Optimization of corrosion protection properties of waterborne 2C epoxy clear coats with Neuburg Siliceous Earth

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  Application viscosity

  Flexibility and hardness

    - Determination of cupping test and cross cut test

  Corrosion resistance

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    - Salt spray test (delamination at scribe)
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- Appendix

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Status Quo

- Coatings for corrosion protection with sufficient preserving properties could be produced so far only with the aid of anticorrosive pigments.
- If using these pigments an application as clear coat is impossible.

➢ Feasibility for achieving both attributes by using Neuburg Siliceous Earth?

Objective

Following requirement profile should be fulfilled by Neuburg Siliceous Earth:

- Enough flexibility and outstanding adhesion on different substrates.
- Good transparency.
- No milky blushing of the clear coat after exposure to humidity.
- Improvement of the corrosion protection properties.

Filler Characteristics

Filler	Description	Surface Treatment
Sillitin Z 89	Neuburg Siliceous Earth d_{50}: 1.8 µm, d_{97}: 7.1 µm	none
Silfit Z 91	Calcined Neuburg Siliceous Earth d_{50}: 2.0 µm, d_{97}: 8.6 µm	none
Aktisil AM	Neuburg Siliceous Earth d_{50}: 2.2 µm, d_{97}: 10 µm	amino functionalized
TP 2008037	Neuburg Siliceous Earth (Experimental product similar to Aktisil AM, but more color neutral) d_{50}: 2.2 µm, d_{97}: 8.3 µm	amino functionalized

Filler Characteristics

Filler	Oil absorption [g/100g]	Specific surface area BET [m²/g]
Sillitin Z 89	55	11
Silfit Z 91	55	8
Aktisil AM	45	9
TP 2008037	61	9

Filler Characteristics

Filler	Color L*	Color a*	Color b*
Sillitin Z 89	94.7	-0.1	3.4
Silfit Z 91	95.3	-0.2	0.9
Aktisil AM	93.0	0.5	8.1
TP 2008037	94.8	-0.1	3.4

Base Formulation

	Description	Parts by weight
A-Component: BECKOCURE™ EH 2260w/41WA *1	Amine hardener	61.1
B-Component: BECKOPOX™ EP 147w *1 BECKOPOX™ EP 386w/52WA *1	Epoxy resin	12.5 37.5
Total		111.1
Solids content [%]		51.4

*1 http://allnex.com/the-easy-cure-system

Formulations

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Parts by weight

	Control	Sillitin Z 89 15 pbw	Silfit Z 91 15 pbw	Aktisil AM 15 pbw	Aktisil AM 25 pbw	TP 2008037 25 pbw
A-Component (Amine hardener)	61.1	61.1	61.1	61.1	61.1	61.1
+ Filler	0	15	15	15	25	25
B-Component	50	50	50	50	50	50
Total	111.1	126.1	126.1	126.1	136.1	136.1
Solids content [%]	51.4	57.1	57.1	57.1	60.3	60.3
PVC [%]	0	9.9	9.9	9.9	15.5	15.5

Formulations

Parts per cent

	Control	Sillitin Z 89 15 pbw	Silfit Z 91 15 pbw	Aktisil AM 15 pbw	Aktisil AM 25 pbw	TP 2008037 25 pbw
A-Component (Amine hardener)	55	48.45	48.45	48.45	44.90	44.90
+ Filler	0	11.90	11.90	11.90	18.37	18.37
B-Component	45	39.65	39.65	39.65	36.73	36.73
Total	100	100	100	100	100	100
Solids content [%]	51.4	57.1	57.1	57.1	60.3	60.3
PVC [%]	0	9.9	9.9	9.9	15.5	15.5

Preparation / Application / Substrates / Drying

Preparation:	Dissolver with pearl mill APS 1000
Application:	Compressed air, Walther Pilot spray gun, nozzle diameter 2 mm, approx. 1.7 bar
Substrate:	Aluminum: Gardobond F, Steel: Gardobond OC, both without any surface treatment
Drying:	30 min at 60 °C
Dry Film Thickness:	50-80 µm
Testings:	After 7 days at 23 °C / 50 % rH

Adjustment of Application Viscosity

Component A and B mixed, plus water for dilution

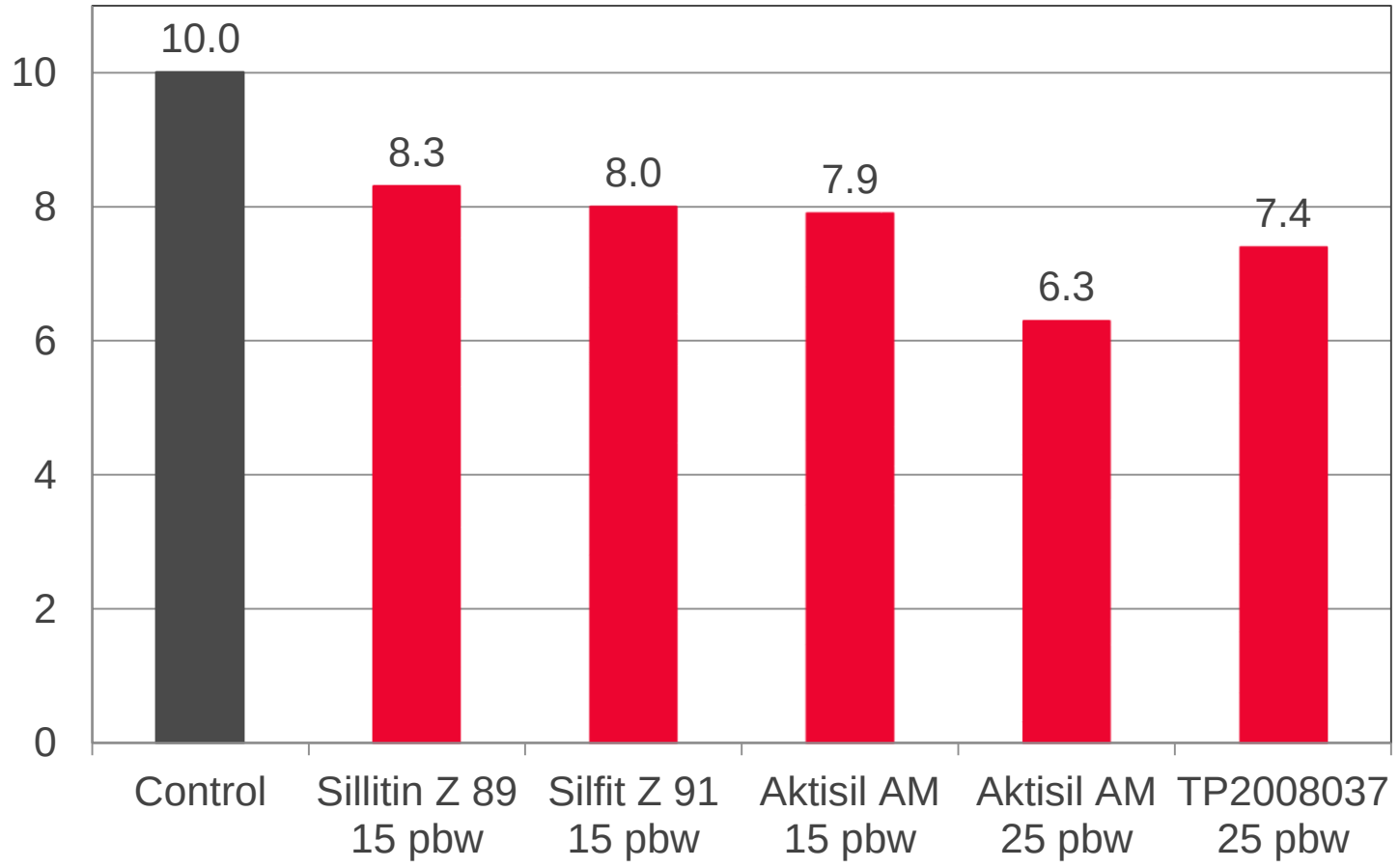
	Control	Sillitin Z 89 15 pbw	Silfit Z 91 15 pbw	Aktisil AM 15 pbw	Aktisil AM 25 pbw	TP 2008037 25 pbw
Water [%]	Setpoint ~ 3.0 Real value 3.8	4.7	5.0	Setpoint ~ 5.0 Real value 6.8	Setpoint ~ 7.5 Real value 5.6	Setpoint ~ 7.5 Real value 4.9
Resulting viscosity at 25 s^{-1} [Pa*s] *1	1.5	1.9	1.8	1.4	2.6	2.4
Solids content [%]	49.5	54.6	54.4	53.5	57.1	57.5

