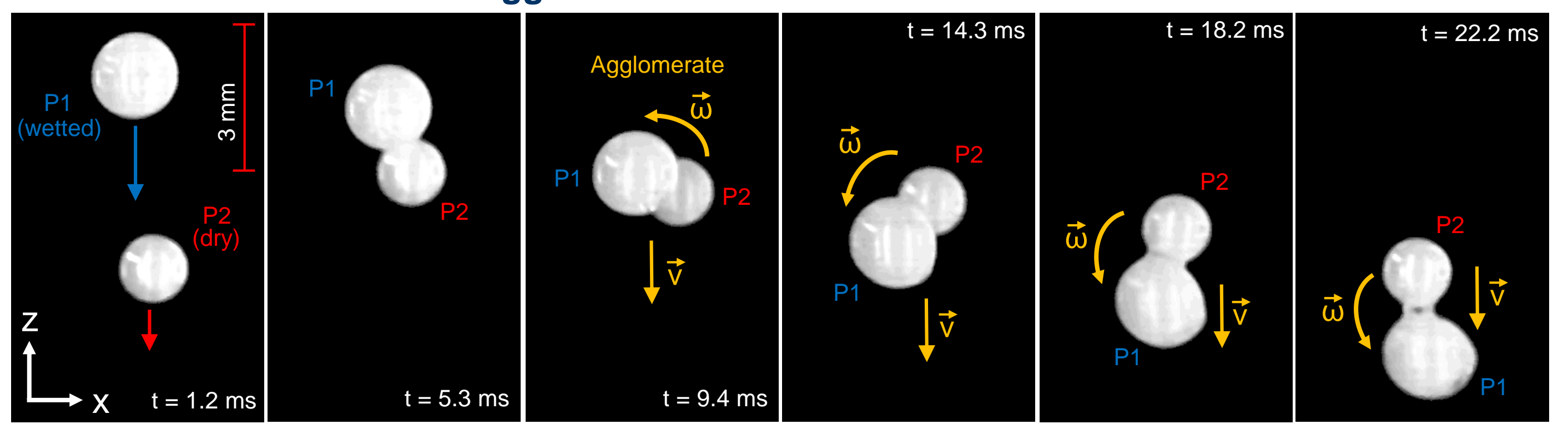


Fig. 12: Wetted collision of two zirconium oxide particles. The upper Particle (P1) is wetted with a 194.7 μm thick layer of silicon oil with a viscosity of 150 cSt at 20°C. The lower particle (P2) is dry.

