NADCA - Die Materials Committee Meeting

EVALUATION OF SOLDERING AND WASHOUT IN HEAT RESISTANT MATERIALS FOR DIE INSERTS

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OBJECTIVES

- Evaluate thermal fatigue resistance of nickel and cobalt based superalloys (718, 706, 625 and Incoloy 909).

- Develop a soldering/washout test with a molten aluminum jet impinging on the test piece. H13 serves as control/baseline material.

- Evaluate soldering/washout of copper, nickel base and refractory alloys with good thermal fatigue properties.

- Evaluate soldering/washout of nitro-carburized H13 pins.

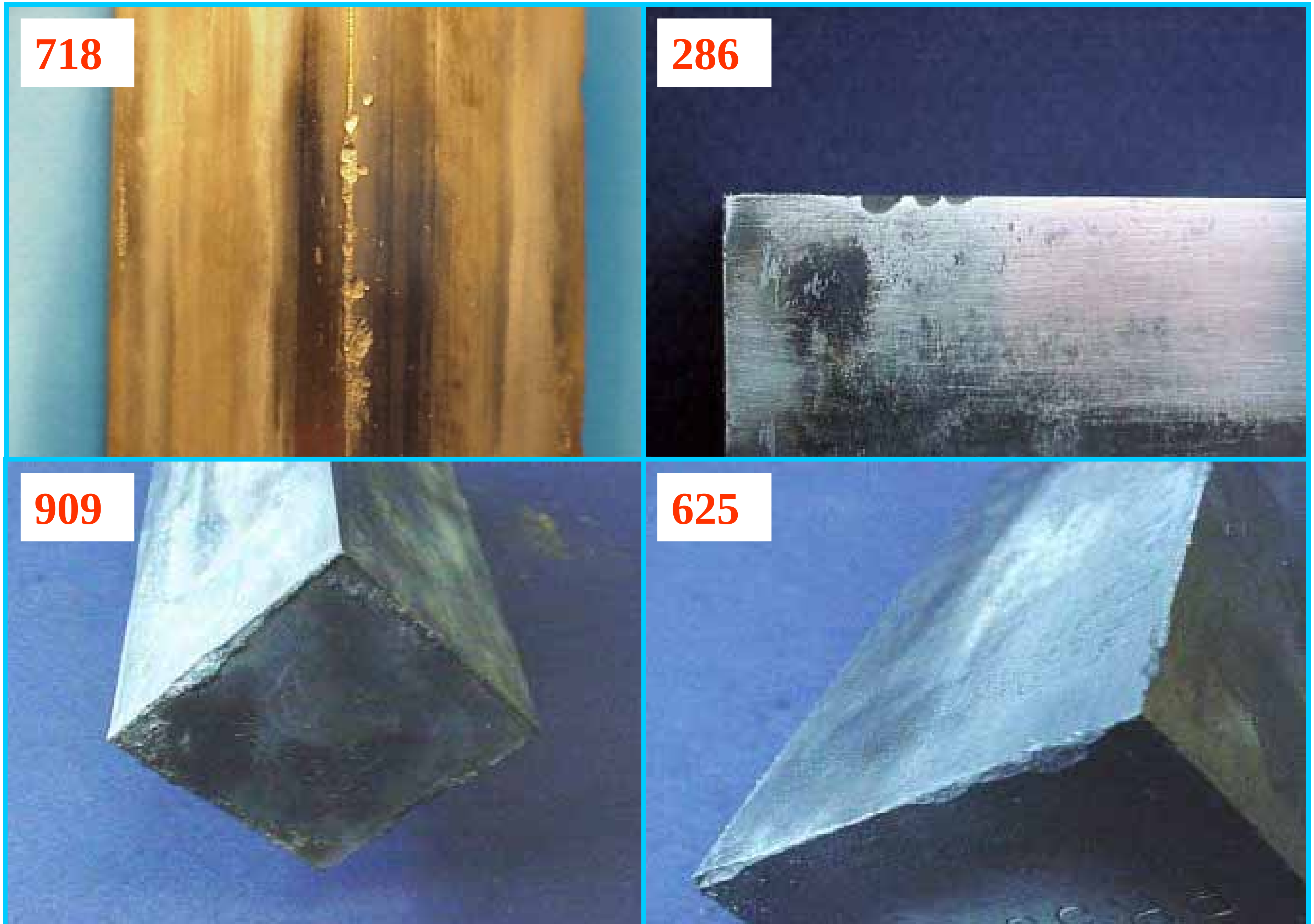
- Evaluate soldering/washout of PVD coated H13 pins.

Evaluation of nickel-based alloys for use as inserts in die casting applications.

JUSTIFICATION

Nickel-based superalloys have high strength at high temperatures and superior thermal fatigue resistance. They are widely used in turbine blade applications and commercially available.

Dissolution at the Corners of Nickel Alloy Specimens

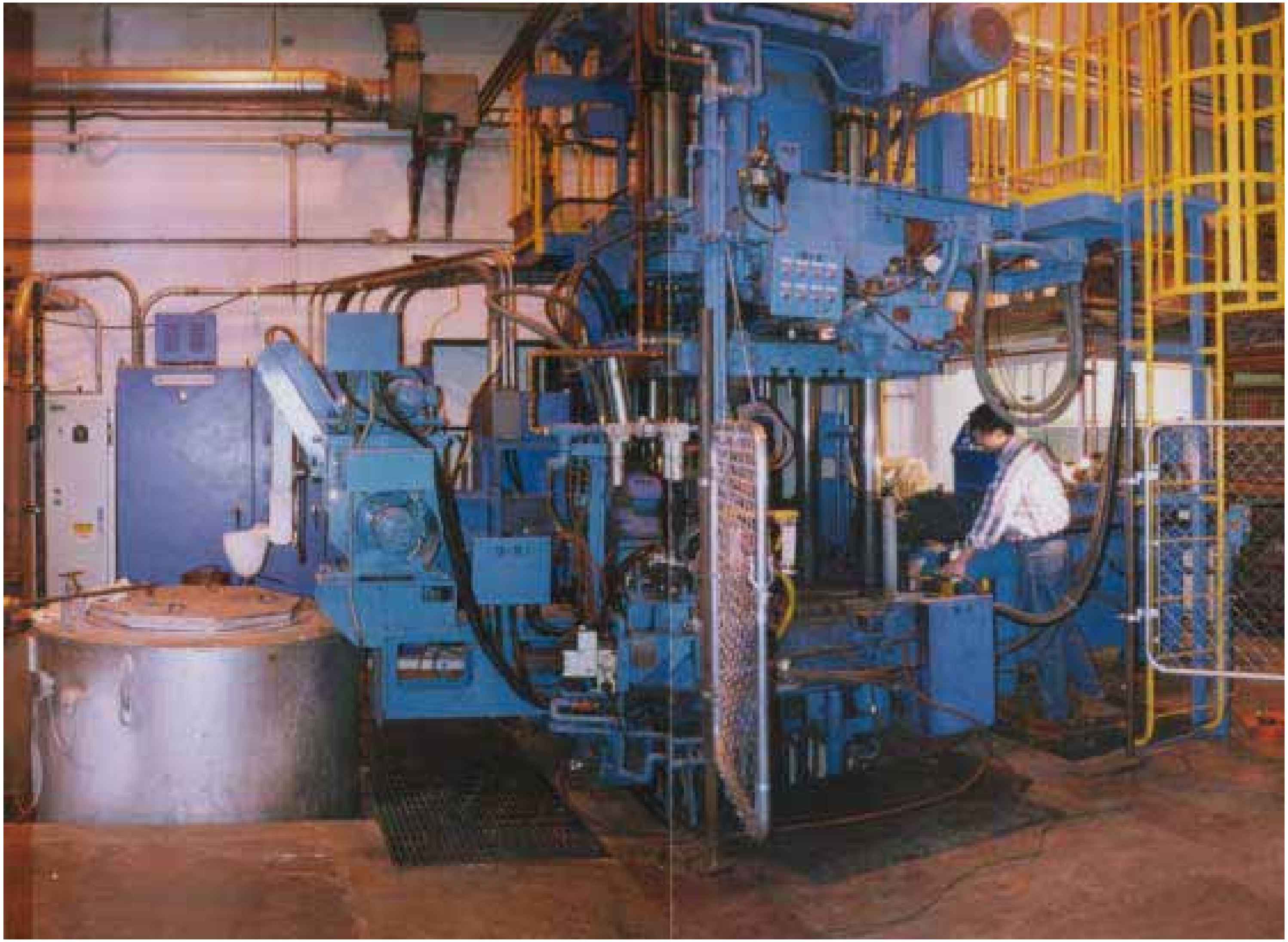


CONCLUSIONS

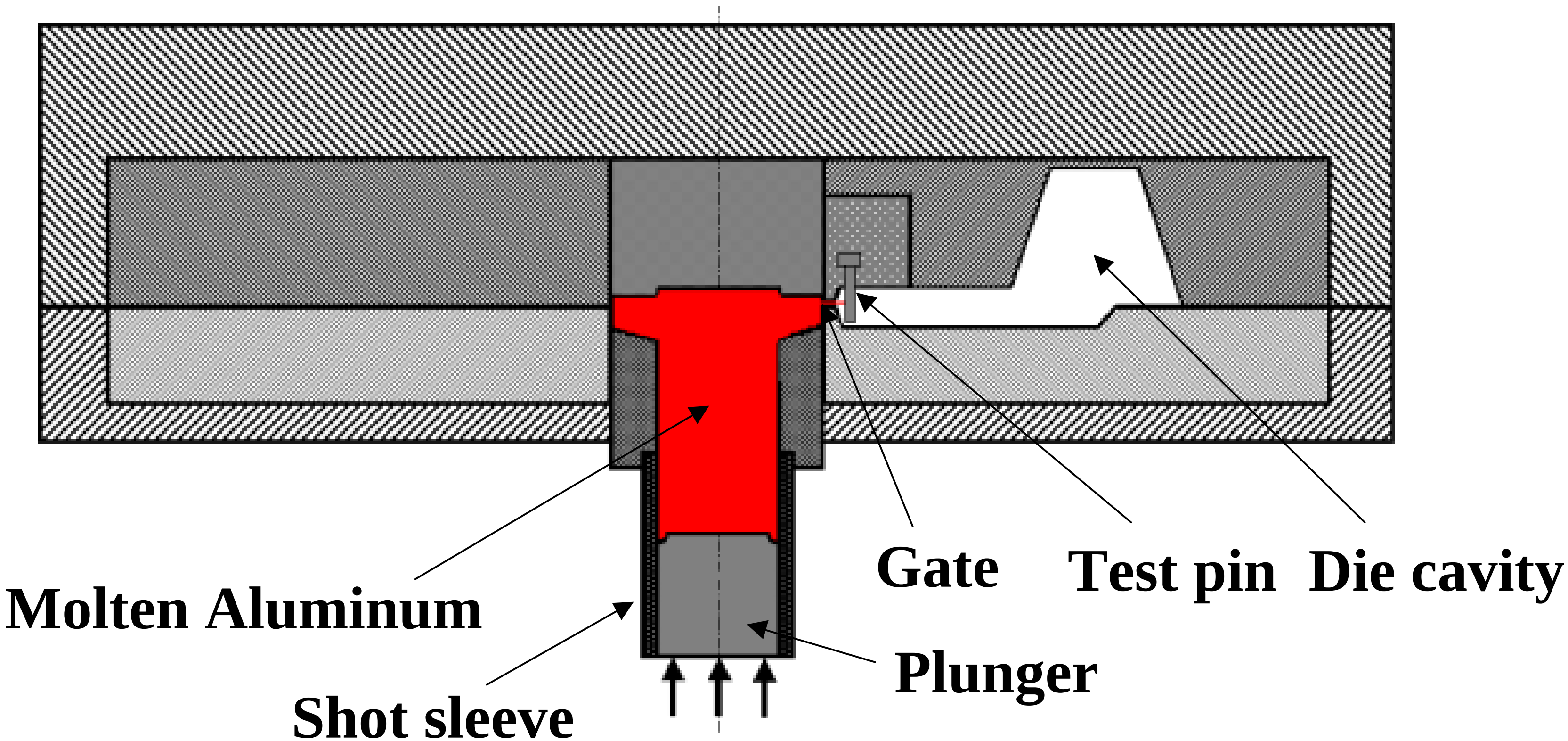
- **While they posses good thermal fatigue resistance, nickel-containing superalloys are prone to dissolve in molten aluminum when overheated.**

