



UNIMORE
UNIVERSITÀ DEGLI STUDI DI
MODENA E REGGIO EMILIA



ADHESION OF COLD SPRAY COATINGS: EFFECT OF SUBSTRATE ROUGHNESS AND HARDNESS

Gärtner F.²,

Bruera A.¹, Puddu P.¹, Theimer S.², Villa-Vidaller M.², Bolelli G.¹, List A.², Klassen T.², Lusvarghi L.¹

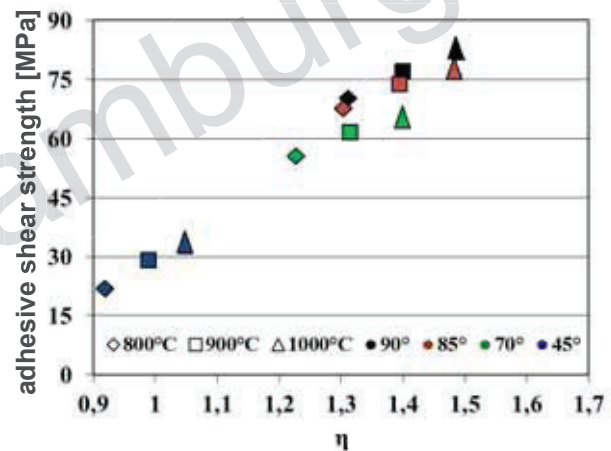
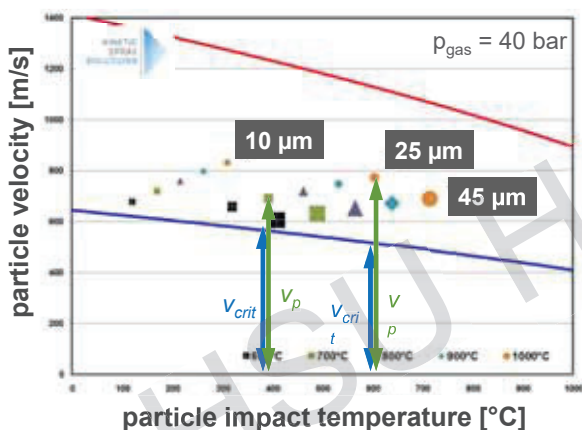
¹Department of Engineering "Enzo Ferrari", University of Modena and Reggio Emilia - Modena, Italy

²Institut für Werkstofftechnik, Helmut-Schmidt-Universität - Hamburg, Germany

Cold Spray Club, Hamburg, October 20th 2023

CS: Influences on adhesion

- amount of adiabatic shear instabilities (ASI): ratio $\eta = v_p / v_{crit}$



Ref.: K. Binder, „Kaltgasspritzten von ermüdungsfesten Titanschichten, PhD.-Thesis, Hamburg 2013

CS: Influences on adhesion



UNIMORE
UNIVERSITÀ DEGLI STUDI DI
MODENA E REGGIO EMILIA



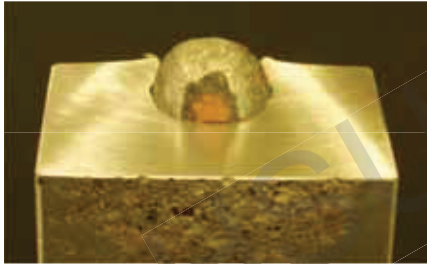
- **location of adiabatic shear instabilities (ASI):**

- relative hardness, ASI on particle or substrate sites

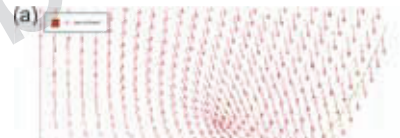
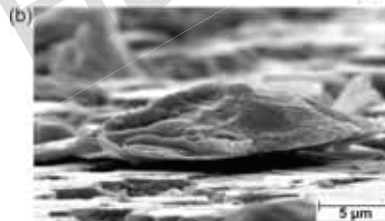
hard on soft, ASI on substrate sides

soft on hard, ASI on substrate sides

20 mm Cu ball on Al substrate,
 $v = 800\text{m/s}$



**Cu-particle on S-HVOF
sprayed Al_2O_3 layer.**



**2-d simulation: Cu on rigid
substrate, $v = 500\text{ m/s}$**

Process Science of Cold Spray, T. Klassen, F. Gärtner, H. Assadi; chapter 2 in: High Pressure Cold Spray: Principles and Applications (Editors: C.M. Kay and J. Karthikeyan), ASM International, ISBN: 978-1-62708-096-5, p. 17-66