*1 Intended application viscosity at 25 s^{-1}: 1.8 – 2.0 Pa*s

Cupping Test

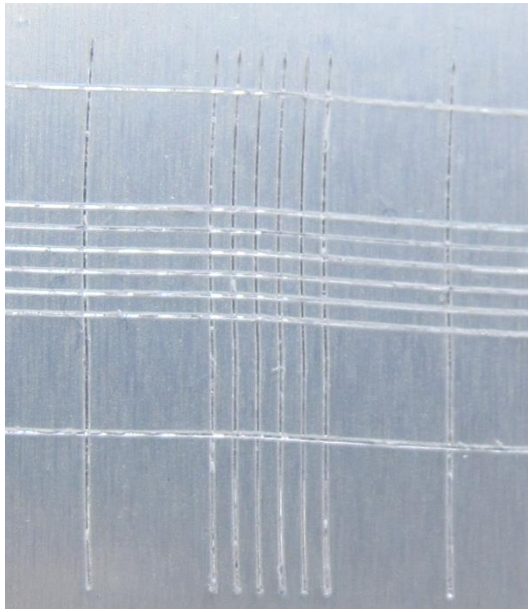
Steel

[mm]



Cross Cut Test (1mm)

Steel and aluminum



All formulations showed excellent adhesion to the substrate with GT 0

Humidity Test 240 h

DIN EN ISO 6270-2 CH

Evaluation of:

– Degree of Blistering DIN EN ISO 4628-2

– Degree of Rusting DIN EN ISO 4628-3

– Transparency / milky blushing by measuring Delta E

Humidity Test 240 h
Blistering and Rusting

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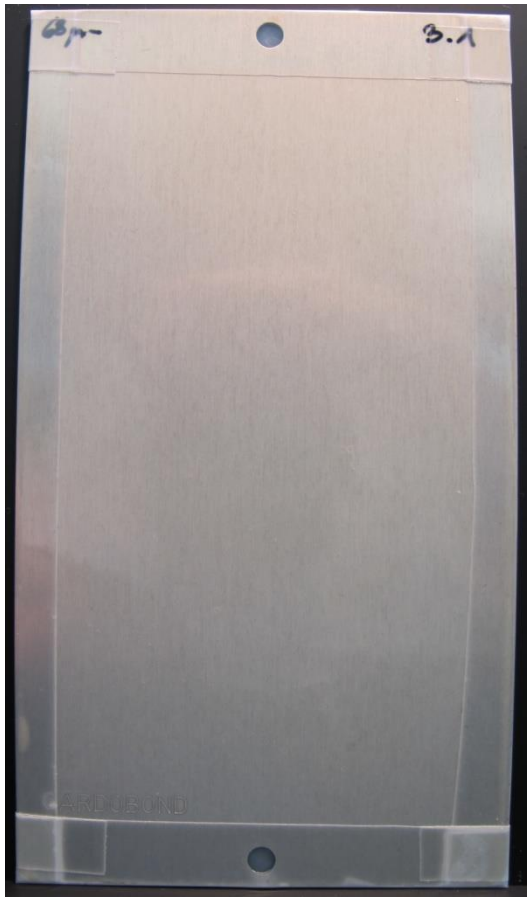
Steel and aluminum



All formulations showed
no blistering and
no rusting = Ri 0

Humidity Test 240 h
Transparency Delta E

Steel

Control	Silfit Z 91
	
Delta E: 6.6	Delta E: 2.5
Strong milky blushing, marked negative impact to transparency	Significant Improvement

Humidity Test 240 h
Transparency Delta E

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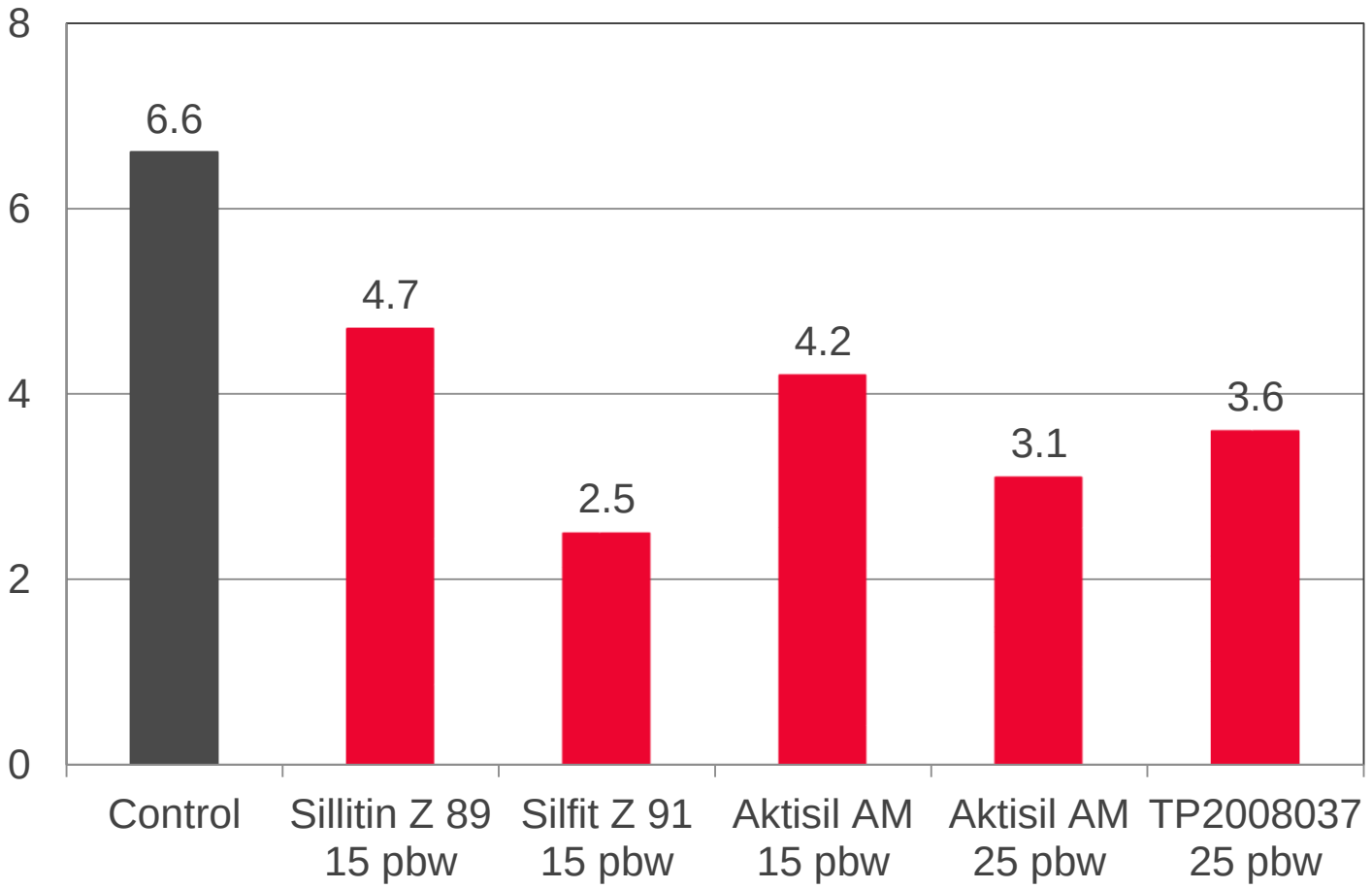
Steel



Salt Spray Test 240 h

DIN EN ISO 9227

Scribed with a scratching tool according to van Laar

Evaluation of:

– Delamination at scribe DIN EN ISO 4628-8

Salt Spray Test 240 h
Delamination

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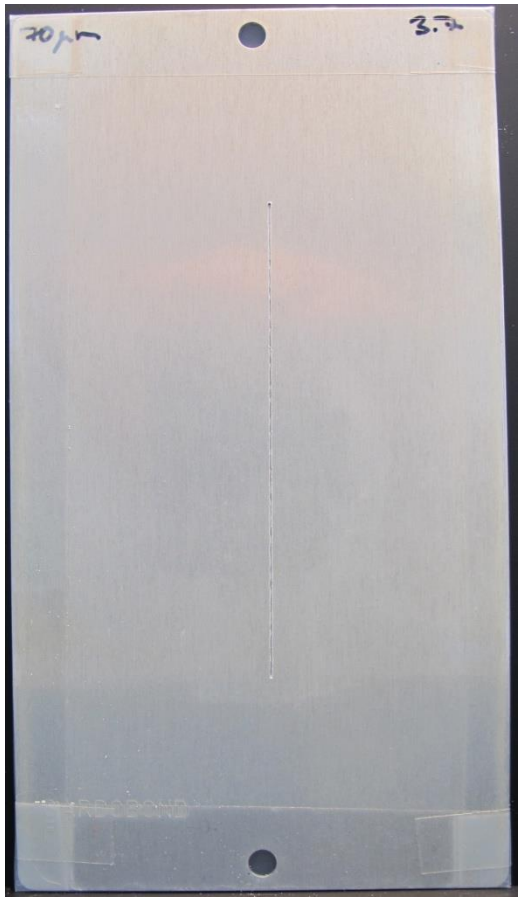
Aluminum



