Adhesion of powders to metal surfaces during compaction

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Adhesion of powder particles to metallic surfaces during compaction

The goal of the project is to design a diagnostic tool to determine if a powder will stick to the punch-face in full scale production. Objectives:

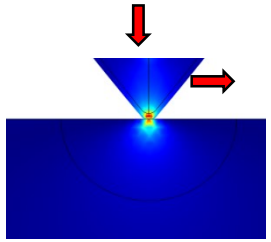
- To establish a test method.
- To identify the key factors.
- To establish a predictive criteria.

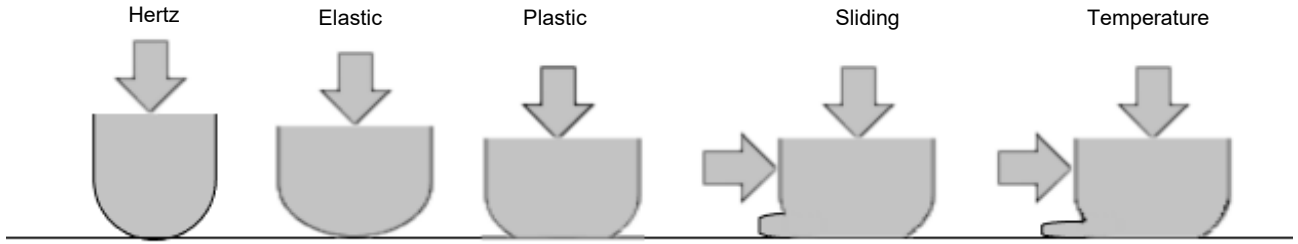
Sticking hypotheses:

1. Temperature. Sticking can be understood as a coupled thermo-mechanical problem with two sources of heat: 1) Powder compaction involves dissipative processes that generate heat and 2) At the tool interface heat is also generated due to friction. As sticking (gradual deposition of the material to the tool surface) progresses the properties of the materials and surfaces evolve, e.g. phase transformations due to stress, strain rate, temperature.

2. Humidity. Moisture uptake by hygroscopic materials leads to water acting as a binder at the interfaces, leading to sticking.

3. Particle breakage. Brittle materials or granules break during compaction, creating new surfaces. These new unlubricated surfaces give cohesion/strength to compact but also lead to sticking.





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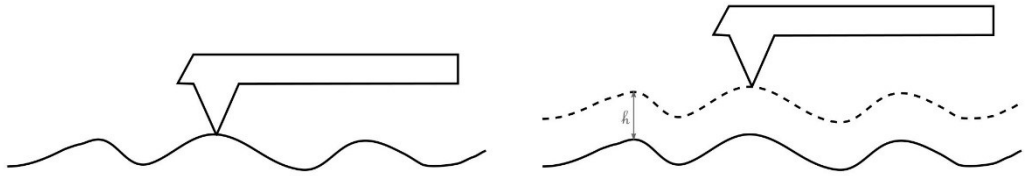
Materials

Material	Formula	Type	Sticking/ Not Sticking	Grade/Provider	Melting Point [°C]
Ibuprofen	$CH_{13}H_{18}O_2$	API	Sticking	Ibuprofen 50- GMP Pharma Grade (BASF)	50-85
Acetylsalicylic Acid (Aspirin)	$C_9H_8O_4$	API	Sticking	A5376 (Sigma Aldrich)	135
Paracetamol	$C_8H_9NO_2$	API	Not Sticking	A5000 (Sigma Aldrich)	159
Mannitol (Pearlitol)	$C_6H_{14}O_6$	Excipient	Sticking	SD200 (Roquette)	166 - 170
NEOSORB (Sorbitol)	$C_6H_{14}O_6$	Excipient	Sticking	P100C (Roquette)	97
Maize Starch B	$(C_6H_{10}O_5)_n + (H_2O)$	Excipient	Sticking	Maize starch (Roquette)	257
Microcel	$(C_6H_{10}O_5)_n$	Excipient	Not Sticking	MC102 (Roquette)	260 - 270
Lactose Granulated	$C_{12}H_{22}O_{11}$	Excipient	Not Sticking	SuperTab 30GR (DFE Pharma)	202 - 222
Lactose Spray Dried	$C_{12}H_{22}O_{11}$	Excipient	Not Sticking	SuperTab 11SD (DFE Pharma)	202 - 222
Glycolys	$(C_2H_4O_3)_nNa_n$	Excipient	Sticking	Potato starch (Roquette)	-
Solutab A (Crosscarmellose sodium)	$C_8H_{16}NaO_8$	Excipient	?	Solutab A (Roquette)	Greater than 90
Magnesium Stearate	$[CH_3(CH_2)_{16}CO_2]_2Mg$	Lubricant	-	-	89