CS: Influences on adhesion



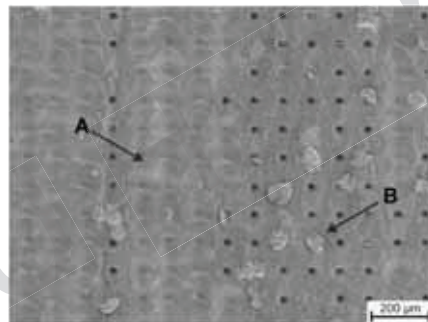
UNIMORE
UNIVERSITÀ DEGLI STUDI DI
MODENA E REGGIO EMILIA



- **Surface topographies: trapping particle ASI**

soft Cu on hard Al_2O_3 , ASI on particle sides,

- **adhesion only at locations of surface cavities (B), not smooth surface areas**

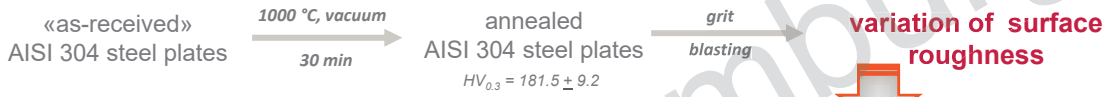


Ref.: K. Ernst, „Kaltgasspritzten für die Leistungselektronik – Werkstoffkombinationen und geometrische Randbedingungen, PhD.-Thesis, Hamburg 2021

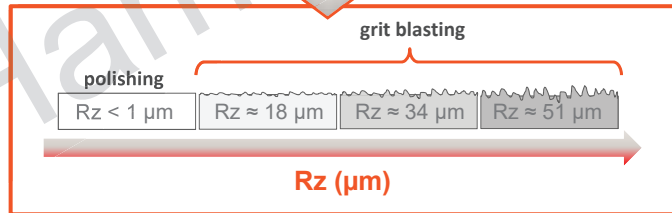
CS Influences on adhesion,
 ➤ **here: soft Cu on hard steel 304**



defined starting conditions



- **as grit blasted:**
 - **set of HARD SUBSTRATES**
- **additional vacuum annealing (30 min @ 1000°C):**
 - **set of SOFT SUBSTRATES**



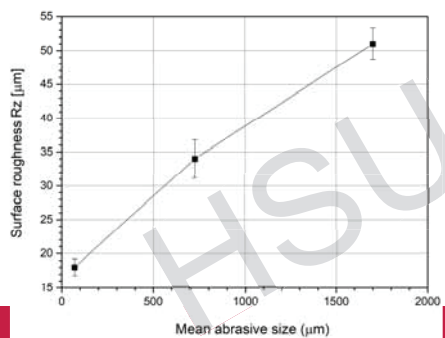
Surface & Coatings Technology 466 (2023) 129651

Surface roughness

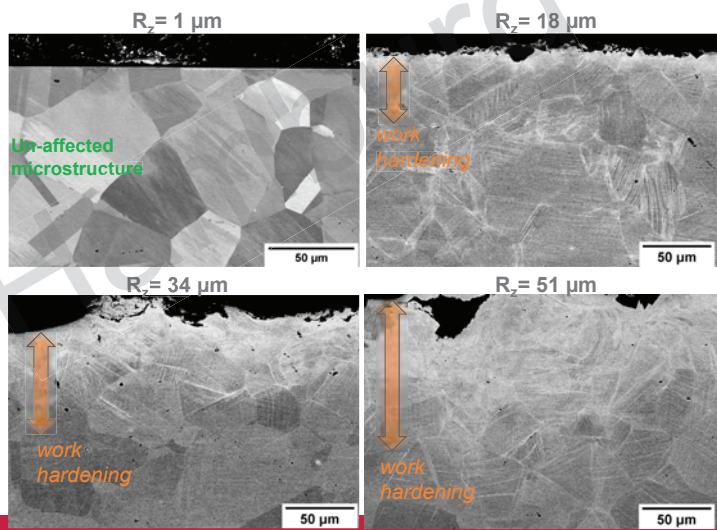


effect of grit size on substrate roughness

Grit	Size (μm)	P (MPa)	Rz (μm)
Corundum	46-90	0.8	18.1 ± 1.2
Corundum	600-850	0.8	34.0 ± 2.8
Corundum	1400-2000	0.8	51.2 ± 2.3



substrate microstructure and roughness (Set I)