All formulations

no delamination = 0 mm

Salt Spray Test 240 h
Delamination

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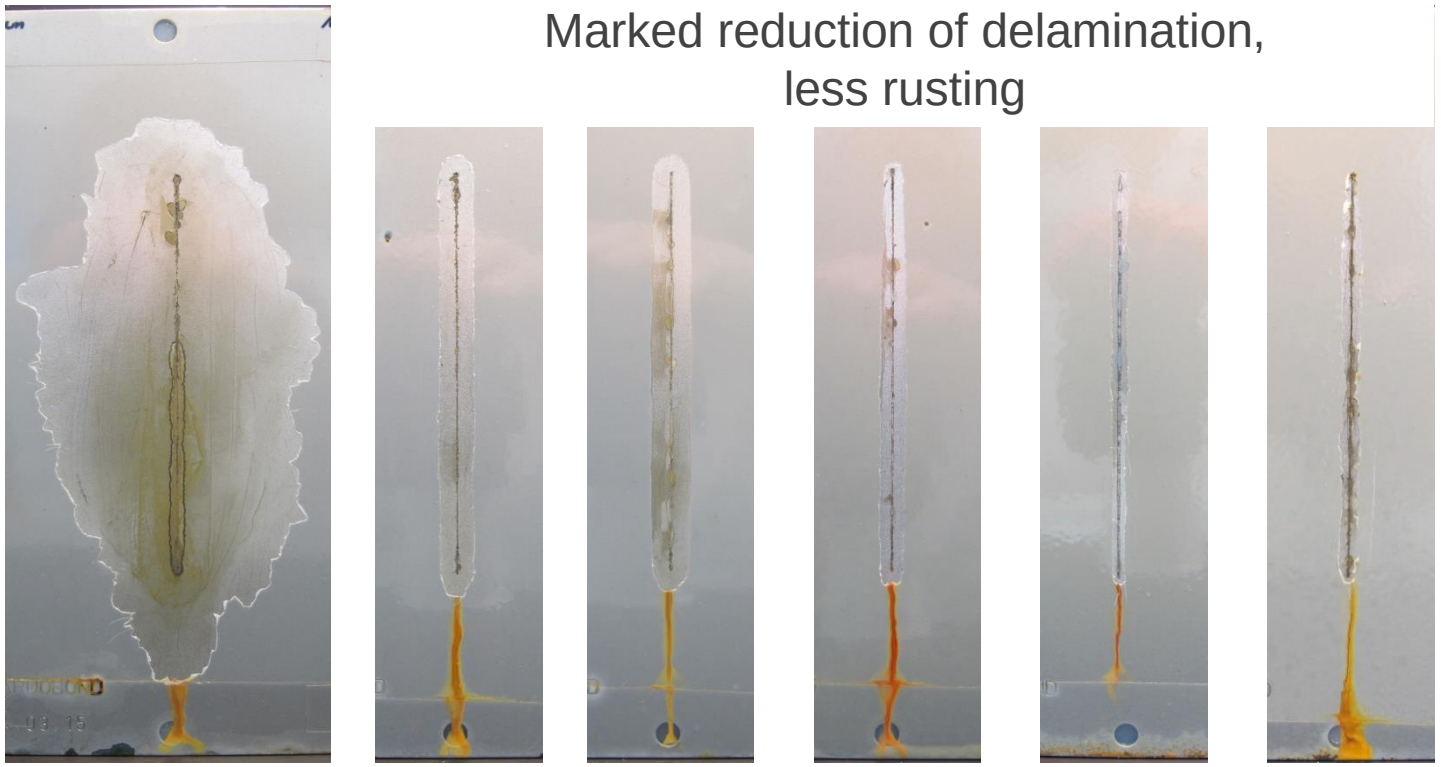
Steel

Marked reduction of delamination, less rusting



Control	Sillitin Z 89 15 pbw	Silfit Z 91 15 pbw	Aktisil AM 15 pbw	Aktisil AM 25 pbw	TP2008037 25 pbw

Salt Spray Test 240 h
Delamination

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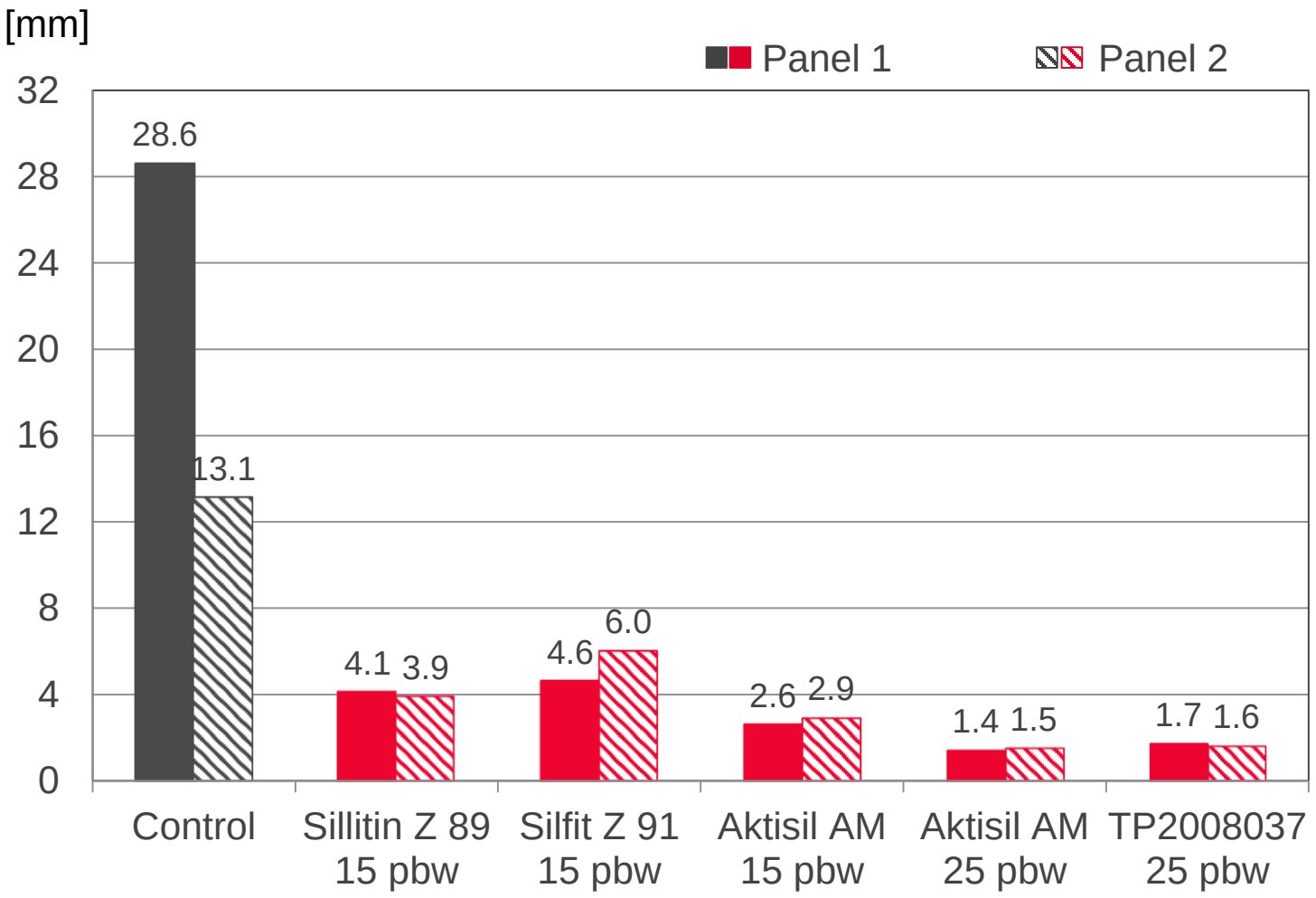
Steel



Further Results in the Appendix

- General information (preparation of the formulations)
- Optical properties
  - Color L* a* b*
  - Gloss
- Mechanical properties
  - Pendulum hardness (König)
- Corrosion resistance
  - Humidity test, adhesion and gloss
  - Salt spray test, gloss and color change

Summary

A waterborne 2C-Epoxy Clear Coat with Neuburg Siliceous Earth provides the following quality profile:

- Transparency and color neutrality, especially by using Silfit Z 91
- High gloss (60°: > ~ 80)
- Excellent adhesion to the substrate (GT 0-1)
- Good flexibility (cupping test 6-8 mm)
- Best price/performance ratio, especially by using Sillitin Z 89

Summary

A waterborne 2C-Epoxy Clear Coat with Neuburg Siliceous Earth provides the following quality profile:

- No milky blushing of the clear coat after exposure to humidity, maintaining transparency and color neutrality over the time, especially by using Silfit Z 91 with 15 pbw.
- Significant improvement of corrosion resistance: by using Aktisil AM oder the more color neutral TP 2008037 the delamination at scribe is nominal. This optimization is obviously visible in the PVC raised version (25 pbw).