AFM: surface roughness and Kelvin Probe surface potential

Kelvin Probe Microscopy (KPM) is a type of scanning force microscopy in which the contact-potential-difference between two surfaces is measured.



(a) Surface topography scan. (b) Surface potential measurement.

Working Principle

To measure the surface potential, a typical topography scan using the AFM contact mode is performed. Subsequently, the tip is lifted by a certain height, h, and the surface potential is measured while tracing the captured topography.

Diagnostic Tool

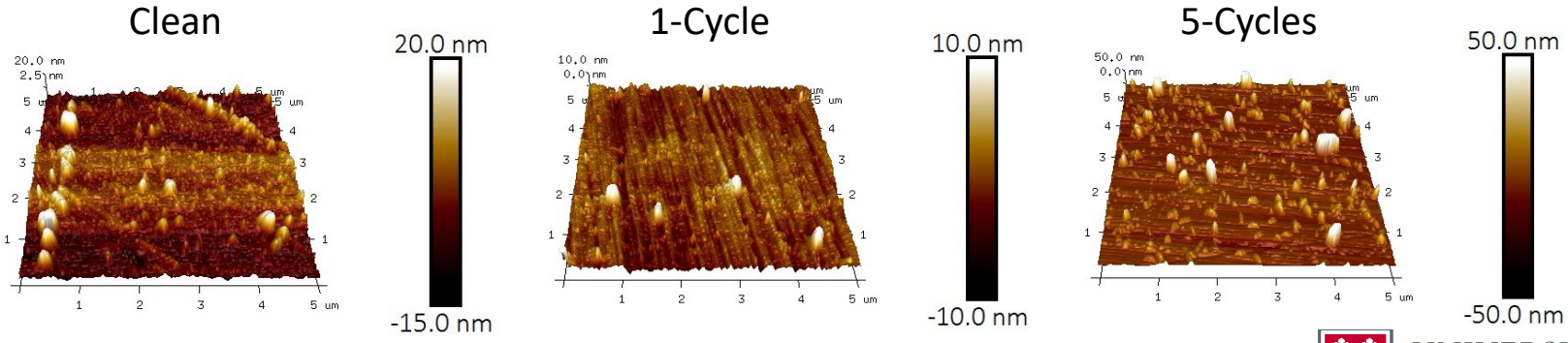
Since the tip is tracing the topography of the surface, the change in the surface potential is recorded when the tip hovers over a powder particle, while the potential elsewhere remains uniform.

Test Parameters

Powder:	Mannitol
Punch Material:	Stainless steel
Scan Size:	5µm × 5µm
Lift Height (h):	30.0 nm
Tip Velocity	1 µm/s