- **In order to be utilized in die casting, these alloys require a protective coating yet to be identified or developed. Conversely, better cooling with internal cooling lines closer to the surface would be required.**

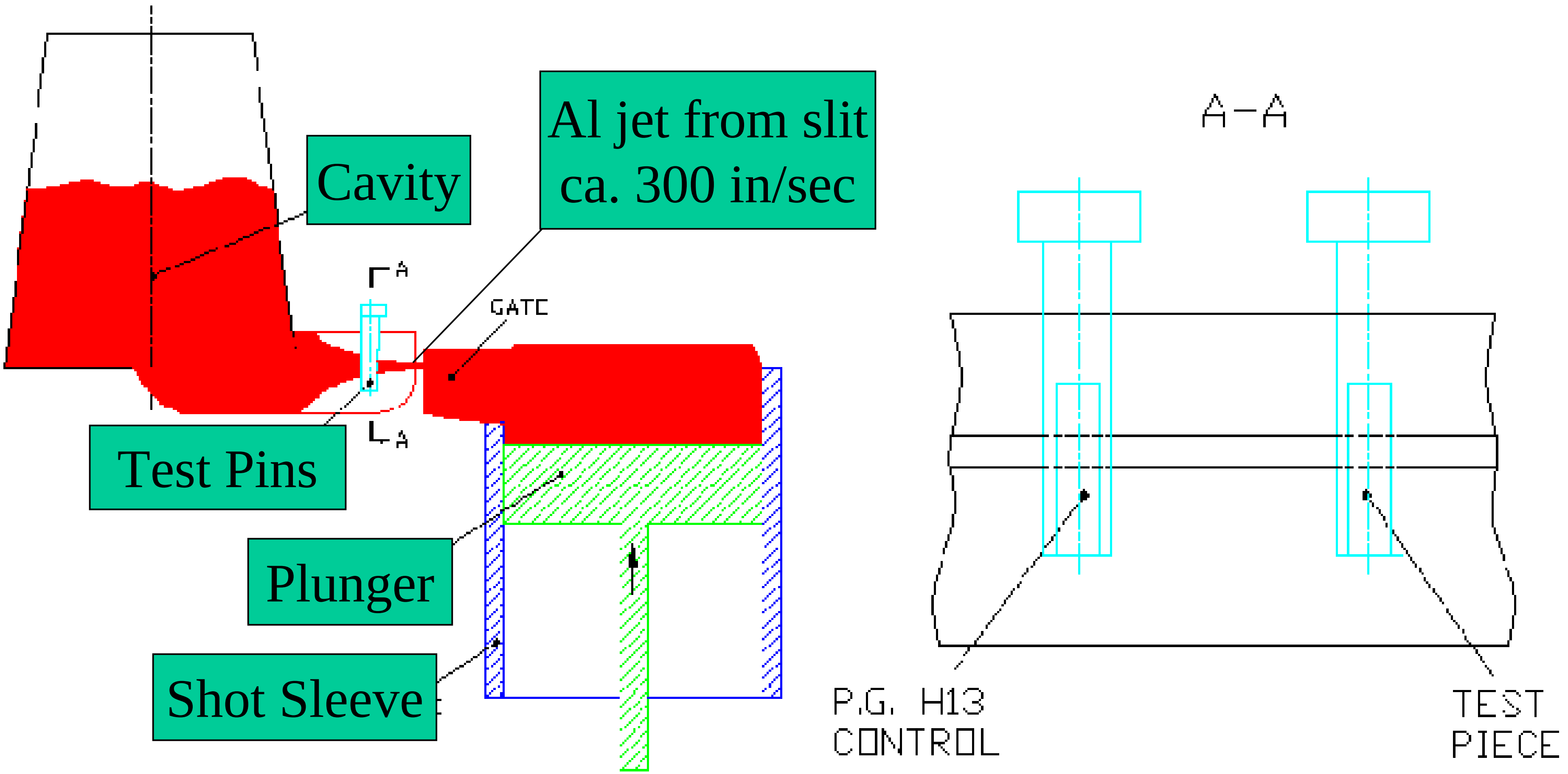
UBE VSC 315 Ton Squeeze Cast Machine at CWRU



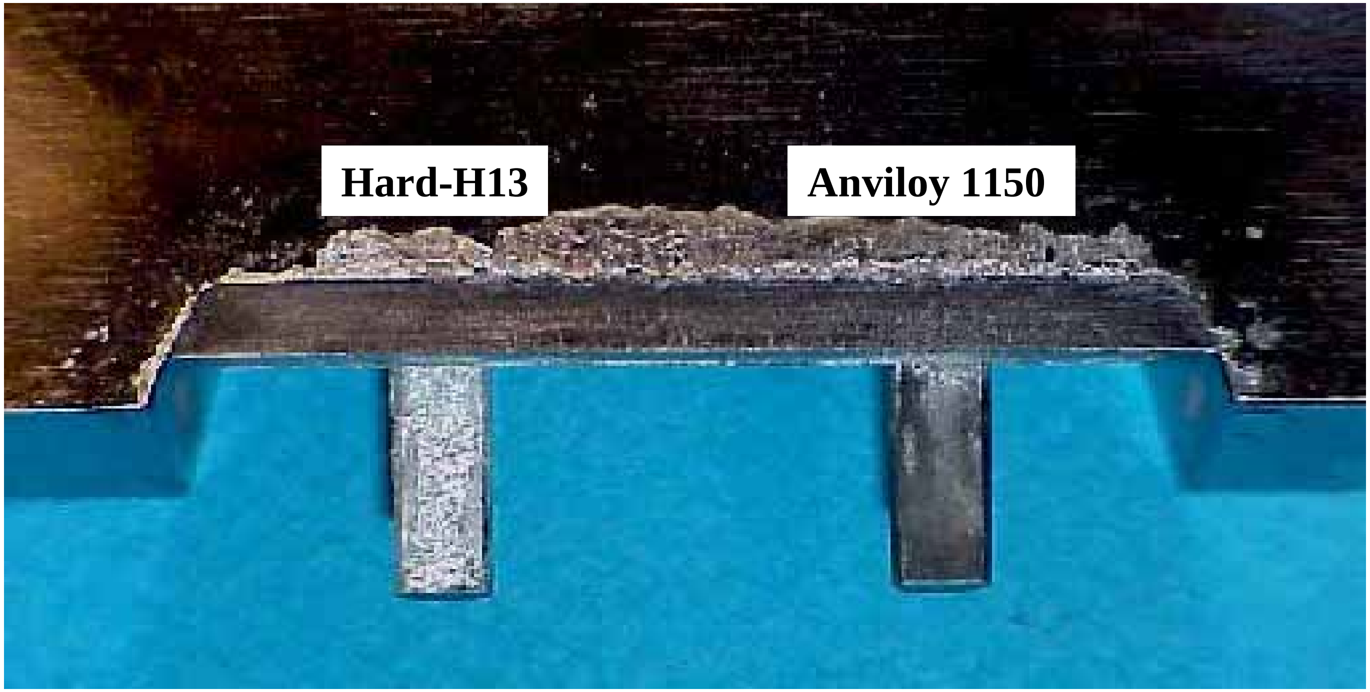
Schematic Diagram of the Accelerated Soldering Test



Schematic of the Washout/Soldering Testing Set-up

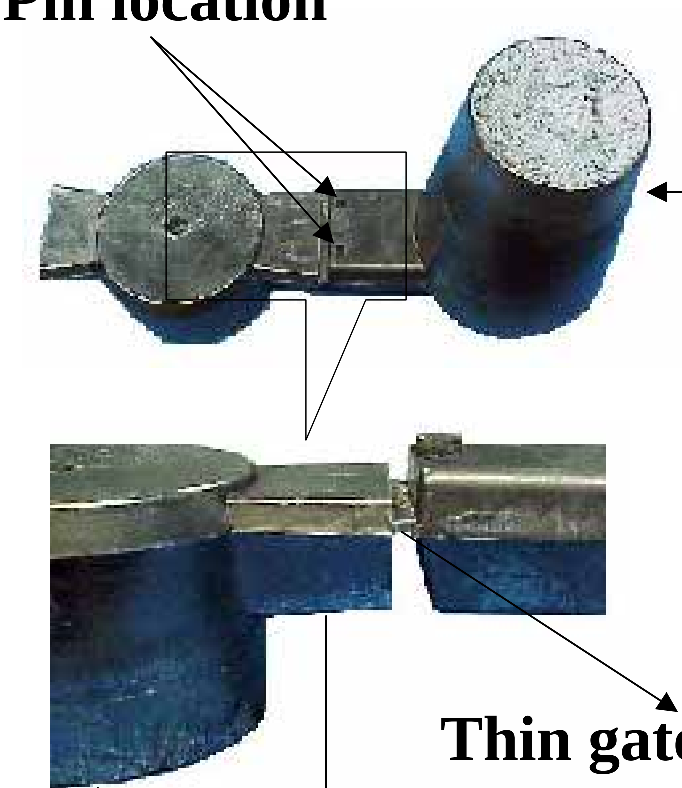


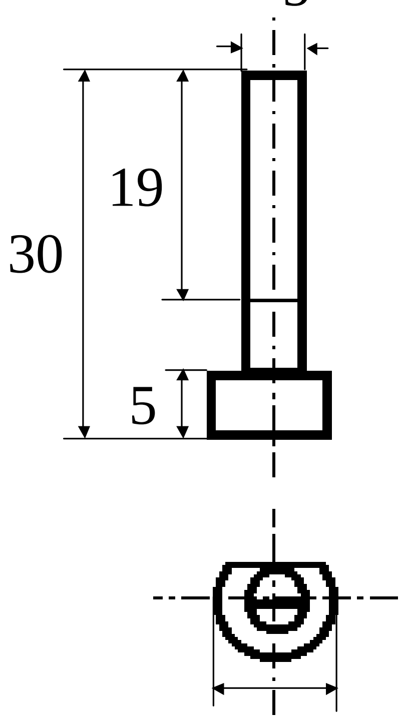
Sub-Insert for Soldering&Washout Experiments



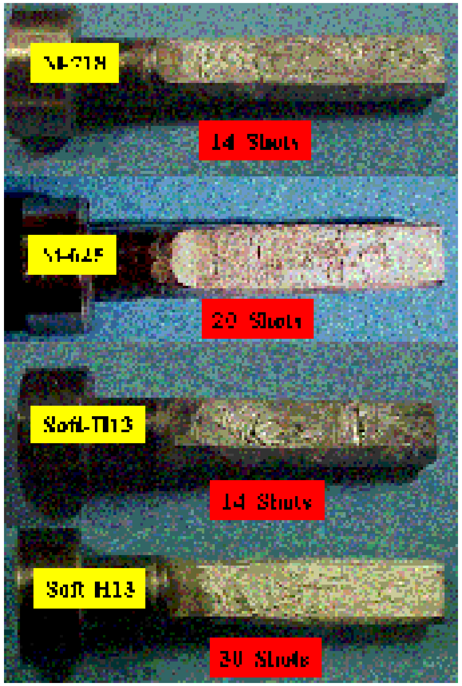
The hard H-13 pin is coated with a silvery soldered layer of Al