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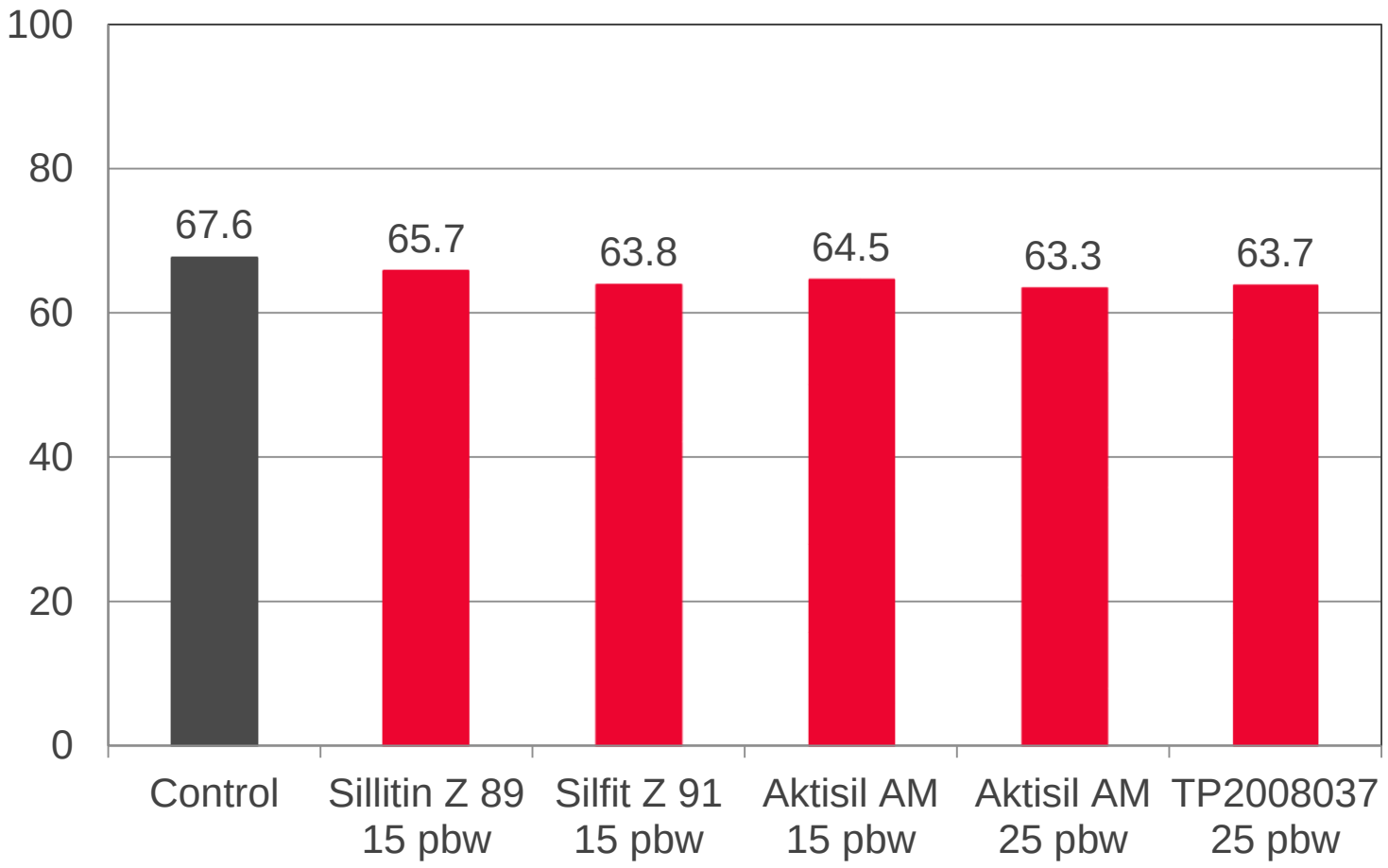
Preparation

- Pearl mill APS 1000 (with glass beads 2mm)
- Batch size A-component approx. 350g ≙ 290mL
- Milling:10 min at 2000 rpm ≙ 7,9 m/s, with water cooling
  → temperature approx. 30 °C