Three punch surfaces

1. Clean punch.
2. Punch used for 1 cycle.
3. Punch used for 5 cycles.



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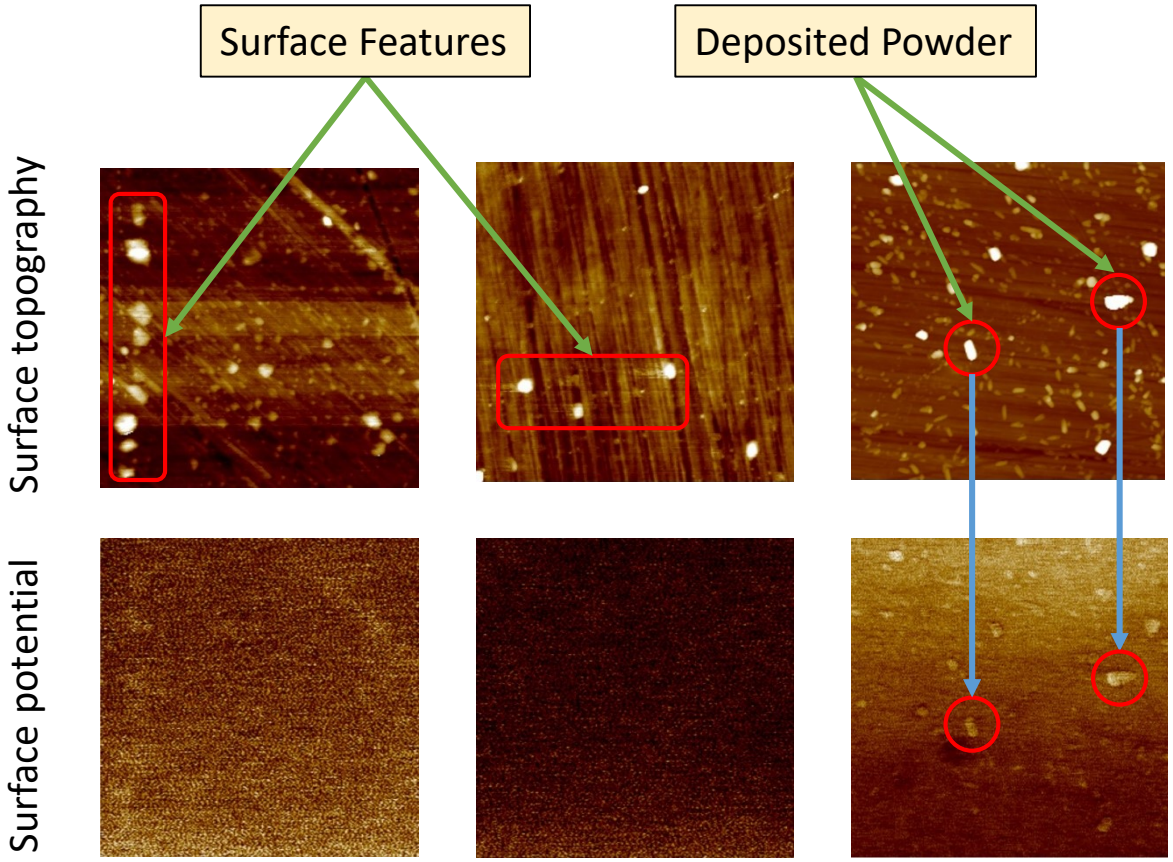
AFM: surface roughness and Kelvin Probe surface potential



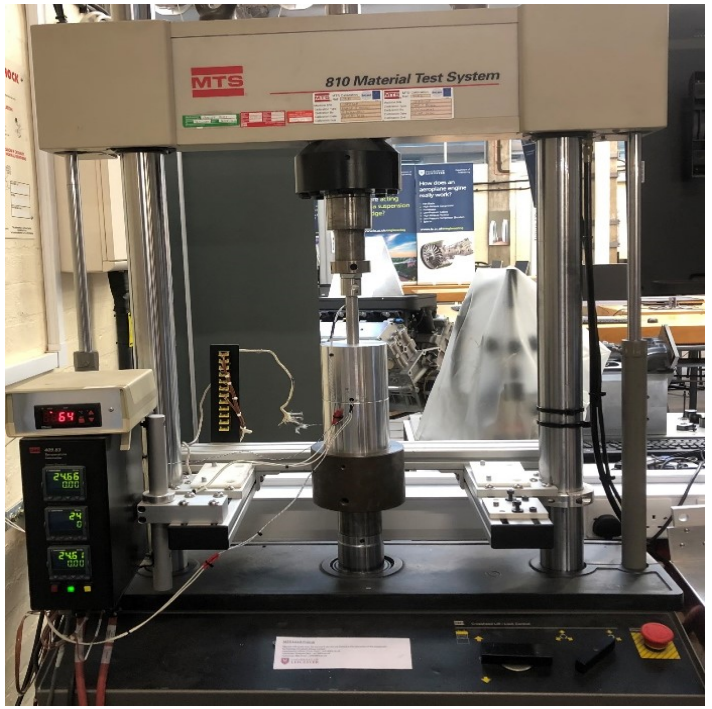
Conclusions:

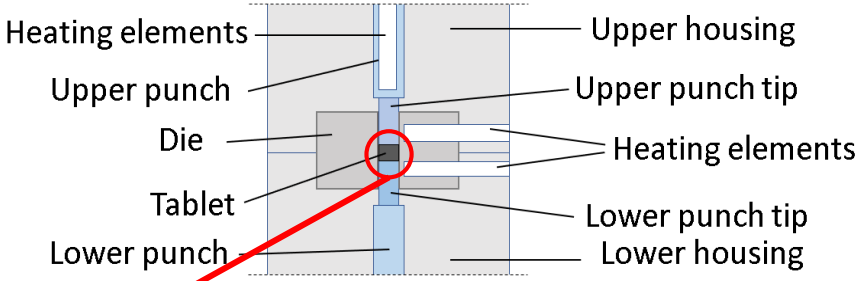
- A significant height variation (roughness), relative to the surface, is noticed in all three metal surfaces. These features are NOT powders deposited on the surface.
- The potential map shows a significant variation in the potential only in the 5-cycles sample. Therefore, the height variations in the 1-cycle sample correspond to surface features and not residues of powder.
- KPM is a potential characterization tool for powder depositions on the punch surfaces.

Sticking observations: room temperature and elevated temperature (50-60 °C)

Room Temperature	Pressure [MPa]	Thickness [mm]	Sticking behaviour
Ibuprofen	20	4.5	Hazing
	50	4.3	Minor sticking
	75	4.2	Hazing
	100	4.1	Minor sticking
	150	4.1	Minor sticking
	200	4.1	Minor sticking
	250	4.1	Minor sticking
Paracetamol	20	-	Tablet not formed
	50	-	Tablet not formed
	75	4.1	Weak tablet
	100	3.9	No sticking
	150	3.9	No sticking
	200	-	Lamination, electrostatic
	250	-	Lamination, electrostatic
Mannitol	20	-	Hazing
	50	-	Minor sticking
	75	-	Minor sticking
	100	-	Minor sticking
	150	-	Minor sticking
	200	-	Minor sticking
	250	-	Minor sticking

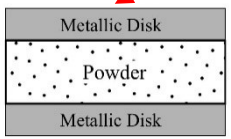
Elevated Temperature	Pressure [MPa]	Thickness [mm]	Sticking behaviour
Ibuprofen	20	4.5	Major sticking
	50	4.3	Major sticking
	75	4.2	Major sticking
	100	4.1	Major sticking
	150	4.1	Major sticking
	200	4.1	Major sticking
Paracetamol	75	4.1	Weak tablet
	100	3.9	Minor sticking
	150	3.9	Minor sticking
	200	-	Lamination
Mannitol	5	-	Attached particles
	20	-	Major sticking
	50	-	Major sticking
	75	-	Major sticking





Testing protocol

- Tablet weight: 400 mg, die diameter: 11 mm compaction rate 10 mm/min.
- Punch: clean; 1 compaction event.
- Cleaning protocol: isopropyl alcohol.
- Imaging: stereo microscope.



Leicester sticking classification

- No sticking: loose particles on surface
- Attached particles: removed with lab tissue
- Hazing: dull appearance, removed with lab tissue
- Severe hazing: solvent removal
- Minor sticking: particles or agglomerates, solvent removal
- Major sticking: blocks of material, solvent removal