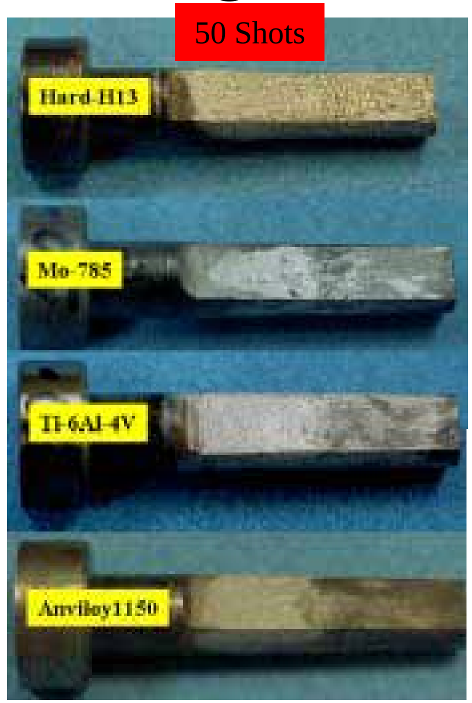
Test Pin Design and Position

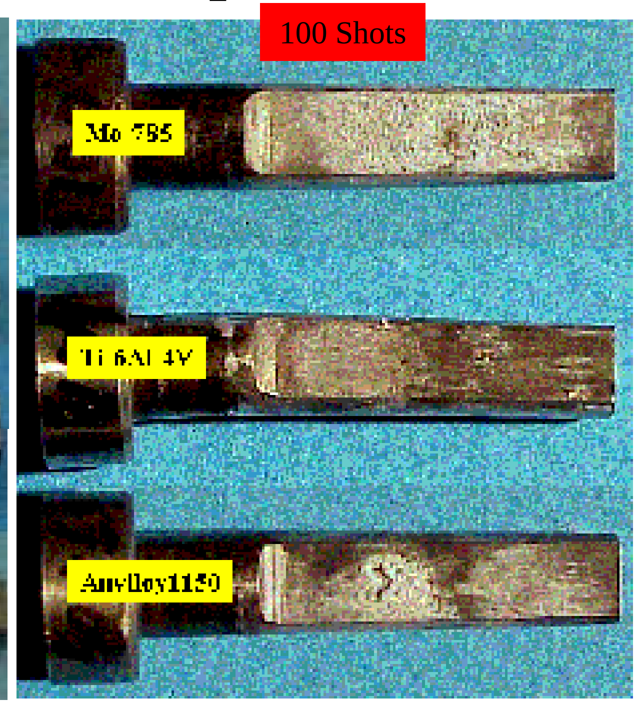


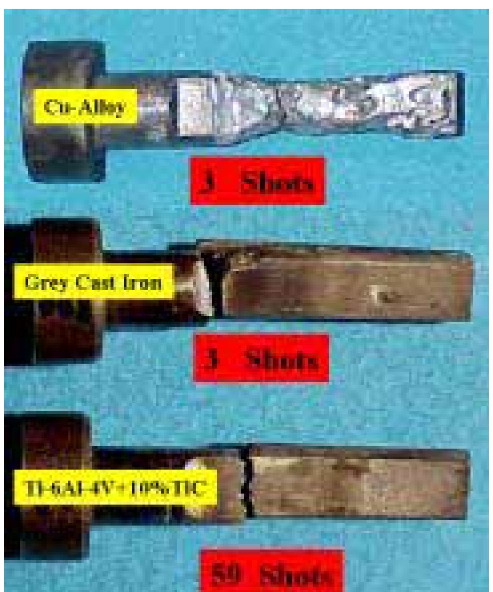


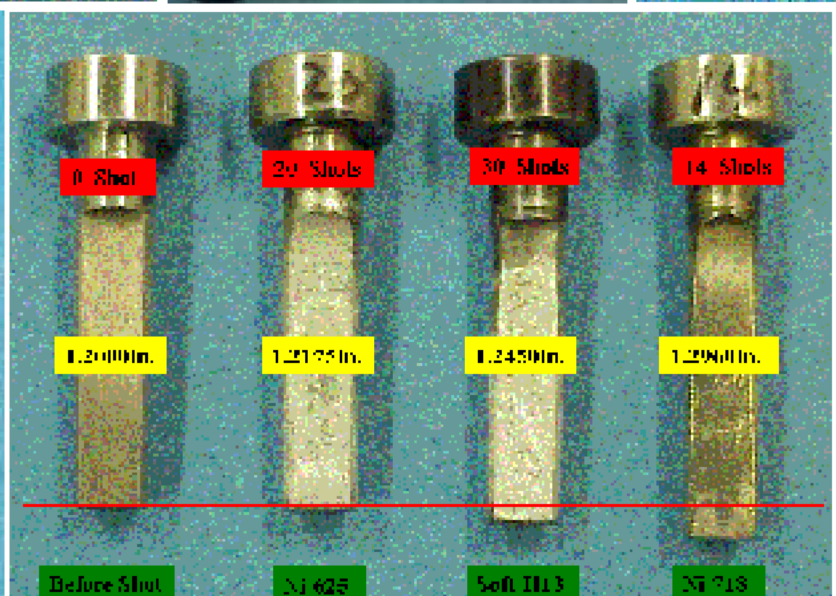
Results of Soldering & Washout Experiment

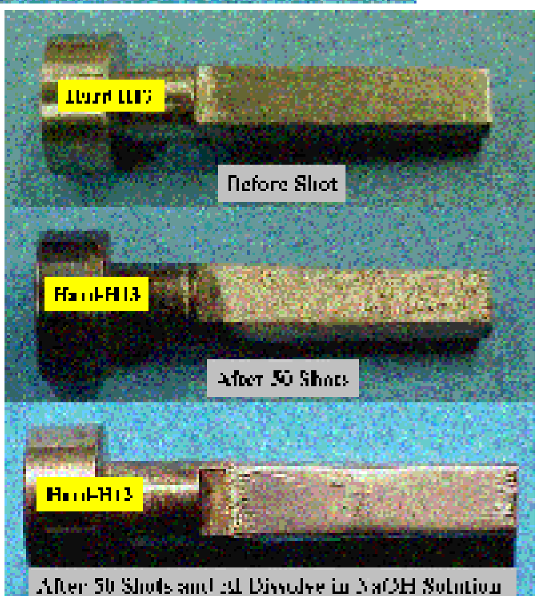












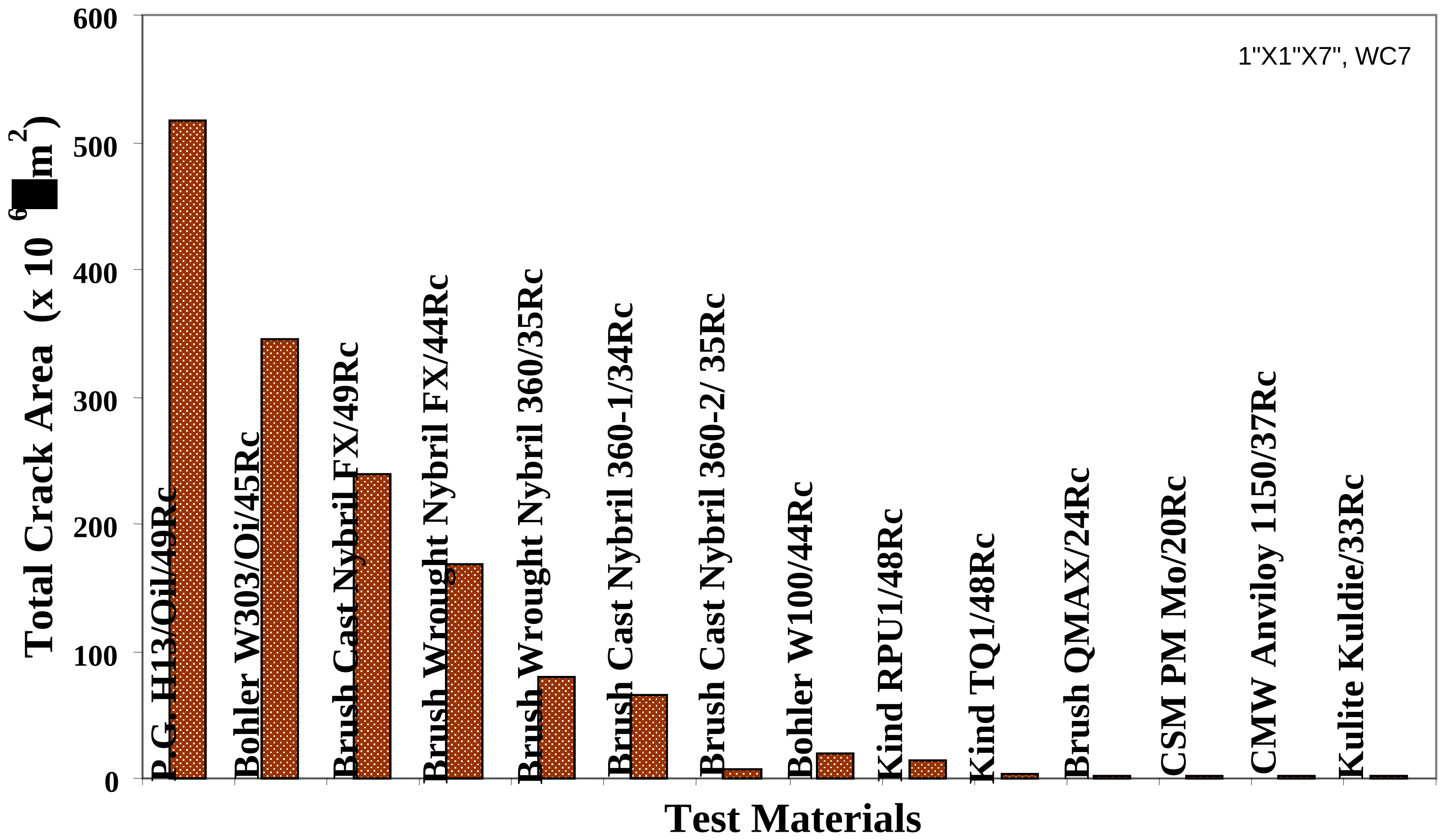
Chemical Composition of Copper Base Pin (wt%)