Color
Brightness

CIE L*, steel



Color
Red/green ratio

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INTRODUCTION

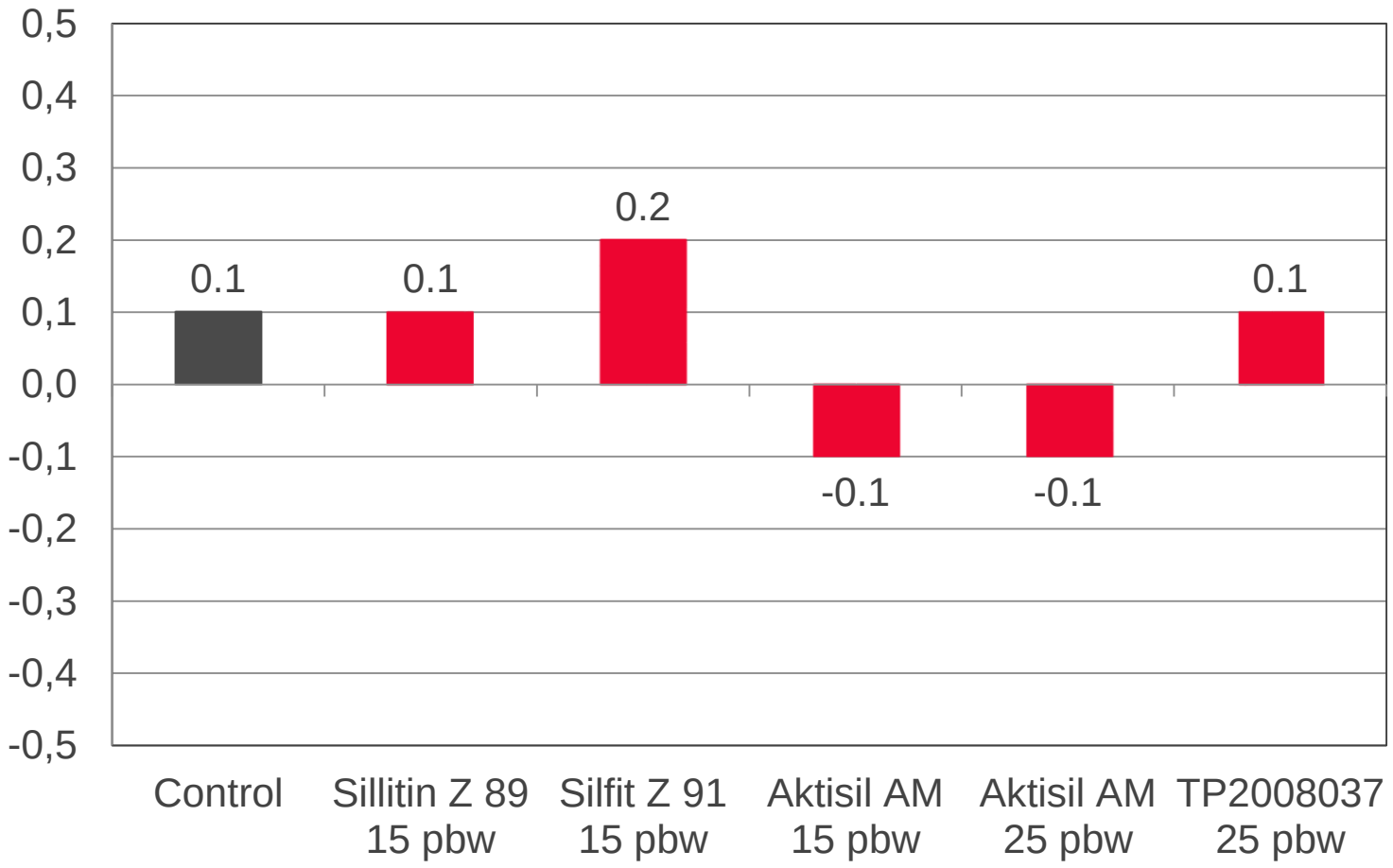
EXPERIMENTAL

RESULTS

SUMMARY

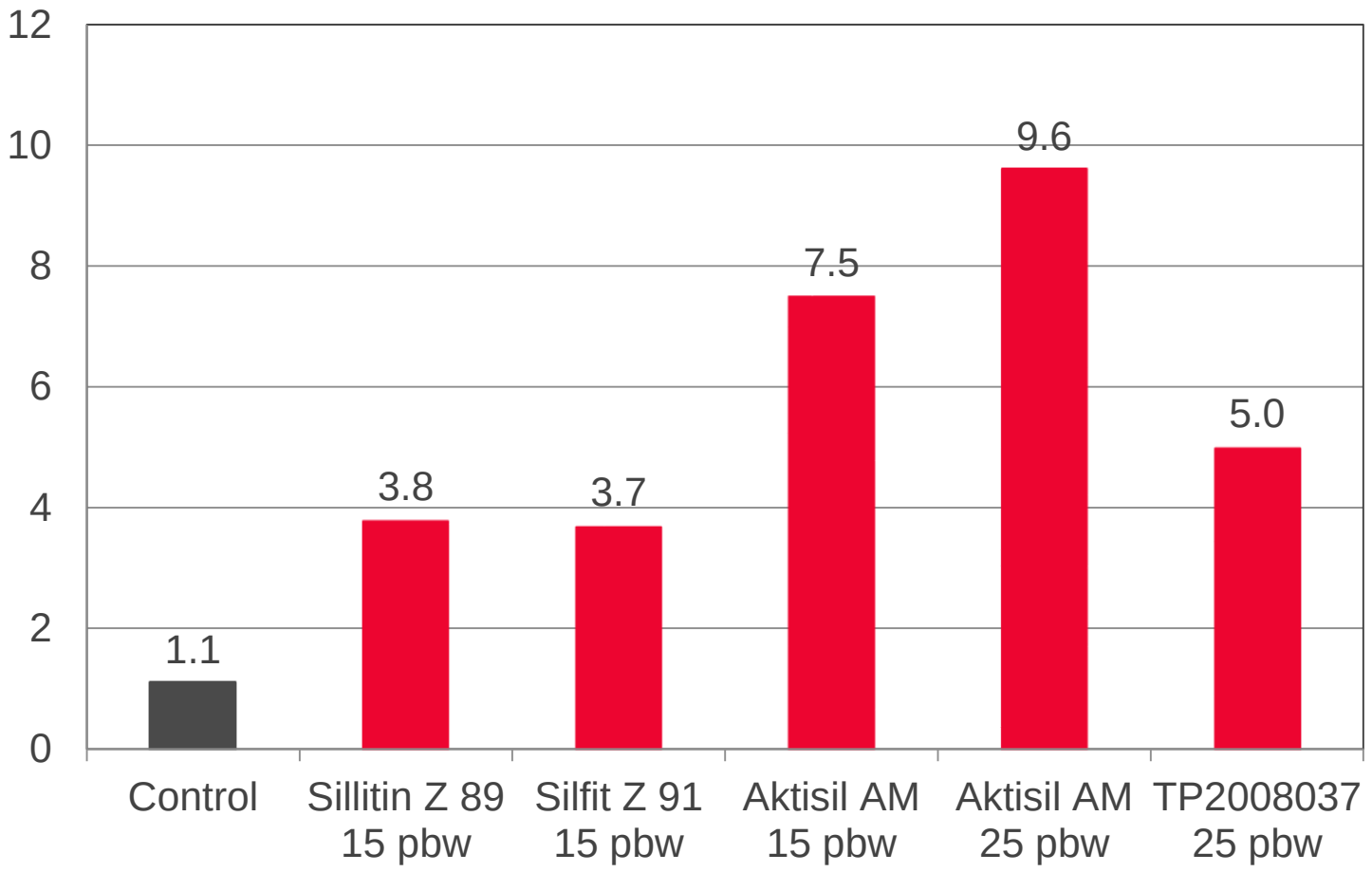
APPENDIX

CIE a*, steel

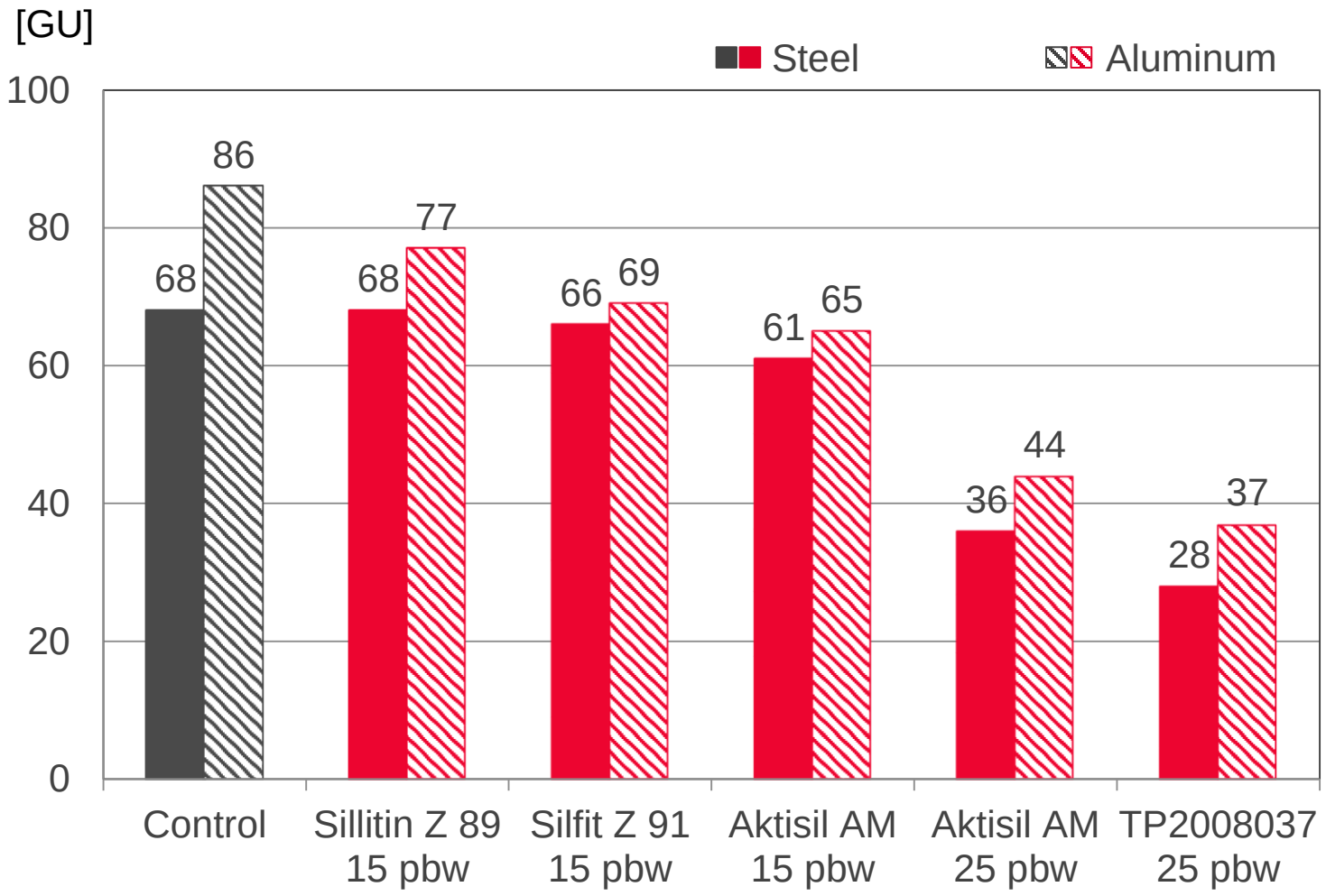


Color