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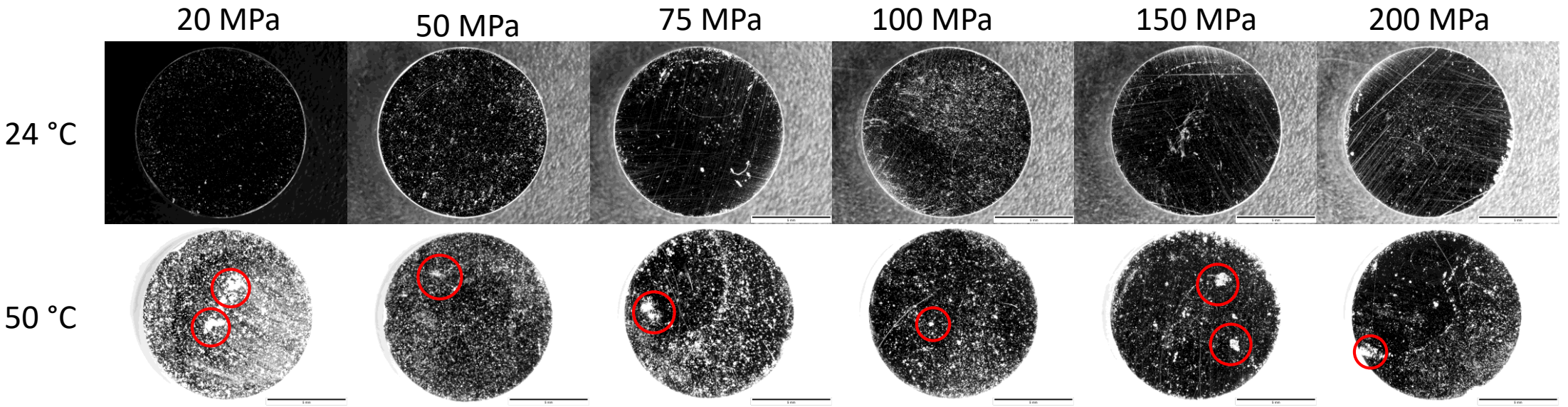
Sticking observations: **Ibuprofen** at room and elevated temperature

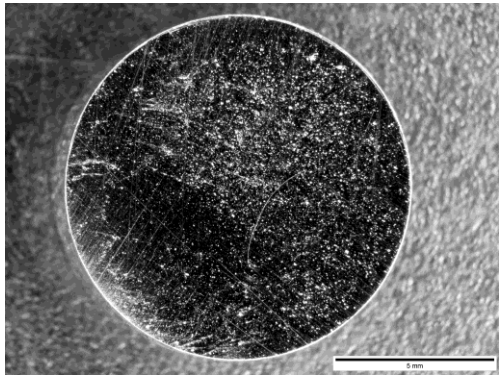


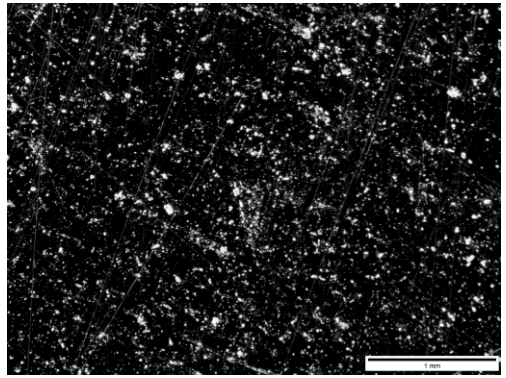
Conclusions:

- Sticking is observed for all compaction pressures at room and elevated temperatures.
- There is a clear increase in the amount of powder sticking at 50 °C compared to room temperature.
- At 50 °C large groups of particles (highlighted in red) are sticking. This feature is not observed at room temperature (assumed to be a consequence of extended plastic deformation).

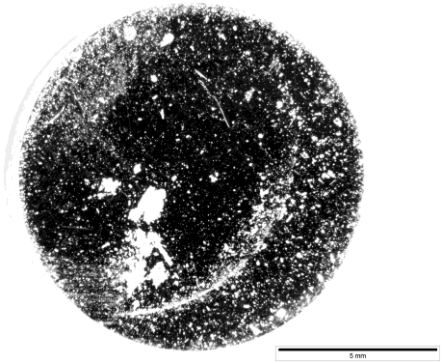
Sticking observations: Ibuprofen at room and elevated temperature