Alloy Type	Be	Co	Cu
Cu Base	1.7	0.2	98.1

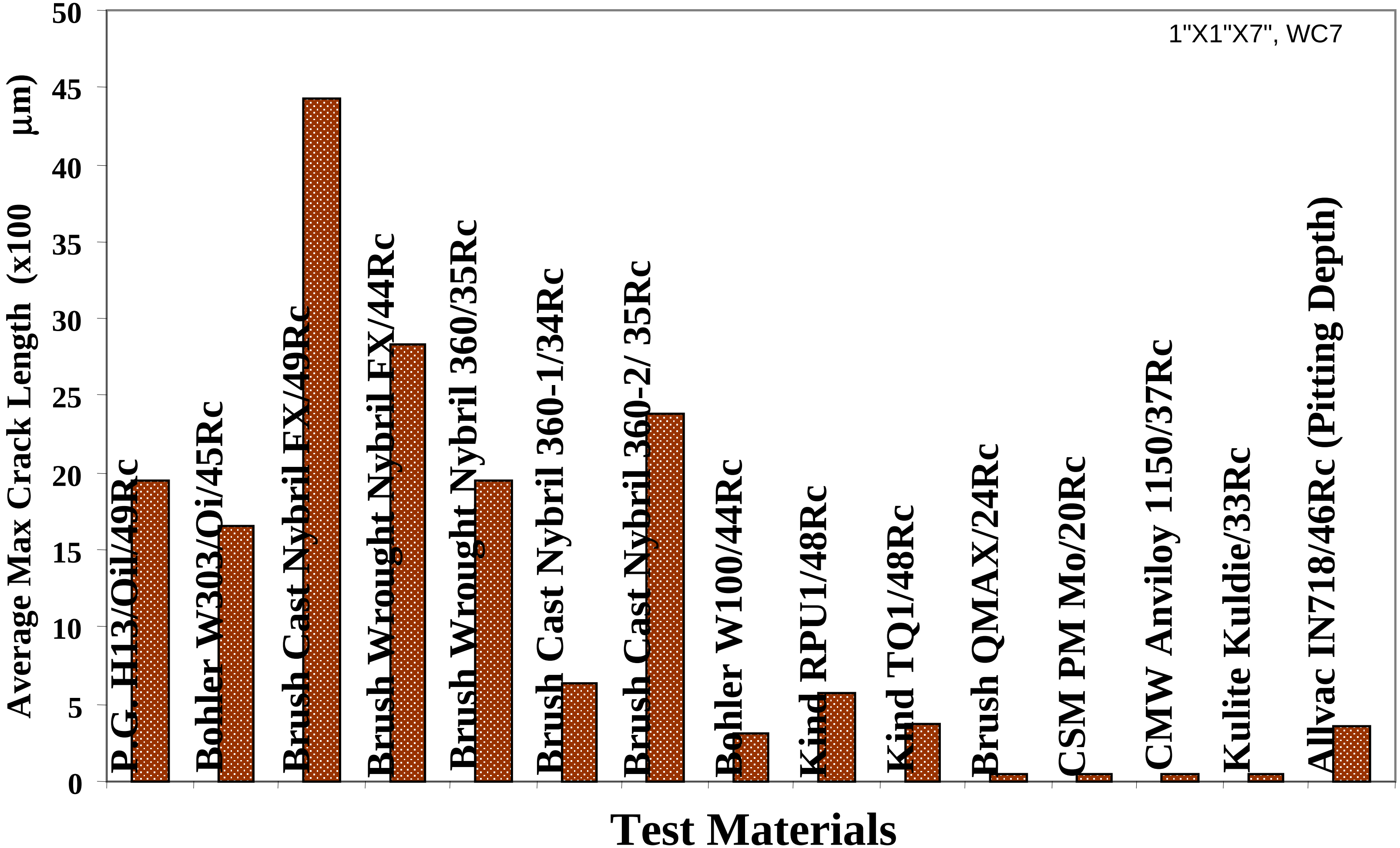
Chemical Composition of Pins (wt%)

Alloy Type	C	Mn	Si	Cr	Mo	Ti	Al	Fe	Ni	Nb	V	W	Hf	Zr
Ni-718	0.02	0.05	0.05	17.6	2.86	1.01	0.48	18.7	53.6	5.09				
Ni-625	0.052	0.06	0.2	20.9	8.45	0.32	0.23	4.38	61.1	3.36				
H13	0.4	0.35	1	5.25	1.5						1			
Mo-785					97.5	0.65							1.18	0.13
Ti-6Al-4V						90	6				4			
Anv. 1150					4			2	4			90		

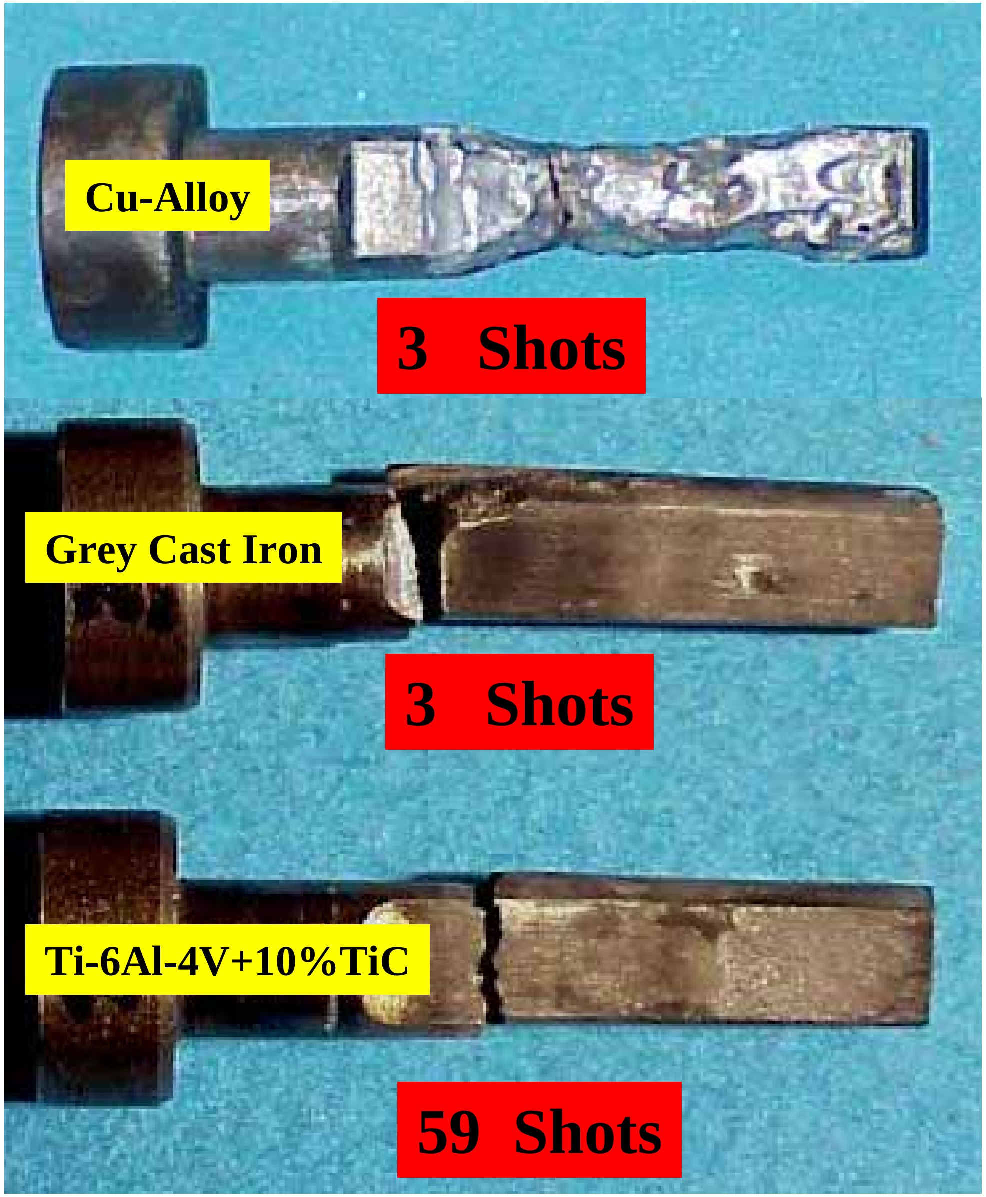
TOTAL CRACK AREA AFTER 15,000 THERMAL FATIGUE CYCLES (1"x1"x7")



AVERAGE MAXIMUM CRACK LENGTH AFTER 15,000 THERMAL FATIGUE CYCLES (1"x1"x7")