Yellow/blue ratio

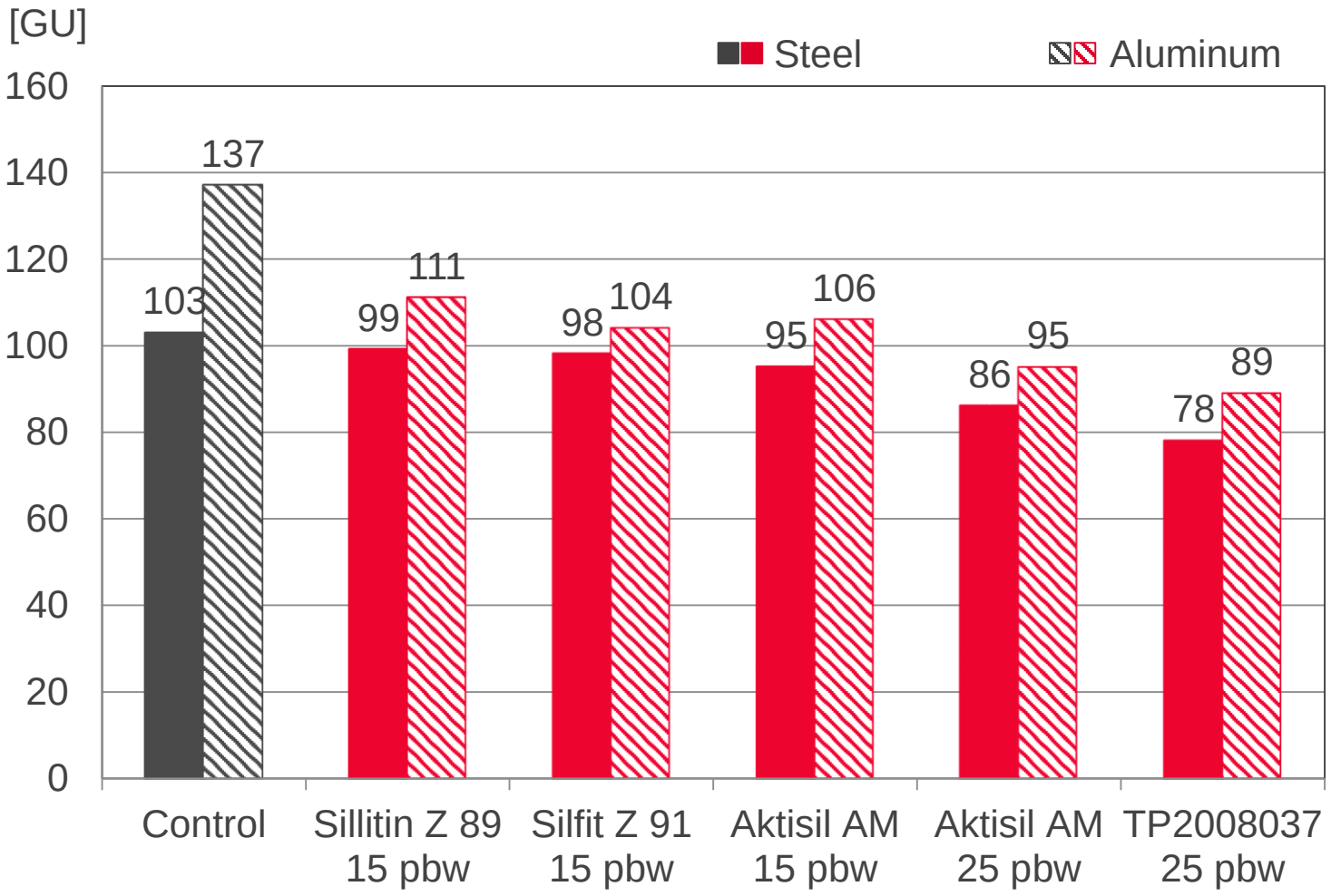
CIE b*, steel



Gloss 20°

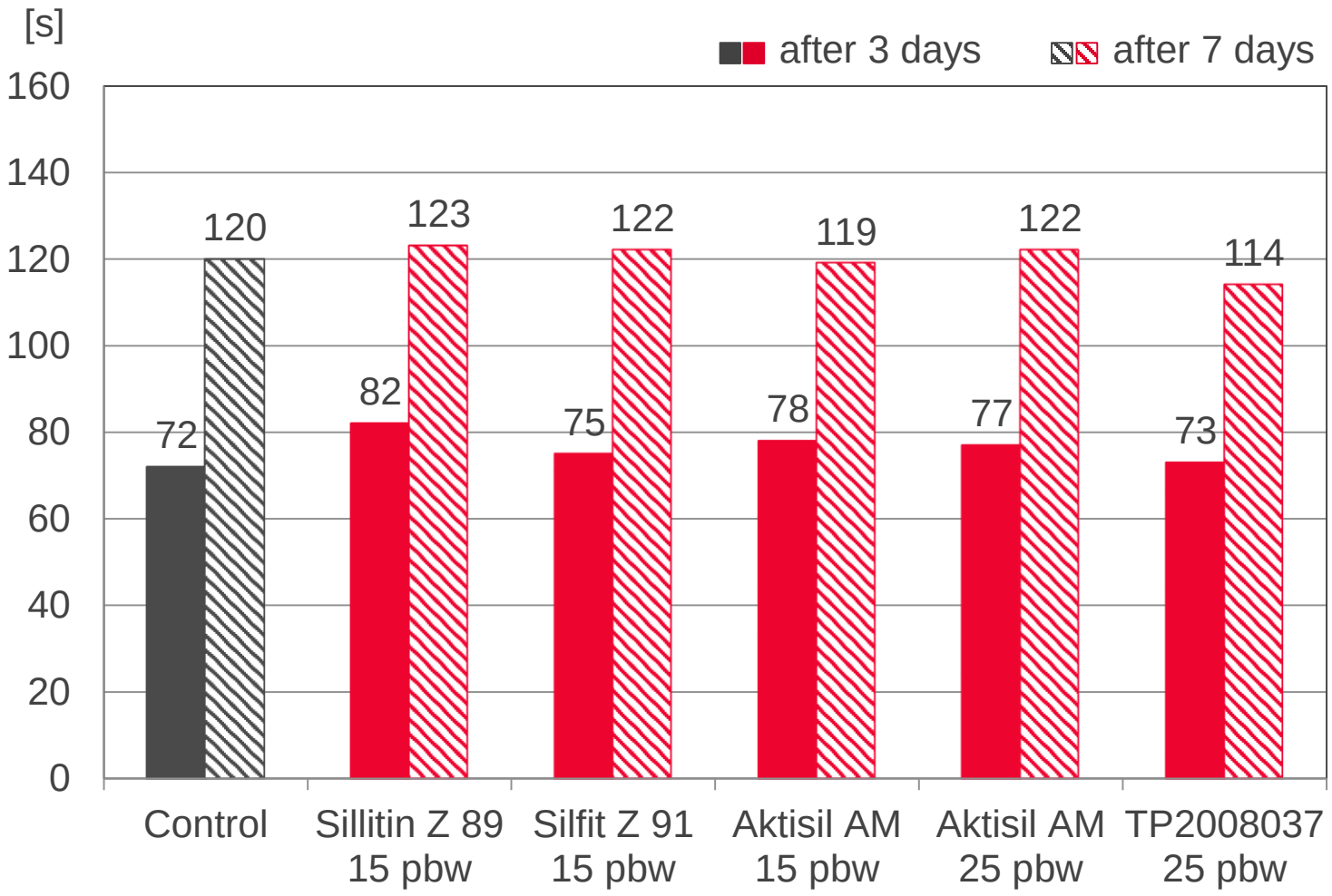


Gloss 60°



Pendulum Hardness

Steel



Humidity Test 240 h
Cross Cut Test (1mm)

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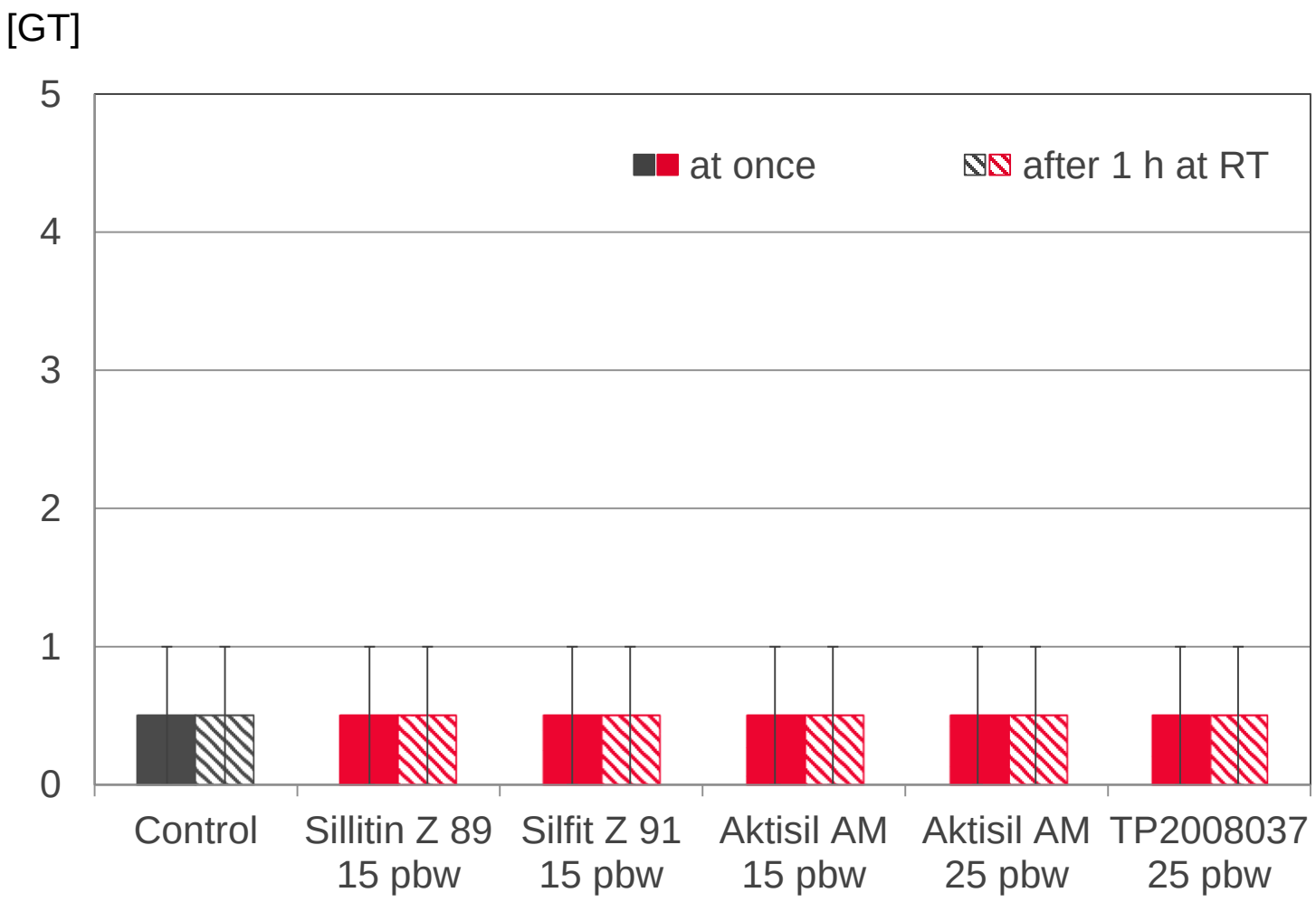
Steel