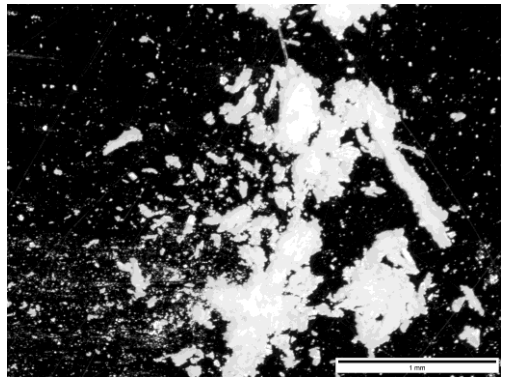
24 °C





50 °C

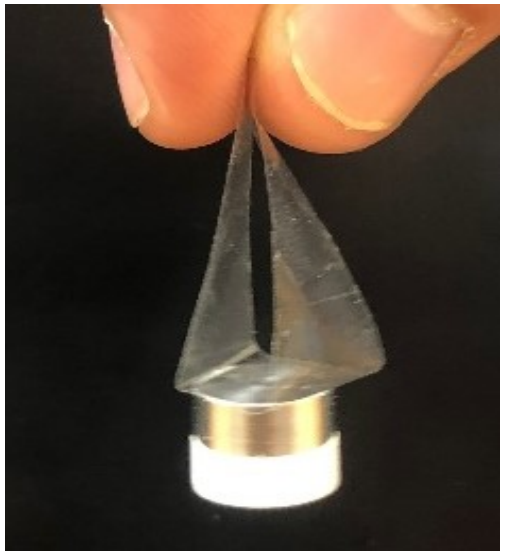




Observations/explanations

- The thermal conductivity of the powder is relatively low. The particles near the metal surface are at around 50 °C, while the particles inside the powder bed have lower temperature.
- Since the elastic modulus and yield strength decrease with temperature the particles near the metal surface experience more extensive elastic and plastic deformation. The powder deformation at less than 500 µm from the punch surface is expected to be non-uniform.

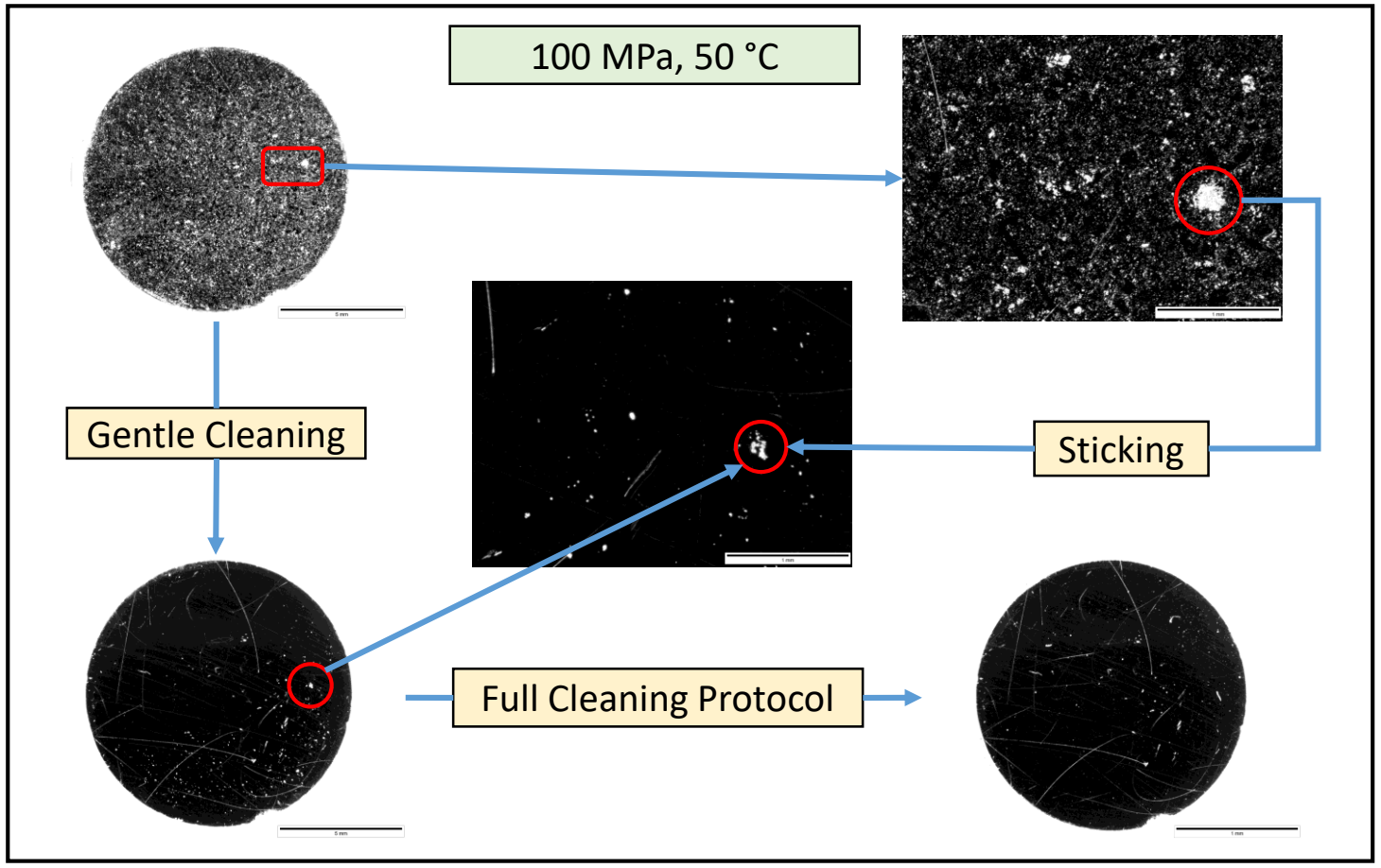
Sticking observations: **Mannitol** at 60 °C temperature