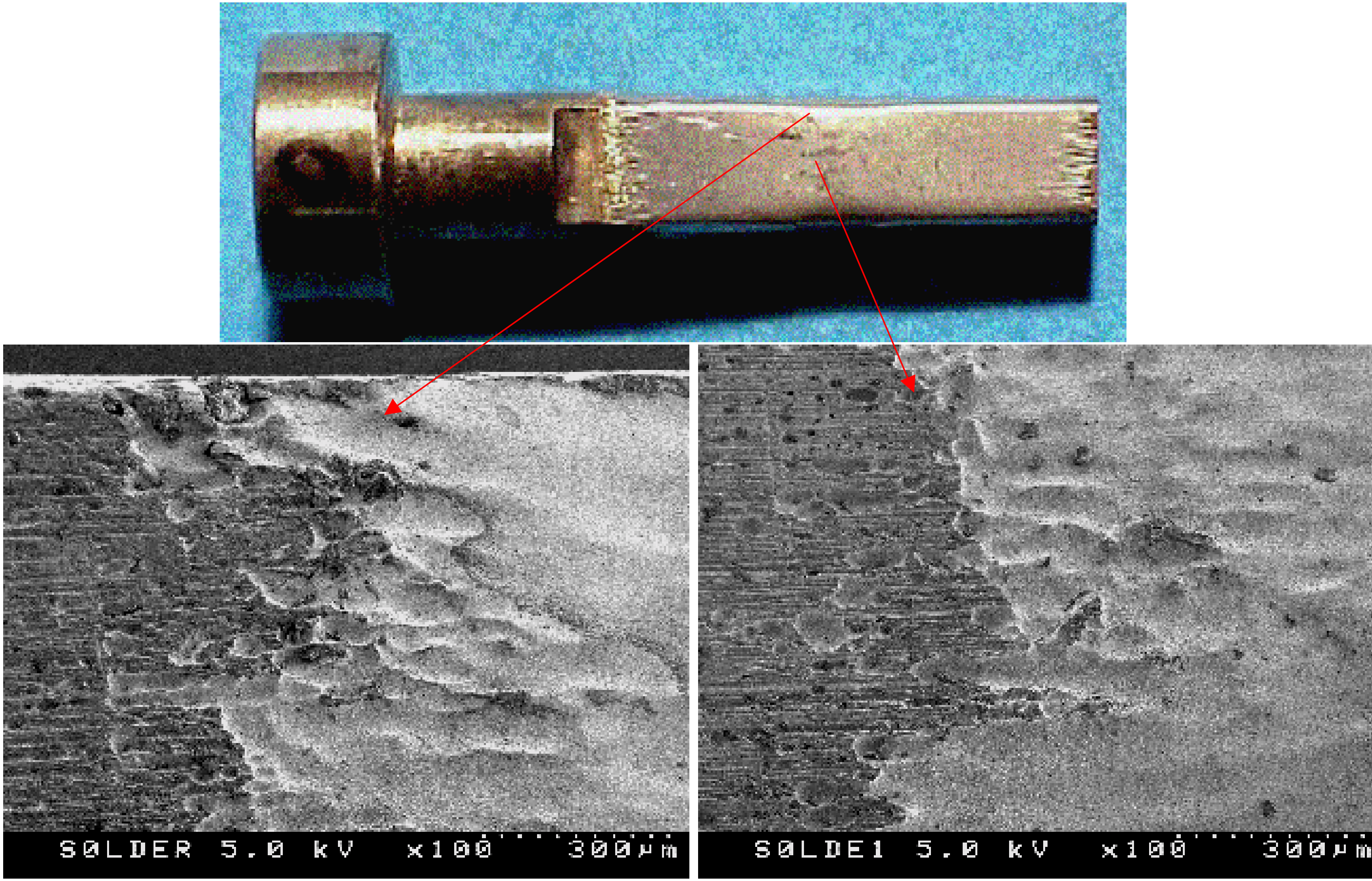


Broken Pins

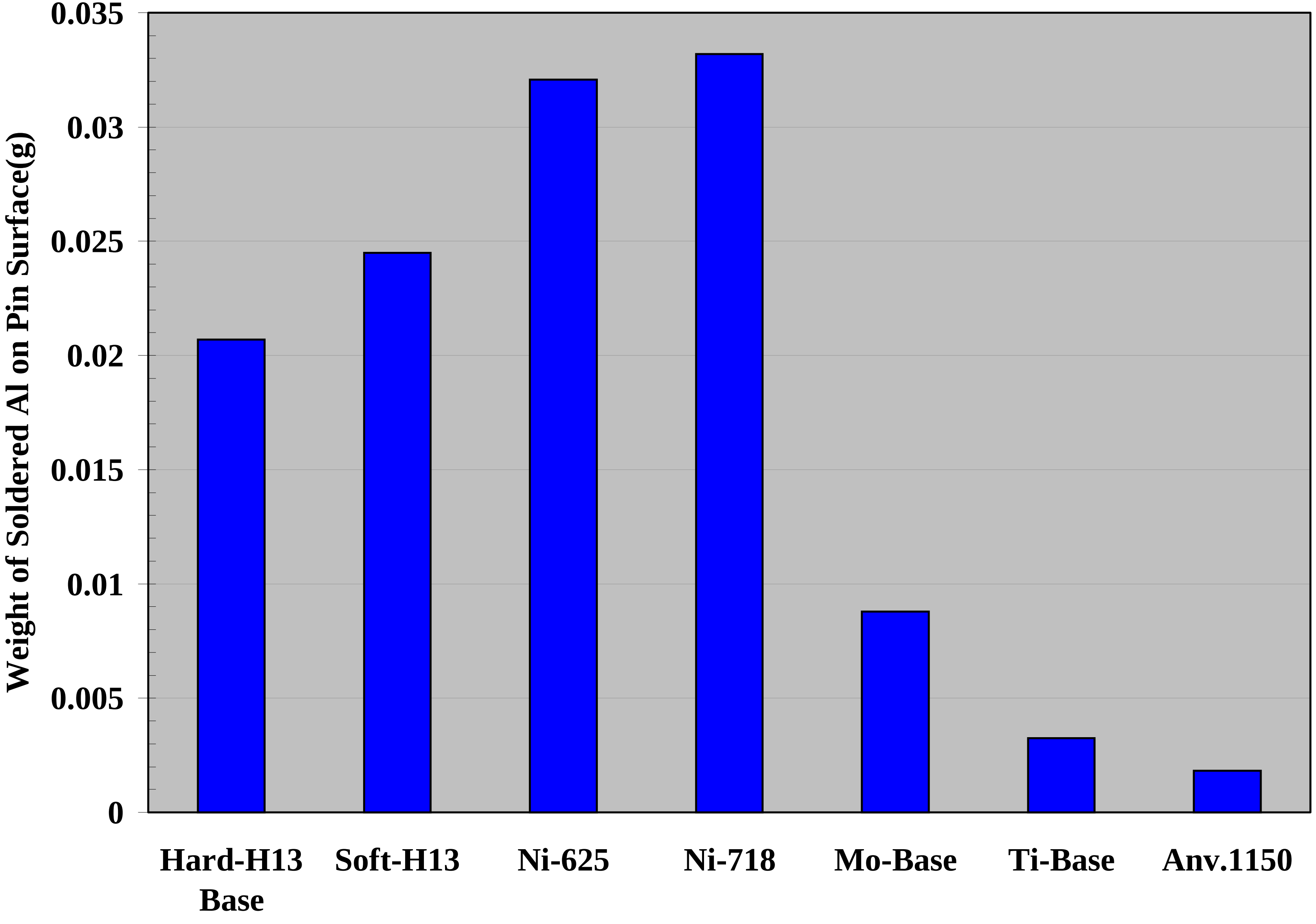

Cu-Alloy
3 Shots
Grey Cast Iron
3 Shots
Ti-6Al-4V+10%TiC
59 Shots

Appearance of Pins after 50 Shots (with Soldered Al Dissolved in NaOH)

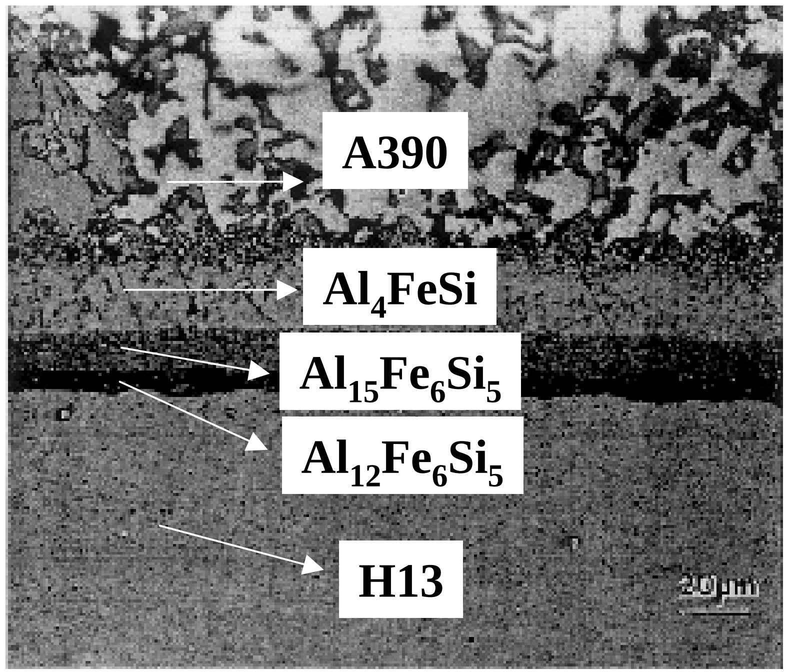
Washout in the Hard H13 Pin Impinged Directly by the Al (50 shots)



Effects of Pin Material on Soldering



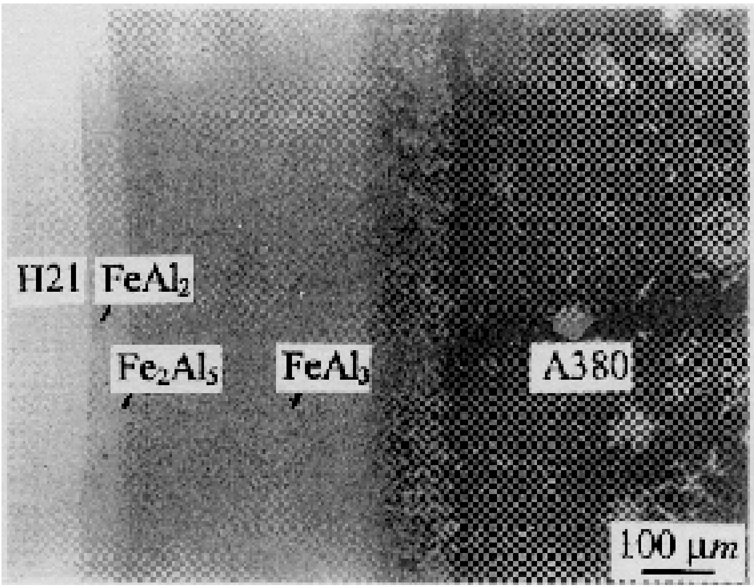
Intermetallic and Soldered Layers on a H13 Pin[1]



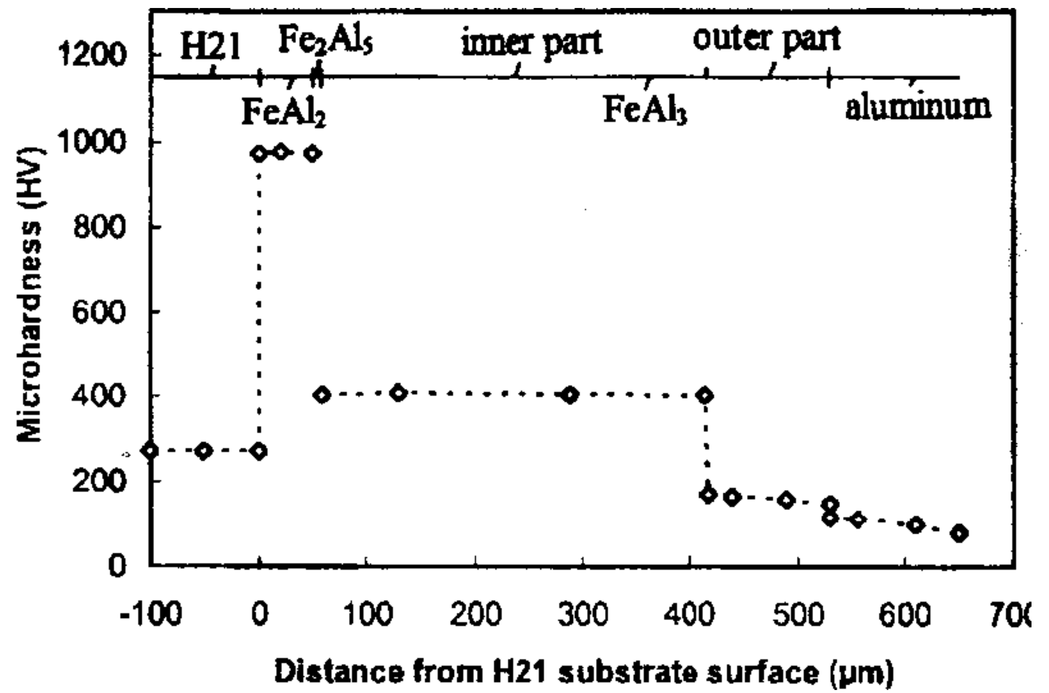
[1] Sandhya Gopal, Anup Lakare and Rajiv Shivpuri, Soldering in Die Casting: Aluminum Alloy and Die Steel Interactions, Die Casting Engineering, May/June 2000, pp70-81

Interfacial Morphology and Microhardness Profile of the Reaction Zone between H21 Steel and Molten A380 Alloy[2]

(Rotating at 1292°F with a Speed of 300 rpm for 9 Hours)



(Rotating at 1292°F with a Speed of 300 rpm)



[2] M. Yan and Z. Fan, The Erosion of H21 Tool Steel in Molten A380 Alloy, Journal of Materials Science, Vol. 35(2000), p1661-1667

Comparison of Microhardness of Intermetallic compounds[3]

Layer	Microhardness (HV, GPa) [30]	Layer	Microhardness (HV, kg/mm^2) [5]	Layer	Microhardness (HV, kg/mm^2) [27]
18Cr-10Ni stainless steel (in pure Al)	1.8 ± 0.2	H13 tool steel (in A380)	239	H21 tool steel (in A380)	271
		$FeAl_2$-type	985	$FeAl_2$-type	974
Fe_2Al_5-type	8.9 ± 0.9	Fe_2Al_5-type		Fe_2Al_5-type	
$FeAl_3$-type	4.5 to 3.06	$FeAl_3$-type	1096	$FeAl_3$-type	403
pure Al	0.6 ± 0.1	A380 alloy	100	A380 alloy	94

*: 1 GPa ≈ 100 kg/mm^2.