Humidity Test 240 h
Gloss 60°

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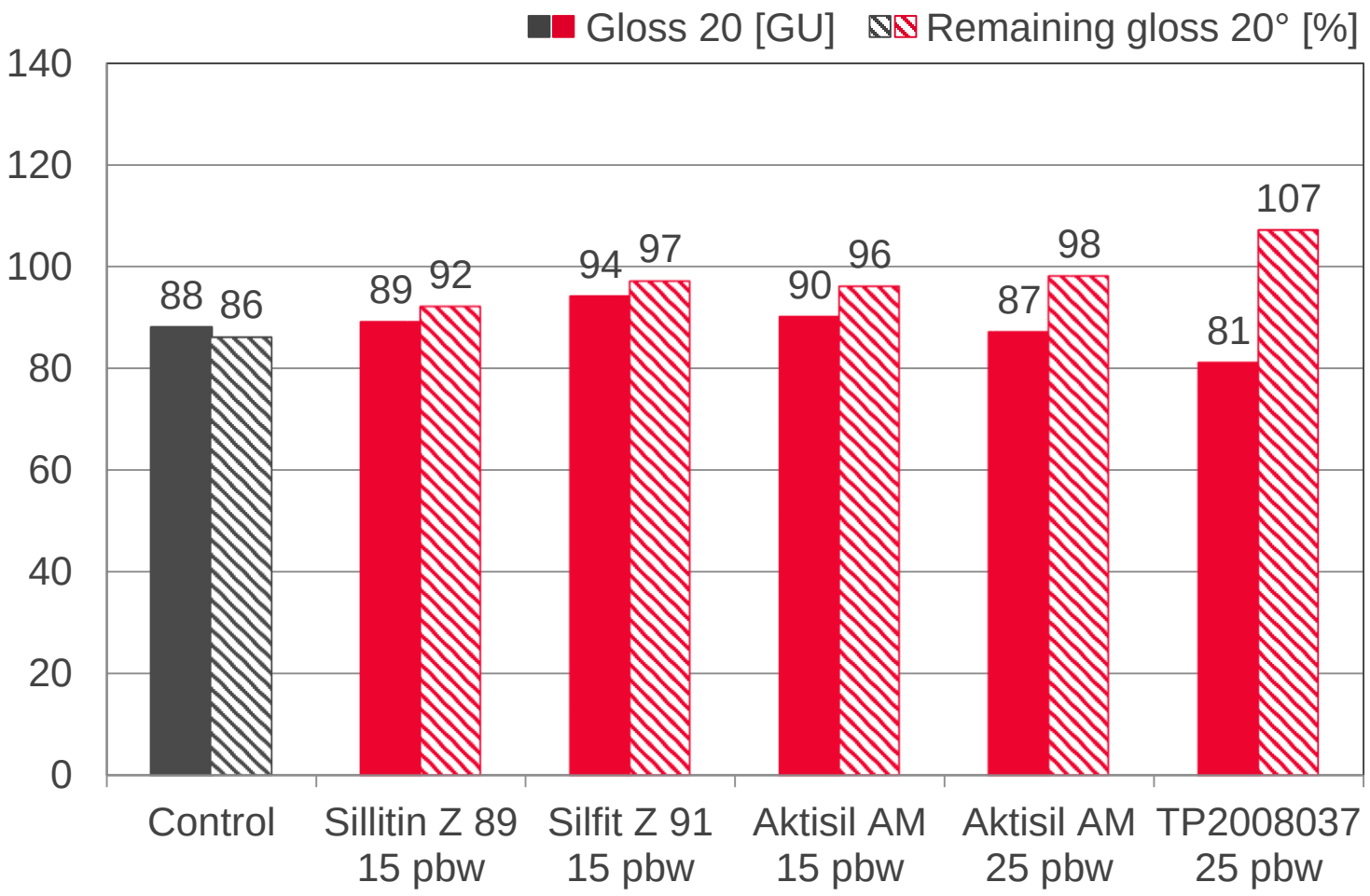
Steel



Humidity Test 240 h
Gloss 20°

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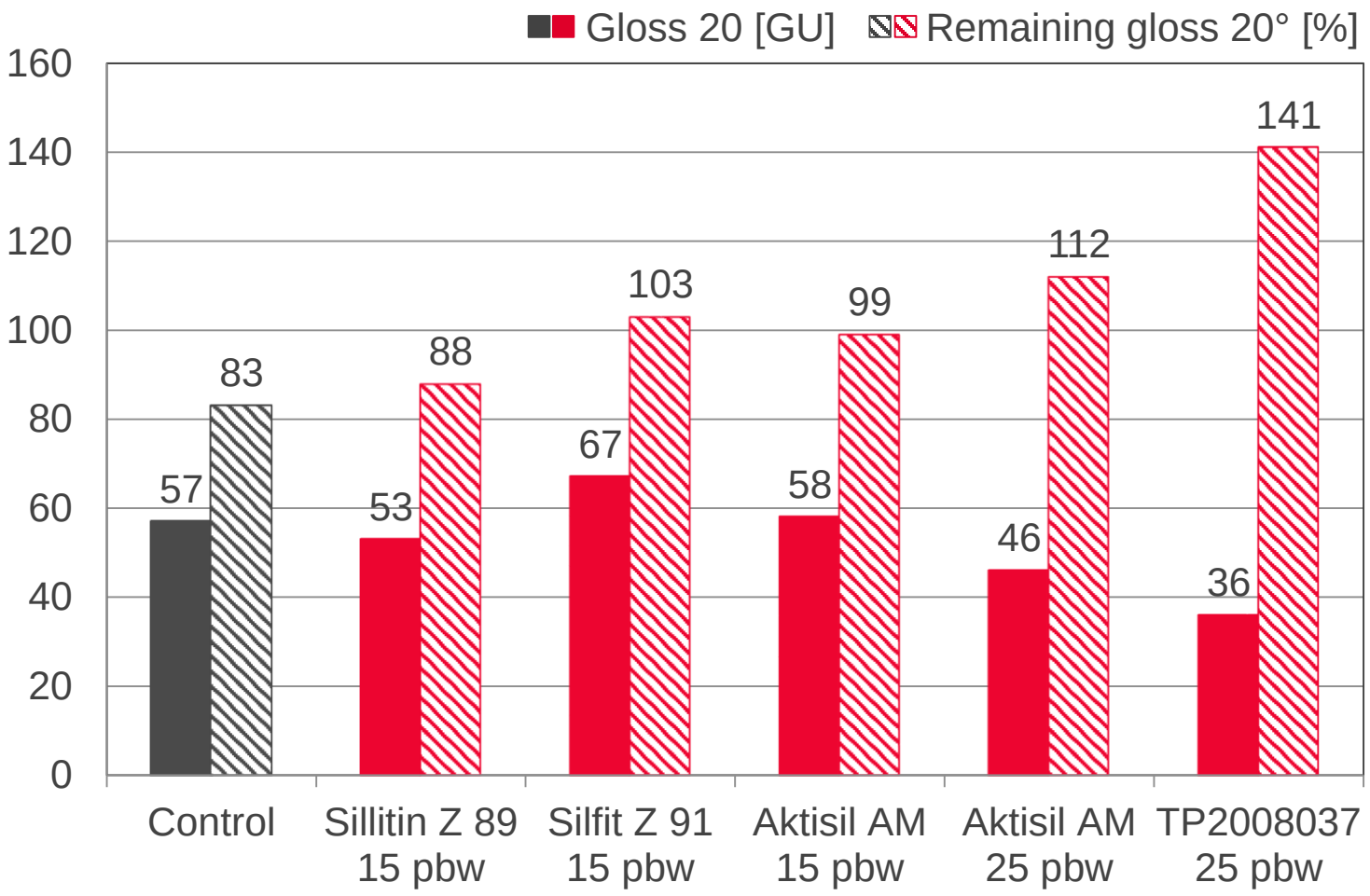
Steel