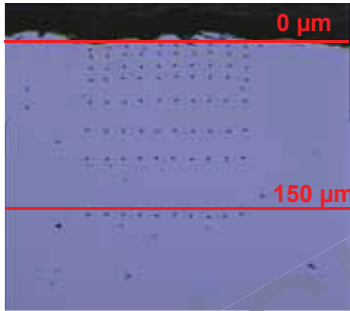


Surface & Coatings Technology 466 (2023) 129651

Surface hardness

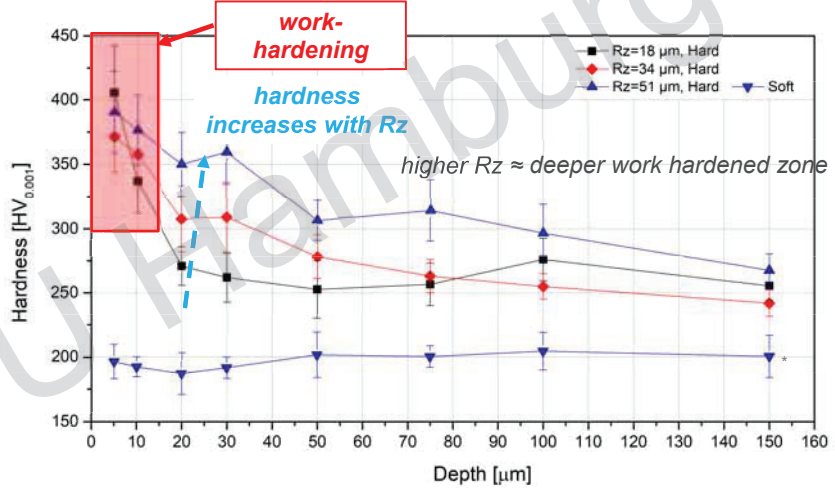


UNIMORE
UNIVERSITÀ DEGLI STUDI DI
MODENA E REGGIO EMILIA



10 indentations at different distance from the surface (depth)
Max load: 10 mN

substrate nano-hardness profiles



*Steel plates hardness before surface treatments: $HV_{0.3} = 181.5 \pm 9.2$

Surface & Coatings Technology 466 (2023) 129651

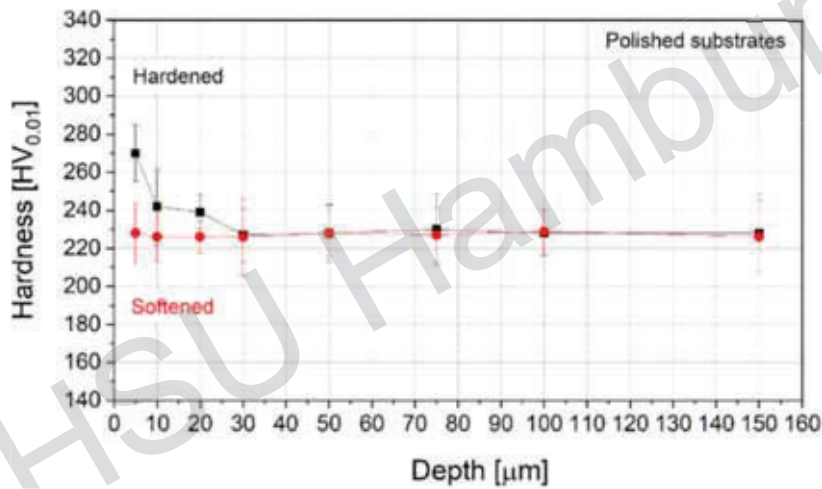
Surface hardness



UNIMORE
UNIVERSITÀ DEGLI STUDI DI
MODENA E REGGIO EMILIA



substrate hardness profiles: polished surfaces



Surface & Coatings Technology 466 (2023) 129651