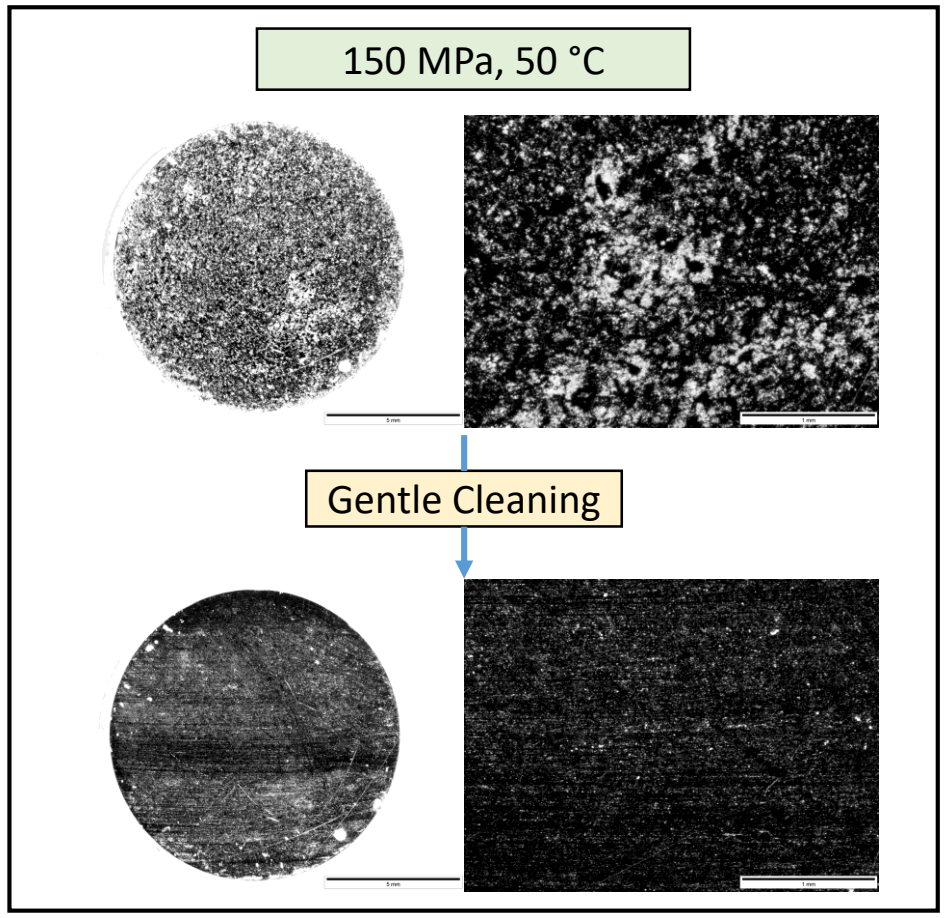
	5 MPa	20 MPa	50 MPa	75 MPa
60 °C				

Conclusions:

- There is no sticking at room temperature but at elevated temperature (60 °C) severe sticking is observed.
- At 20 MPa compaction pressure, the tablet is firmly attached to the punch and severe sticking is observed.
- As the compaction pressure is increased sticking becomes more severe requiring more aggressive cleaning.