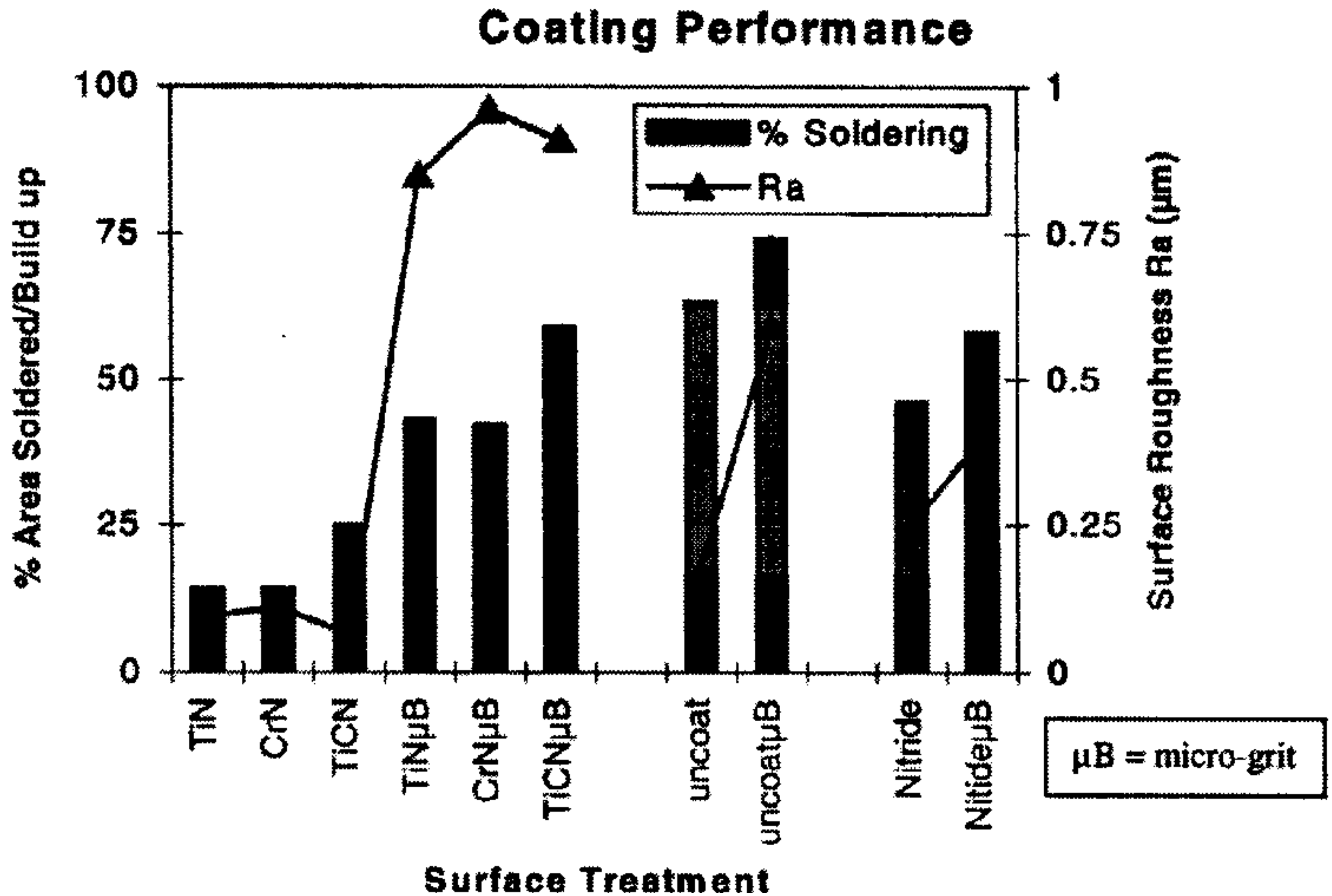
[3] M. Yan and Z. Fan, Review: Durability of Materials in Molten Aluminum Alloys, Journal of Materials Science,Vol.36(2001), pp285-295

[5] M. Sundqvist and S. Hogmark, Tribology Inter. 26(1993), pp129.

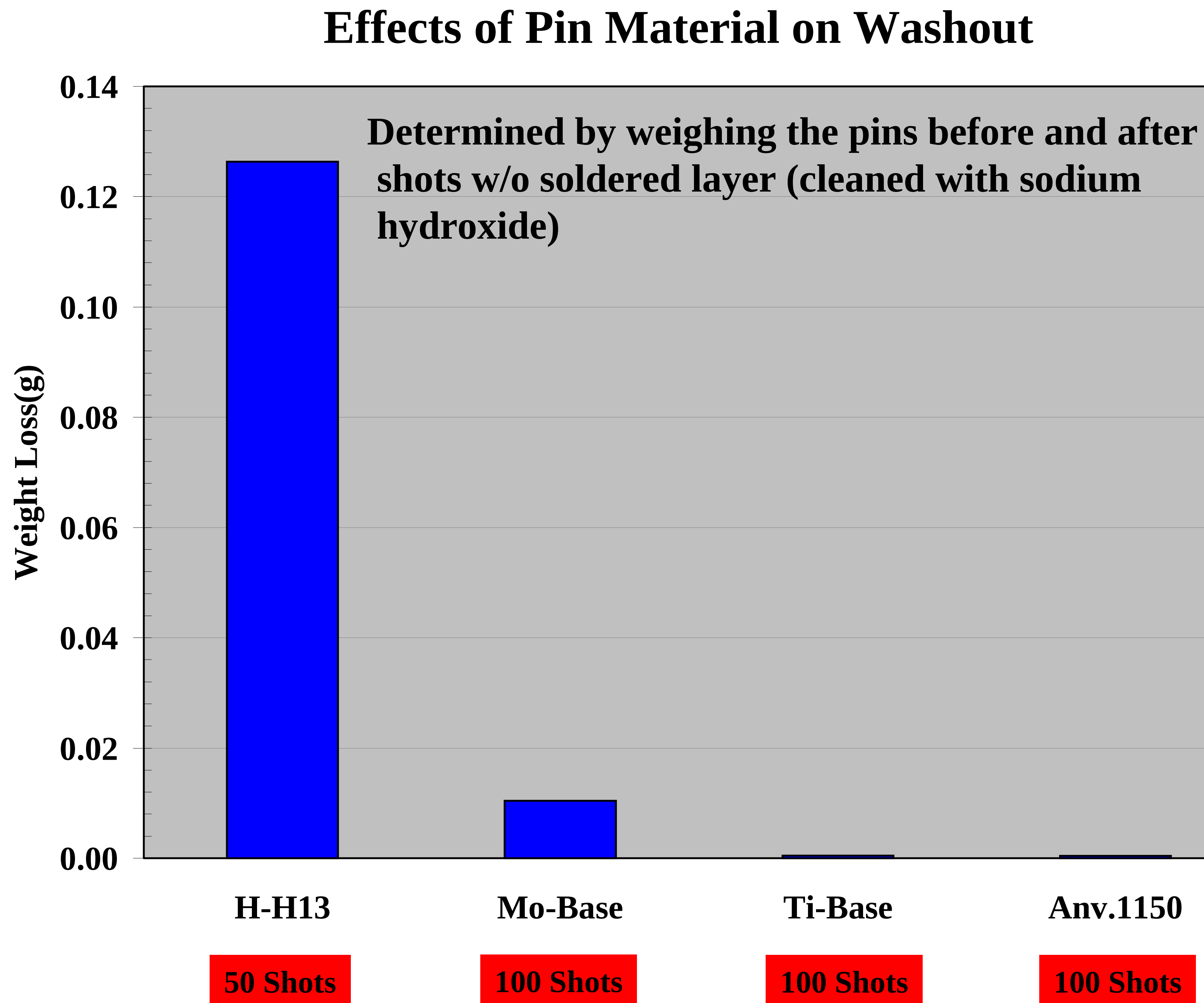
[27] M. Yan and Z. Fan, The Erosion of H21 Tool Steel in Molten A380 Alloy, Journal of Materials Science, Vol. 35(2000), pp1661-1667.

[30] V. I. Dybkov, J. Mater. Sci. 25(1990), pp3615.

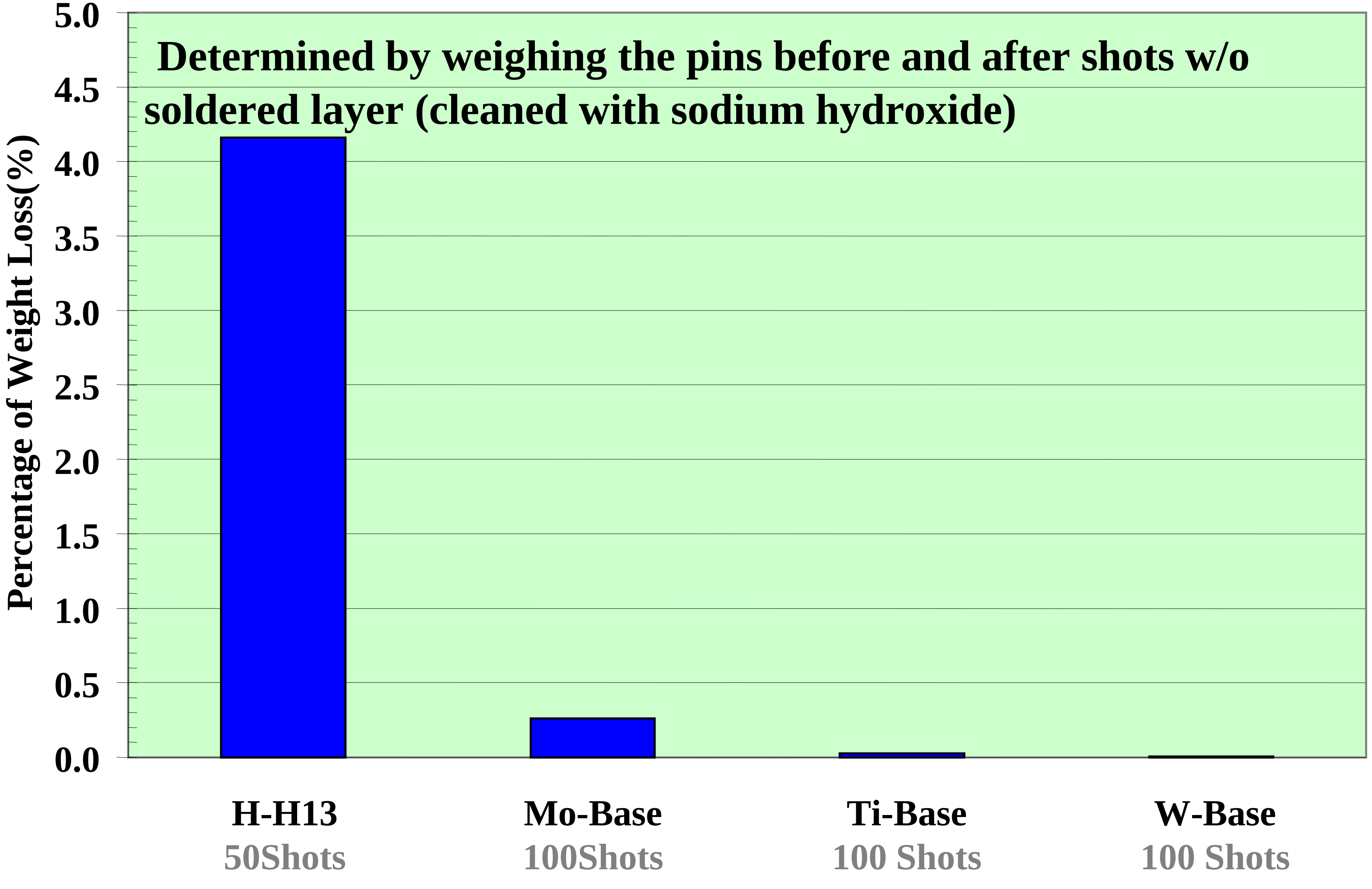
Relationship between Surface Roughness and Percentage of Area Solder/Build up after 50 Shots for PVD TiN, CrN, TiCN, Nitrided and Uuncoated core Pins [4]