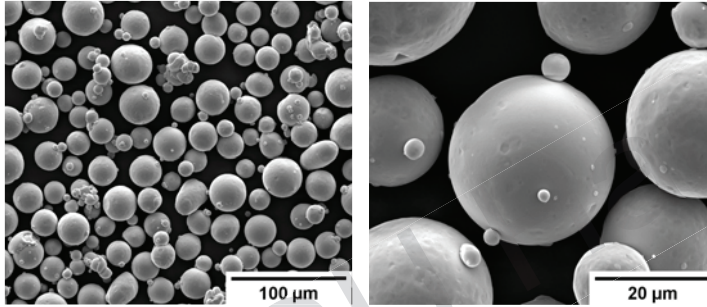
Cu powder feedstock



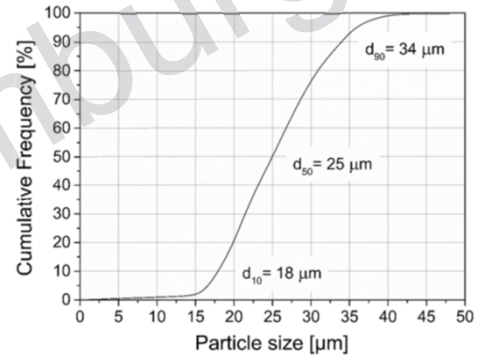
UNIMORE
UNIVERSITÀ DEGLI STUDI DI
MODENA E REGGIO EMILIA



particle morphologies



particle size distribution



Surface & Coatings Technology 466 (2023) 129651

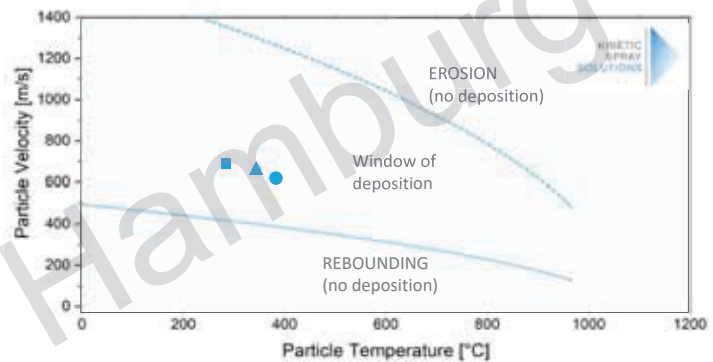
CS process parameters



UNIMORE
UNIVERSITÀ DEGLI STUDI DI
MODENA E REGGIO EMILIA

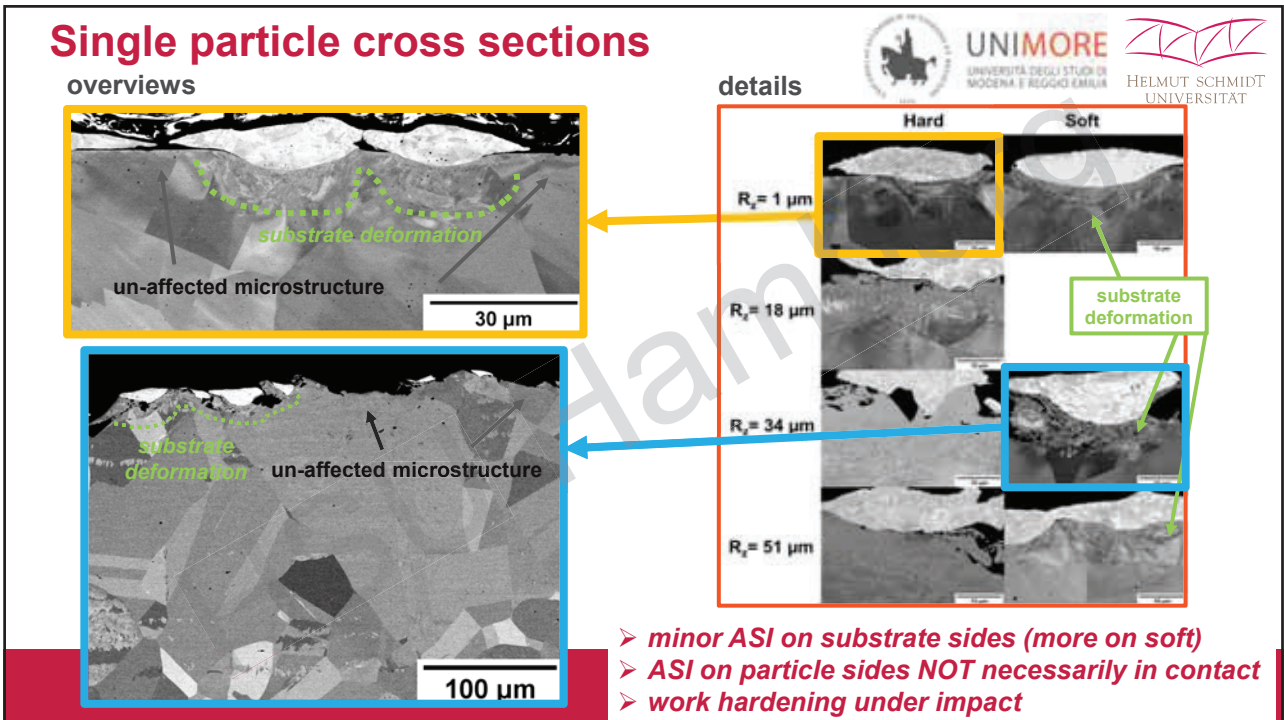
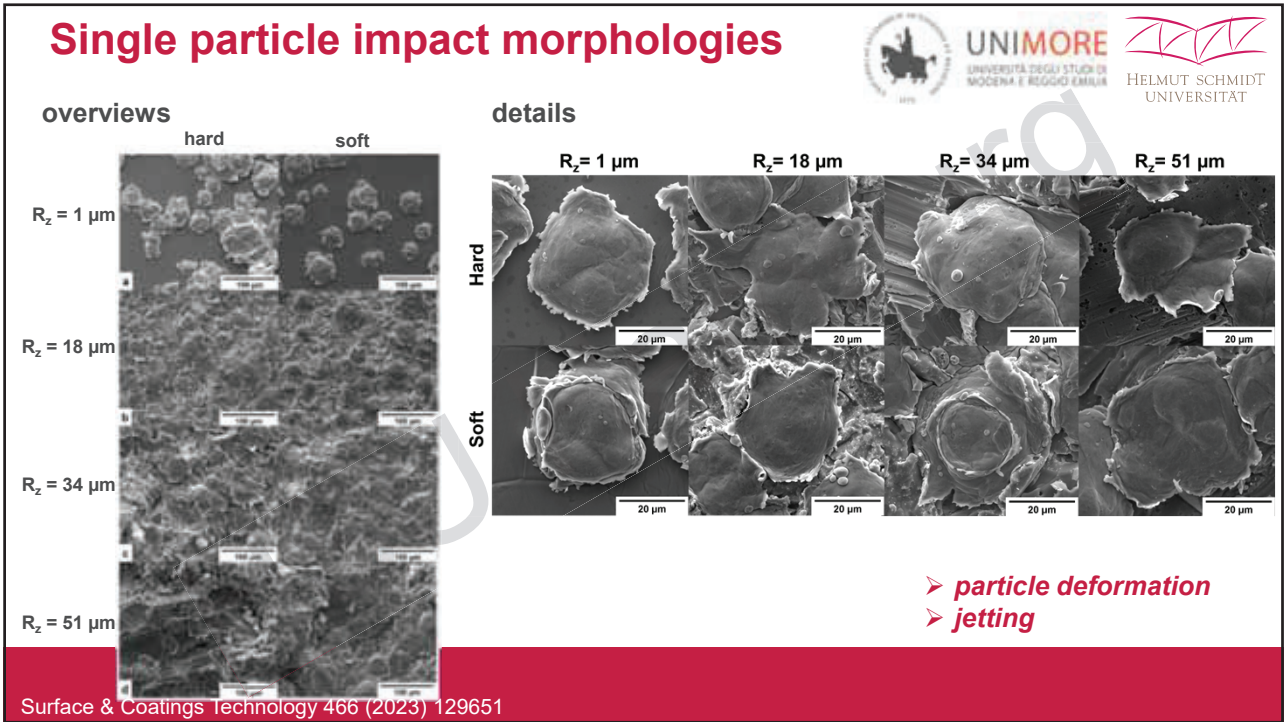


Parameters	Values
Equipment	K8000 X
Powder feeder	PF 4000
Gas	N ₂
Gas temperature	600 °C
Gas pressure	40 bar
Stand-off distance	60 mm
Pre-chamber	60 mm
Nozzle	D24WC



- Ø 18µm – 619m/s, 272°C, $\eta(=v/v_{crit})$: 1.48
- ▲ Ø 25µm – 592m/s, 333°C, η : 1.56
- Ø 34µm – 592m/s, 367°C, η : 1.60

Surface & Coatings Technology 466 (2023) 129651



Single particle removal by cavitation

The schematic shows a cold spray nozzle with an angle of 25° and frequency of 20 kHz, spraying particles into a chamber containing a specimen. The chamber is equipped with a heater, thermopile, and a water bath at 20°C. The specimen is held in a holder with a diameter of 16.5 ± 0.05 mm and a thickness of 4 mm. The distance between the nozzle and the specimen is 0.5 mm. The chamber is connected to a vacuum system with a pressure of 0.1 Torr and a flow rate of 0.4 l/min. The chamber is also equipped with a thermopile and an outlet.