Salt Spray Test 240 h
Gloss 60°

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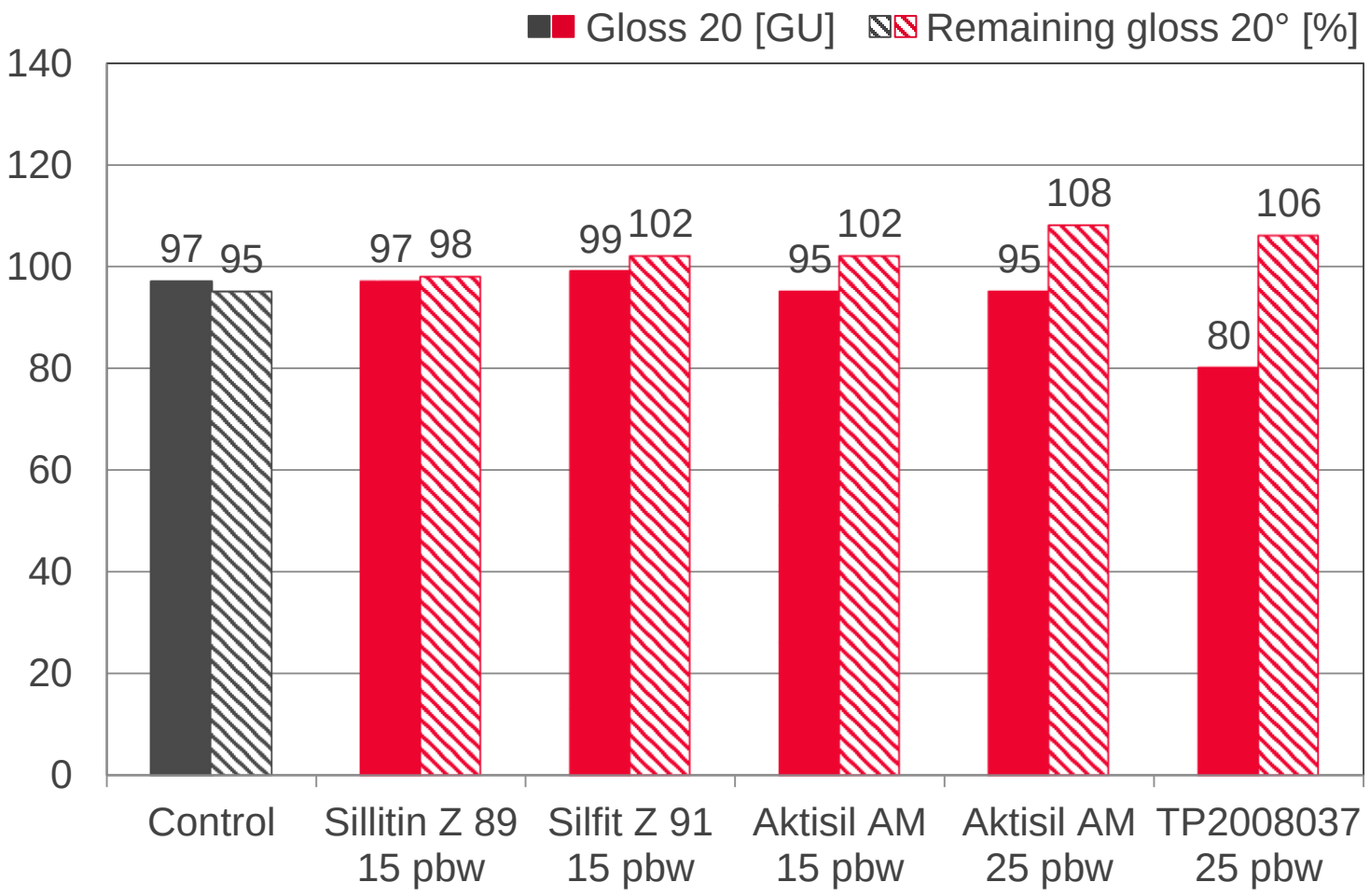
Steel



Salt Spray Test 240 h
Gloss 20°

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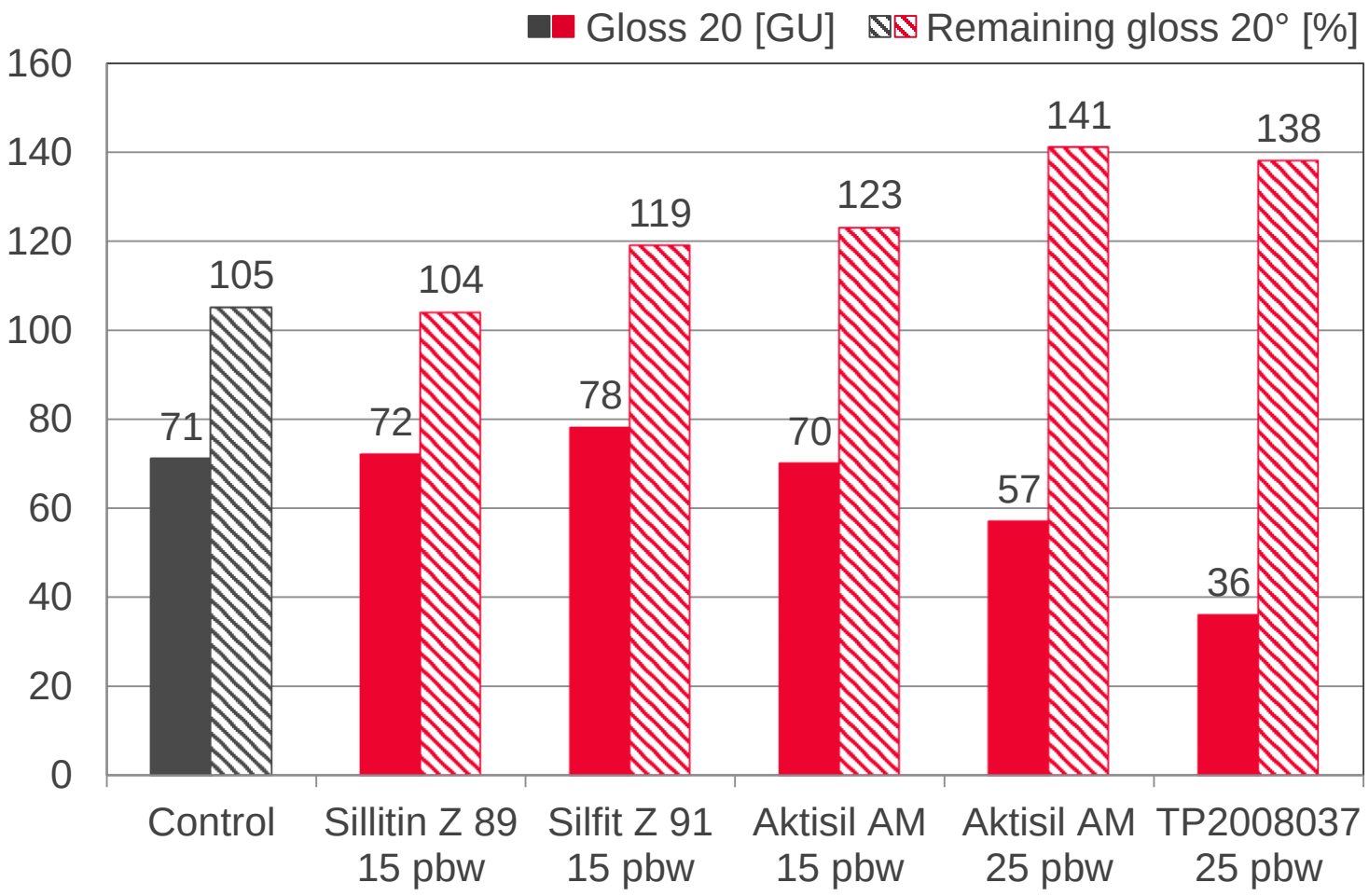
Steel



Salt Spray Test 240 h
Color Change Delta E

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Steel