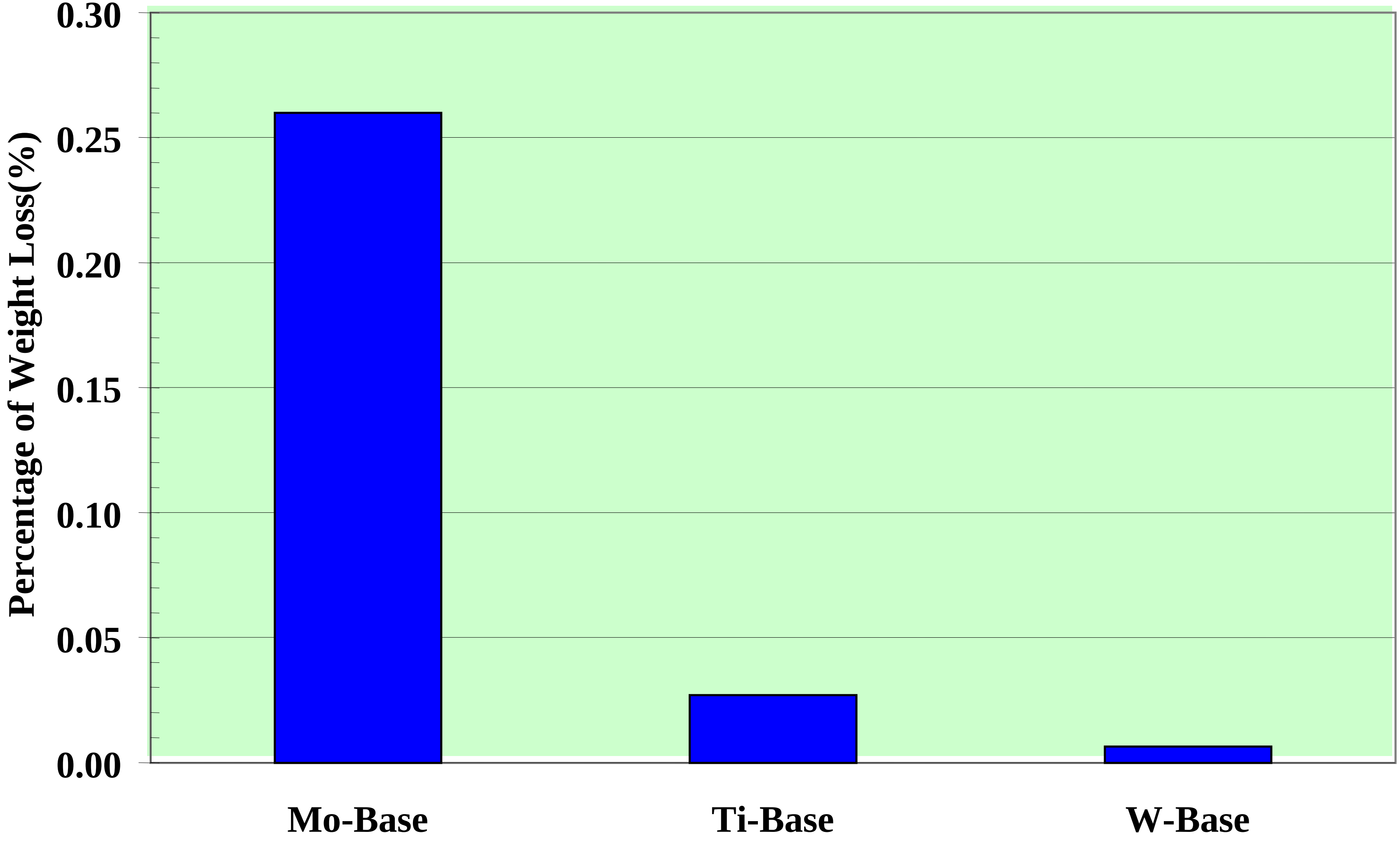
[4] S. Gulizia, M. Z. Jahedi, E. D. Doyle, Performance Evaluation of PVD Coatings for High Pressure Die Casting, Surface and Coatings Technolgy, Vol.140 (2001), pp200-205.


Effects of Pin Material on Washout
Determined by weighing the pins before and after shots w/o soldered layer (cleaned with sodium hydroxide)
Weight Loss(g)
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
H-H13
Mo-Base
Ti-Base
Anv.1150
50 Shots
100 Shots
100 Shots
100 Shots