The SEM images show the state of particles on the substrate at 60 s and 180 s for both hardened and softened conditions. The images are labeled a, a', b, b' for 60 s and c, c', d, d' for 180 s. The images show that particles are more easily removed from the softened substrate compared to the hardened substrate. Red arrows point to the traces of ASI (Adhesive Interfacial Structure) after particle removal.

UNIMORE UNIVERSITÀ DEGLI STUDI DI MODENA E REGGIO EMILIA
 HELMUT SCHMIDT UNIVERSITÄT

Surface & Coatings Technology 466 (2023) 129651

Single particle removal by cavitation

resistance against removal
 ➤ measure for single particle adhesion

The graph plots the percentage of residual bonded particles (Y-axis, 0 to 100) against cavitation time in seconds (X-axis, 0 to 300). The data is categorized by substrate roughness (R_a) and hardness (Hard/Soft). The legend indicates: Hard, R_a = 1 μm; Hard, R_a = 18 μm; Hard, R_a = 34 μm; Hard, R_a = 51 μm; Soft, R_a = 1 μm; Soft, R_a = 18 μm; Soft, R_a = 34 μm; Soft, R_a = 51 μm. The graph shows that the percentage of residual bonded particles decreases over time for all conditions. Higher roughness and hardness generally result in higher resistance against removal, leading to a higher percentage of residual particles at any given time.

UNIMORE UNIVERSITÀ DEGLI STUDI DI MODENA E REGGIO EMILIA
 HELMUT SCHMIDT UNIVERSITÄT

➤ influences by surface topographies
 ➤ influences by surface hardness

Surface & Coatings Technology 466 (2023) 129651

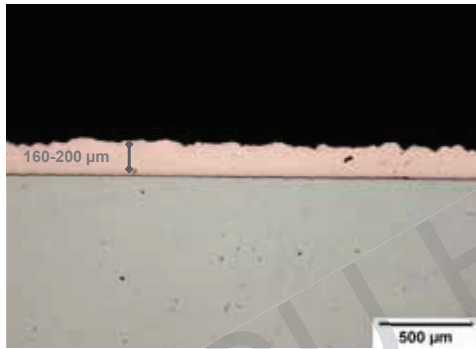
Full coatings: microstructures



UNIMORE
UNIVERSITÀ DEGLI STUDI DI
MODENA E REGGIO EMILIA



1 layer



10 layers



- deposition efficiency > 90%
- porosity < 0.5%
- $HV_{0.3} = 135-145$

Surface & Coatings Technology 466 (2023) 129651

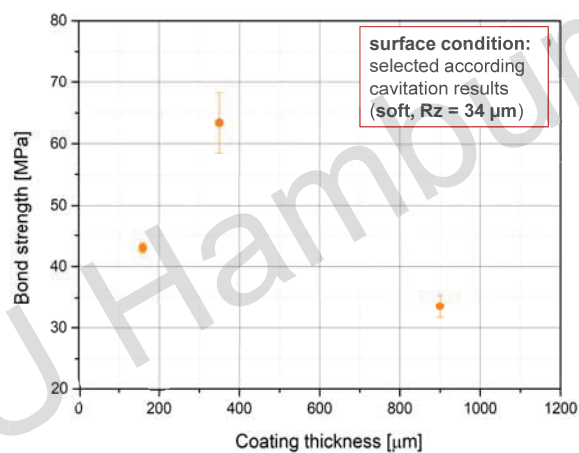
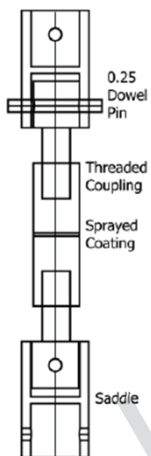
Full coating adhesion: f(thickness)