Sticking observations: **paracetamol** at room and elevated temperature





Conclusions:

- Little or no sticking was observed for paracetamol at room temperature. However, at 50 °C, a small level of sticking was clearly identified (particles attached).
- Sticking for paracetamol at 50 °C is significantly less severe than ibuprofen and less severe than mannitol.

Effect of strain rate during compaction - Ibuprofen

Observations:

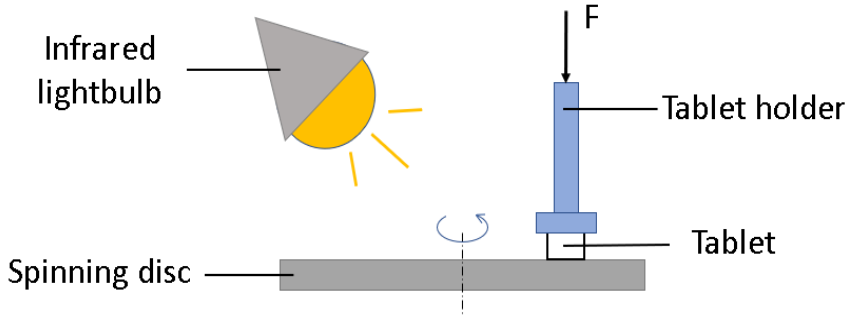
- Very severe sticking observed for Ibuprofen under hammer compaction at room temperature.
- Little or no literature data on strain rate effects on sticking (the processing conditions investigated in the literature are compaction pressure, dwell time and blending time).
- Further investigation is necessary.

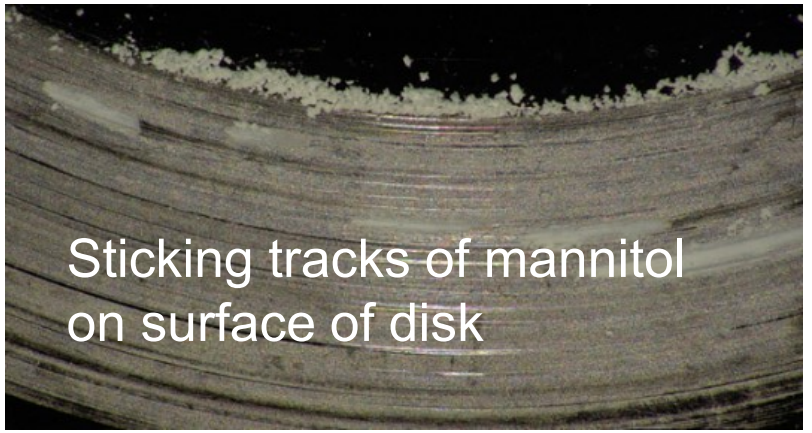
Summary and concluding remarks

- Completed literature review and prepared a review paper on sticking, to be submitted.
- Rationalised sticking hypotheses: temperature, breakage and humidity. Current focus on temperature.
- Developed trilayer punch-tablet system for systematic studies:
  - Ibuprofen: sticking at room and elevated temperature.
  - Mannitol at room temperature: sticking is influenced by compaction pressure and punch roughness. Further studies needed.
  - Mannitol at elevated temperature: sticking.
  - Paracetamol: little or no sticking at room and elevated temperature.
- KP AFM – method to observe sticking.
- Strain rate: identified as potential parameter responsible for sticking. Further studies needed.

Future work and guidance

- Materials tested so far: ibuprofen, paracetamol, mannitol. Materials to be tested: aspirin, maize starch, sorbitol, 2 lactose grades, microcrystalline cellulose.
- Measurement of trilayer interface strength – fixture design.
- Effect of strain rate: compaction with drop impact tester at Leicester. Compaction simulator tests at an industrial partner.
- Other analytical techniques to consider.
- PSD measurements – in progress.
- DSC – for materials where data is not available (e.g. croscarmellose sodium).
- AFM with particle probe to test against different steel surfaces.
- SEM/EDX of sticking of all materials.
- Rotary friction system – redesign.
- Investigation of particle breakage hypothesis.
- Investigation of humidity hypothesis.





Thank you for your **support** and **attention**!