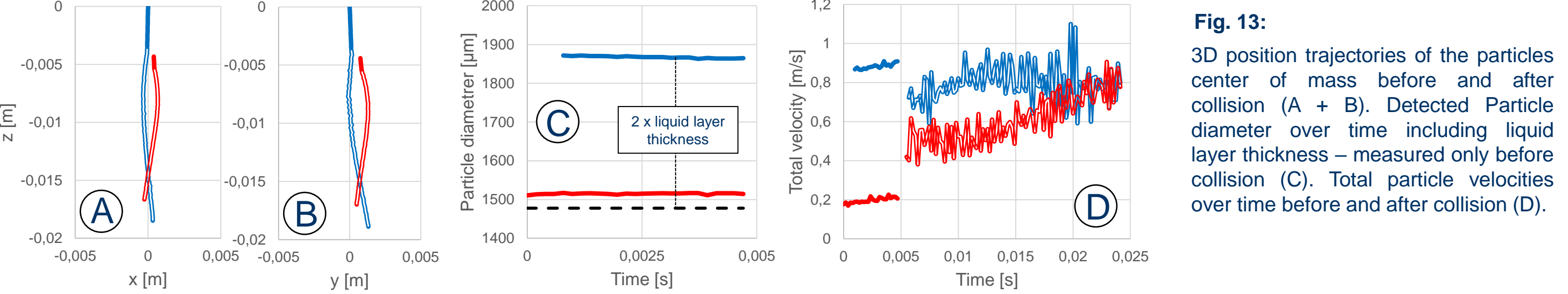
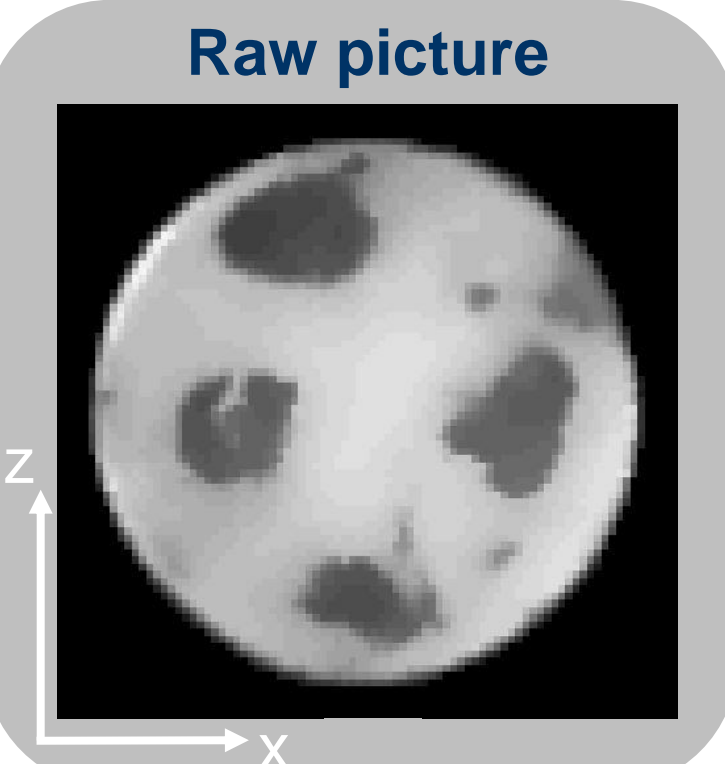


Fig. 13: 3D position trajectories of the particles center of mass before and after collision (A + B). Detected Particle diameter over time including liquid layer thickness - measured only before collision (C). Total particle velocities over time before and after collision (D).

Data analysis methods

The following measurement data is extracted from the raw data collected by the high-speed cameras:

- Position and velocity
- Particle diameter / size
- Rotation speed and orientation of the rotation axis



Digital image processing

Position and velocity	Particle diameter	Rotation
<p>Binarize based on the gray values of the pixels: Background pixel = 0 (black) Particle pixel = 1 (white)</p>	<p>Use of a subpixel accurate edge detection algorithm after Trujillo-Pino et al. (2013) [2] to find all edges.</p> <p>Length of the path L = Particle circumference. Determine the diameter: $d_p = \frac{L}{\pi}$</p>	<p>Binarize based on the gray values of the pixels: Background + rotation marker pixel = 0 (black) Particle pixel = 1 (white)</p> <p>Identify the rotation markers based on the black areas within the particle. Only areas within a specified expected size range are accepted. For each detected marker, the center of mass of the area is determined.</p>
<p>Determine the center of mass S from all pixels i with the value a_i = 1 after: $x_s = \frac{\sum a_i \cdot x_i}{\sum a_i}$ $z_s = \frac{\sum a_i \cdot z_i}{\sum a_i}$</p>		

Track the values for all pictures of both cameras. Convert the data from pixel units to metric units using a conversion factor, which depends on: camera, lens and distance between camera and particle.

a_i = Value of the pixel i
d_p = diameter of the Particle
L = Circumference of the particle
x_i = horizontal coordinate of pixel i
x_s = horizontal coordinate of center of mass
z_i = vertical coordinate of pixel i
z_s = vertical coordinate of center of mass

Funding Funded by DFG Deutsche Forschungsgemeinschaft German Research Foundation

The authors would like to thank the Deutsche Forschungsgemeinschaft (DFG) for funding the project via HE 4526/28-1.

References

[1] Buck, B., Tang, Y., Deen, N. G., Kuipers, J. A. M., and Heinrich, S. (2018). Dynamics of wet particle-wall collisions: influence of wetting condition. Chemical Engineering Research and Design, 135:21-29.
[2] Trujillo-Pino, A., Krissian, K., Alemán-Flores, M., Santana-Cedrés, D. (2013). Accurate subpixel edge location based on partial area effect. Image and Vision Computing 31:72-90.