UNIMORE
UNIVERSITÀ DEGLI STUDI DI
MODENA E REGGIO EMILIA



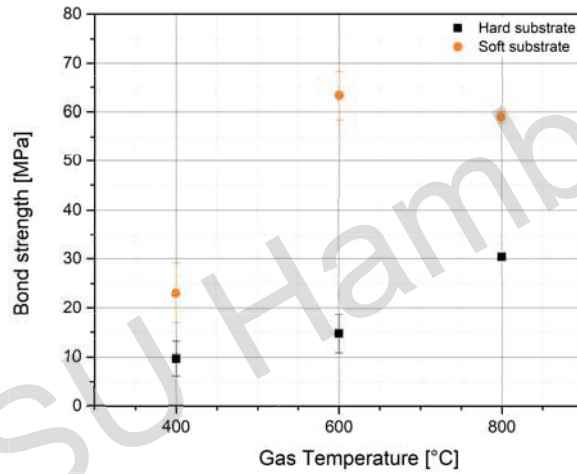
bond strength:
according to DIN EN 10002



- thicker coatings: influences by internal stresses
- optimum thickness ~ 350 μm

Surface & Coatings Technology 466 (2023) 129651

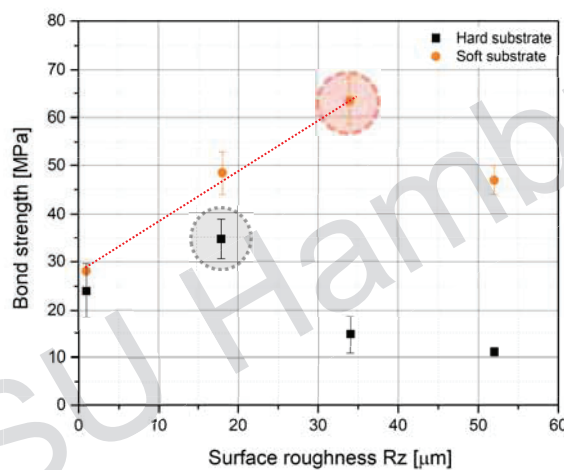
Full coating adhesion: $f(\eta)$



➤ *bond strength ~ process conditions*

Surface & Coatings Technology 466 (2023) 129651

Full coating adhesion: $f(\text{roughness})$



➤ *optimum roughness ~ $f(\text{surface hardness})$*

➤ *optimum roughness (coating) ~ single particle adhesion*

Locations of failure after BS-test

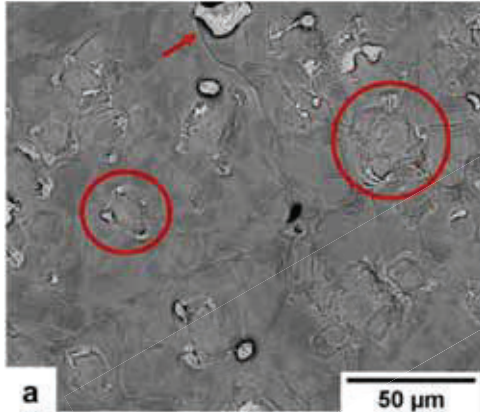
Cu-residues



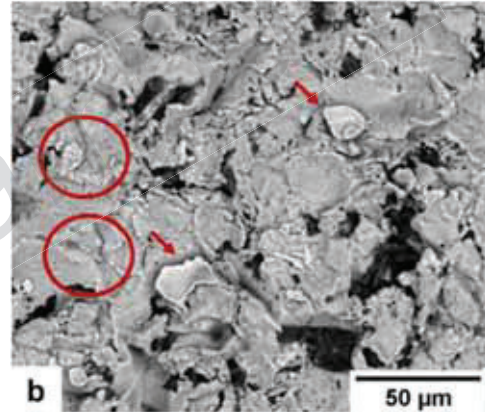
UNIMORE
UNIVERSITÀ DEGLI STUDI DI
MODENA E REGGIO EMILIA



smooth surface (hard)



rough surface (hard)



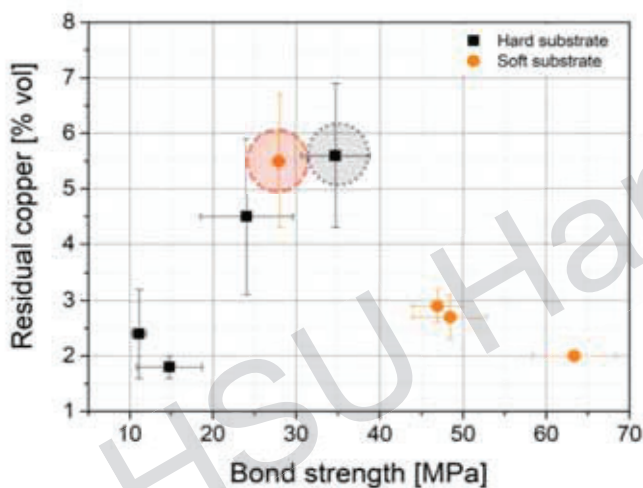
➤ more Cu-residues on rough surface

Locations of failure after BS-test

Cu-residues (by EDS)



UNIMORE
UNIVERSITÀ DEGLI STUDI DI
MODENA E REGGIO EMILIA

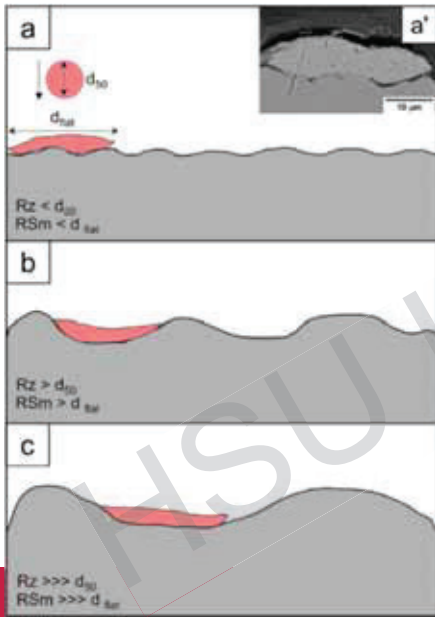


- **hard substrates:**
 - amount of Cu increases with BS
 - ASI on Cu-sides
- **soft substrates:**
 - amount of Cu shows maximum
 - ASI on Cu-sides + ASI on steel-sides

Conditions for ASI in contact



UNIMORE
UNIVERSITÀ DEGLI STUDI DI
MODENA E REGGIO EMILIA



too fine surface topographies to trap ASI

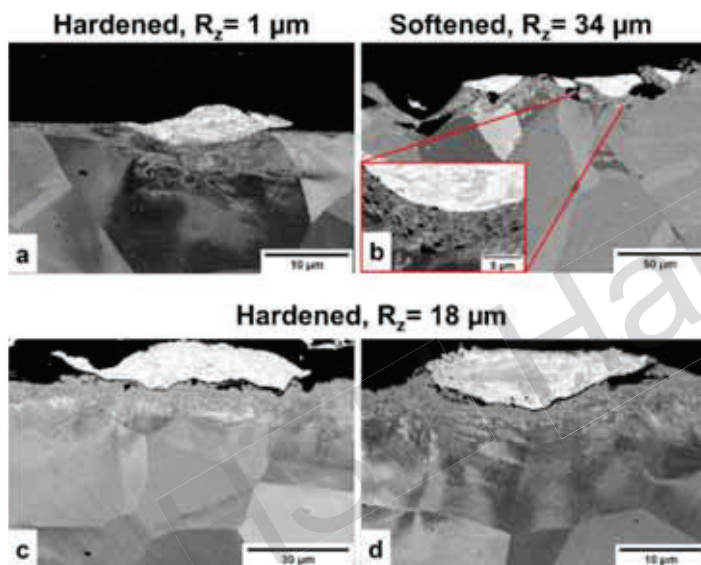
ideal surface topographies to trap ASI

*too coarse surface topographies to trap ASI
(similar to flat surface)*

Locations of ASI, single particle events



UNIMORE
UNIVERSITÀ DEGLI STUDI DI
MODENA E REGGIO EMILIA



but

- roughness

- ASI in contact
- ASI location
- on soft: common ASI on Cu and steel sites

Take home messages: CS adhesion



UNIMORE
UNIVERSITÀ DEGLI STUDI DI
MODENA E REGGIO EMILIA



- **ideal:**

- same particle and substrate deformation, common ASI

- **hard on soft:**

- substrate deformation determines topography and ASI on substrate sites

- **soft on hard:**

- ASI on particle sites must be trapped in suitable surface topographies
- suitable surface topographies depend on particle deformation, flattening

- **substrate softening:**

- enabled surface deformation
- common ASI:
 - better BS
 - wider spread for needed surface topographies

Folie 23

many thanks for your kind attention!

thanks to the team:

Zahra Arabgol
Thomas Breckwoldt
Alessia Bruera
Andreas Elsenberg
Julio Gutierrez
Chunjie Huang
Caroline Hahn
Matthias Hartmann
Thomas Klassen
Marion Kollmeier
Marcel Lewke
Alexander List
Emma Morales
Sören Nielsen
Bastian Oswald
Camilla Schulze
Matthias Schulze
Farrokh Taherkhani
Levke Wiehler
Honjian Wu