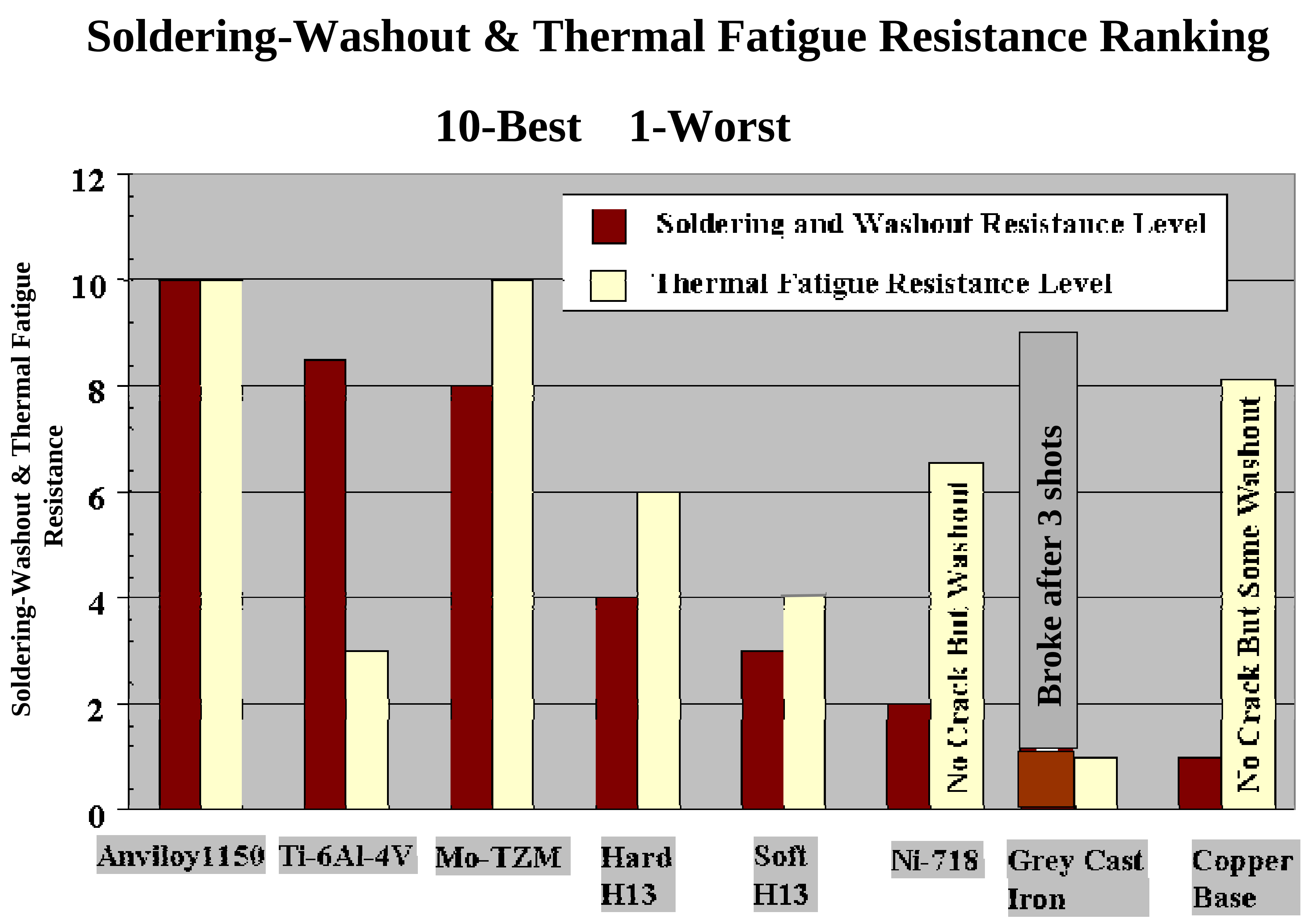
Washout Induced Weight Loss



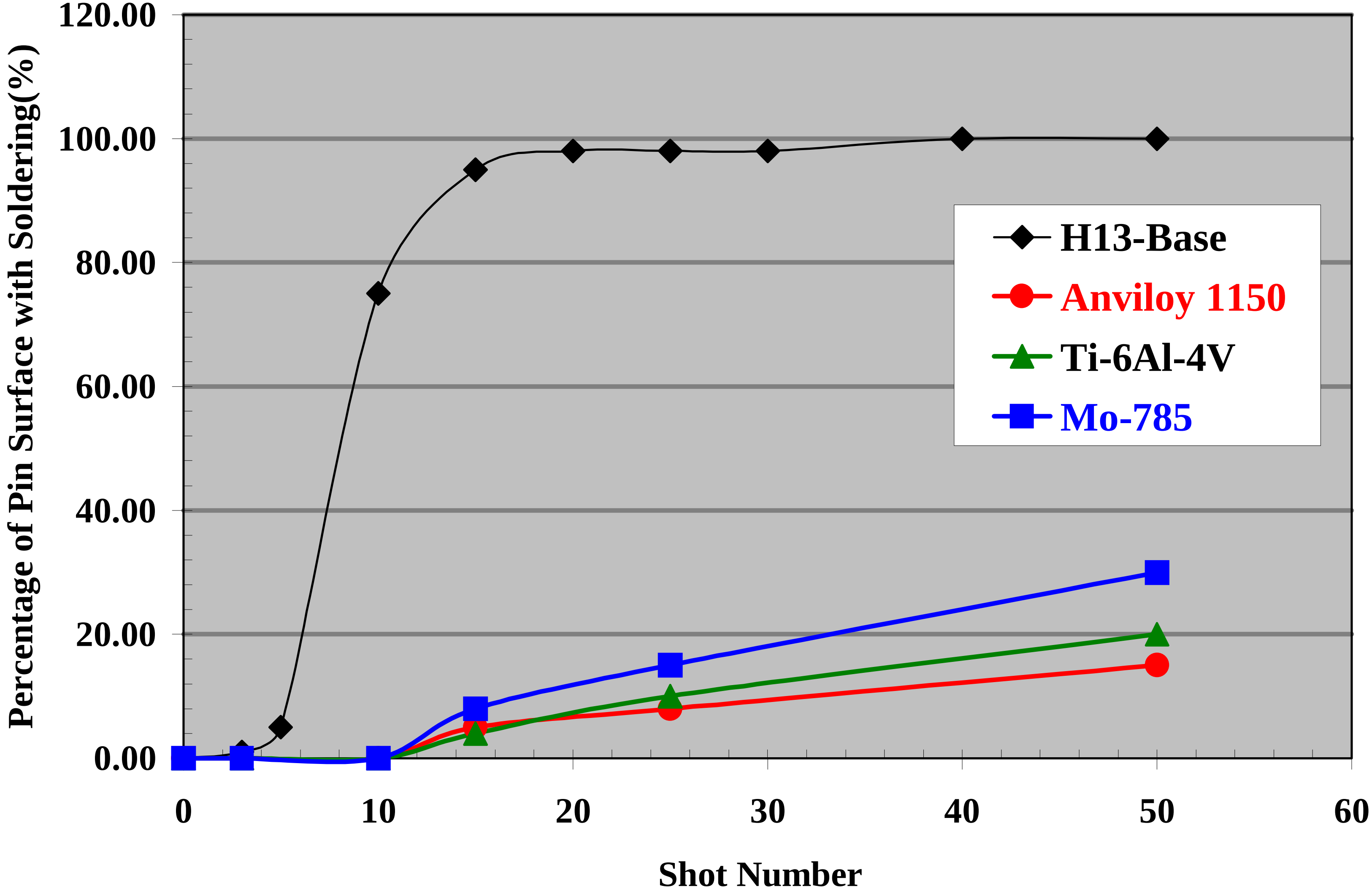
Percentage of Weight Loss



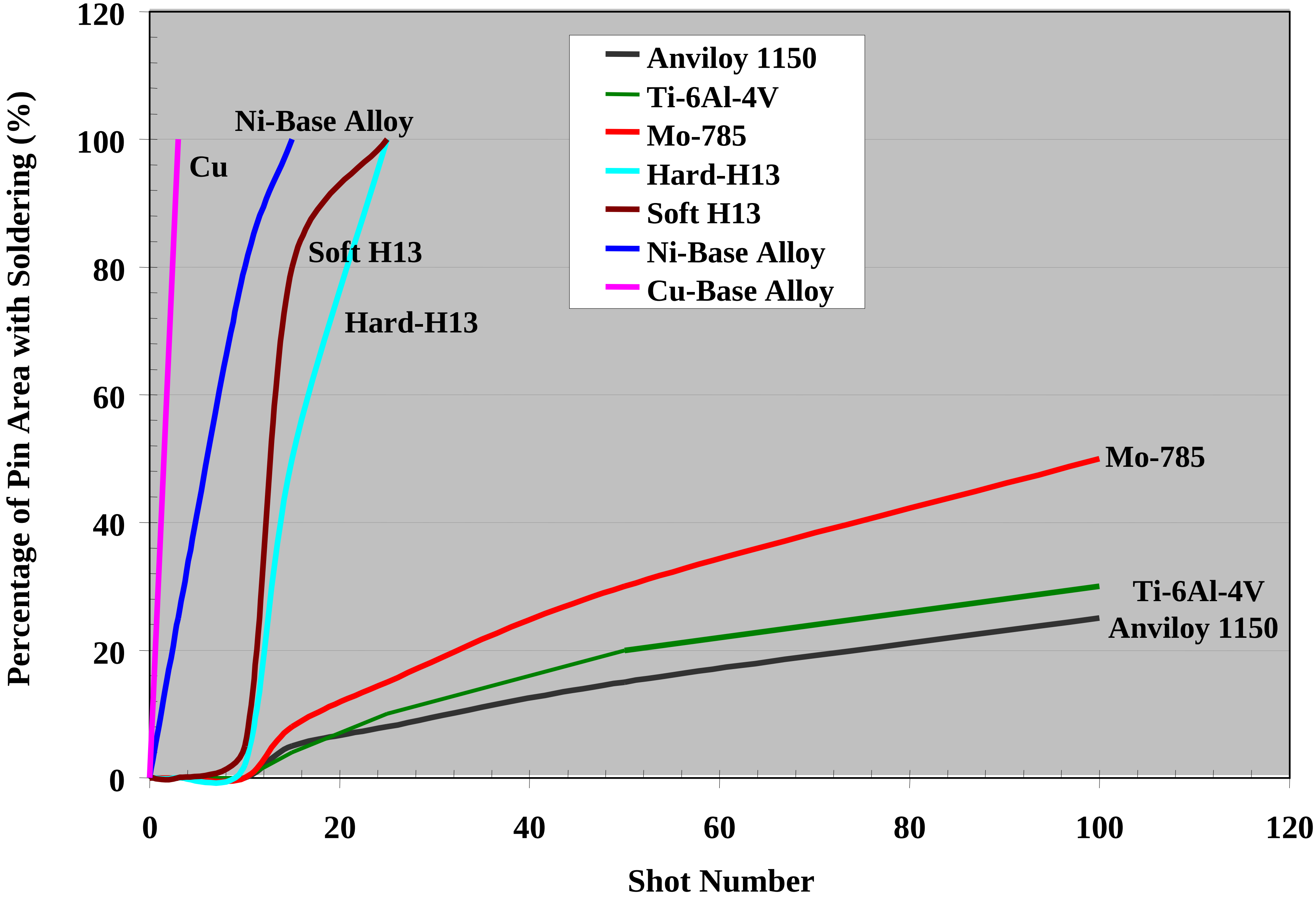
Determined by weighing the pins before and after shots w/o the soldered layer (cleaned with sodium hydroxide)


Soldering-Washout & Thermal Fatigue Resistance Ranking
10-Best 1-Worst
Soldering-Washout & Thermal Fatigue Resistance
12
10
8
6
4
2
0
Soldering and Washout Resistance Level
Thermal Fatigue Resistance Level
No Crack But Washout
Broke after 3 shots
No Crack But Some Washout
Anviloy1150
Ti-6Al-4V
Mo-TZM
Hard H13
Soft H13
Ni-718
Grey Cast Iron
Copper Base

Effect of Pin Material on Soldering



Effects of Pin Material & Number of Shots on Percentage Area Covered with Soldering



CONCLUSIONS

- **The Washout/Soldering set-up allows exposure of die materials to a jet of molten aluminum (ca. 300 in/sec). Each shot is about six pounds of molten metal.**

- **The two pins arrangement allows for comparing the evaluated material to a control H13.**

- **Evidence of severe washout has been observed in copper based alloys after three shots. Alloys with mutual solubility in aluminum are prone to dissolve when impinged by a hot jet of molten aluminum.**

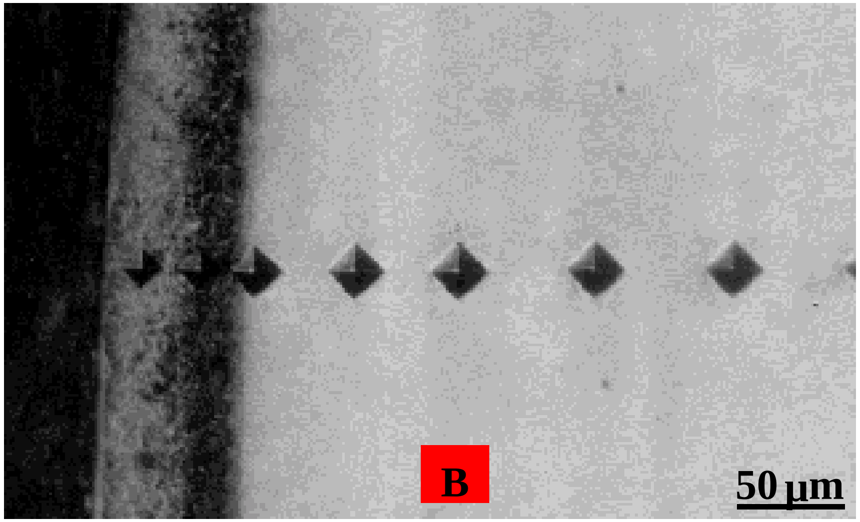
- **Nickel base alloys form intermetallic compounds with aluminum. These compounds promote soldering, and bonding of the casting to the pins. As a result, the pins elongate during ejection.**

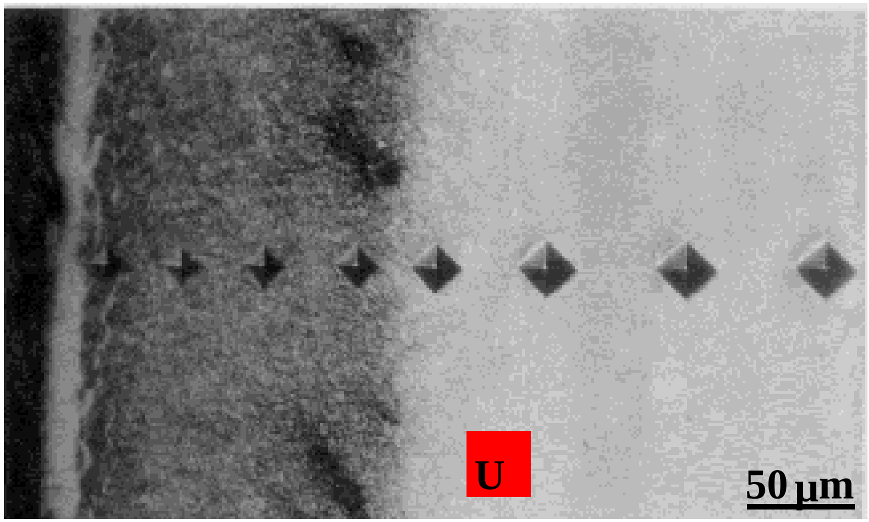
CONCLUSIONS (cont.)

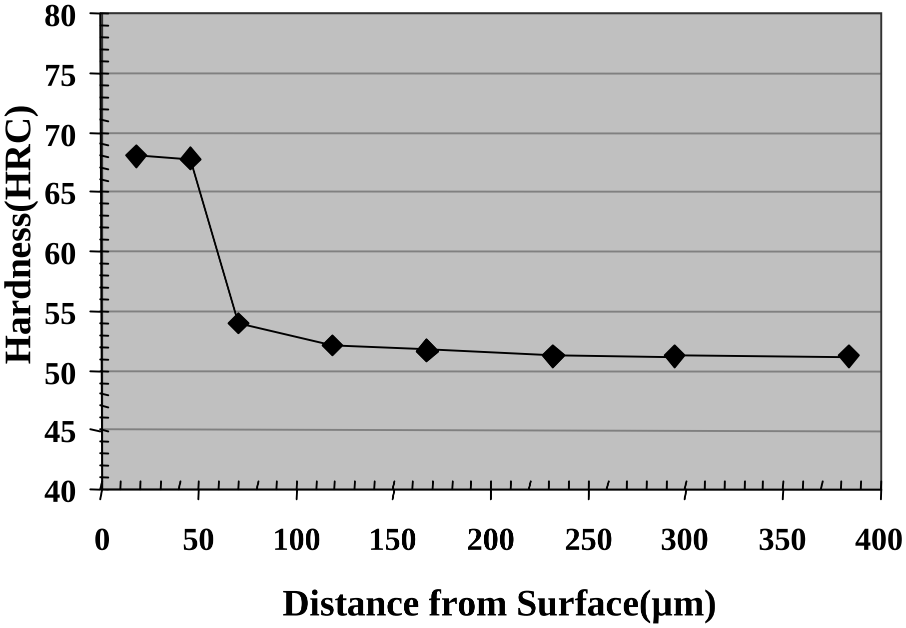
- **Hard H13 has better soldering and washout resistance than soft H13.**

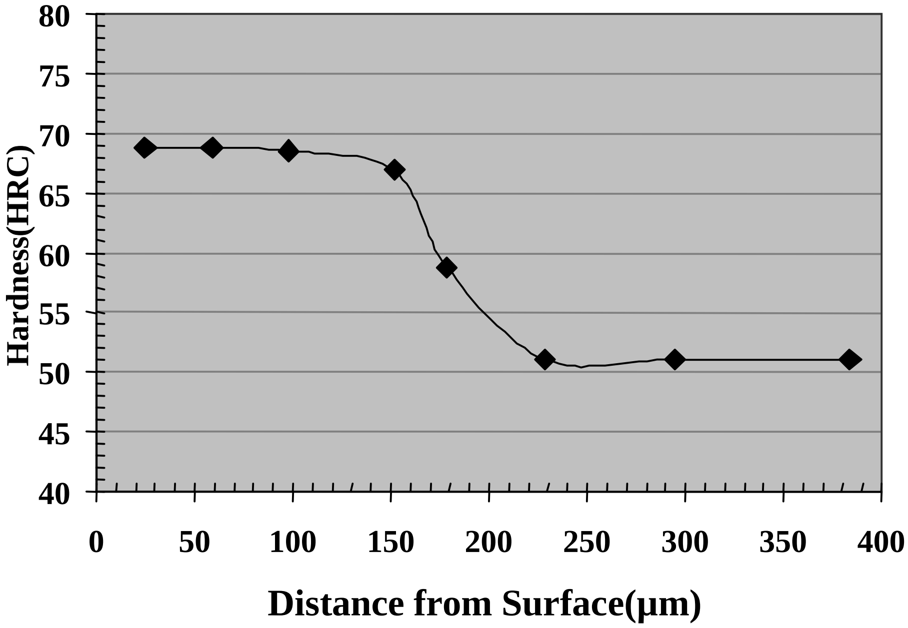
- **The best soldering and washout resistance in this study is exhibited by Anviloy, followed by Ti-6Al-4V and Mo alloys. This behavior is consistent with the low chemical affinity to Al and high melting temperature of these alloys.**

Hardness Distribution in Nitro-Carburized Diffusion Layers

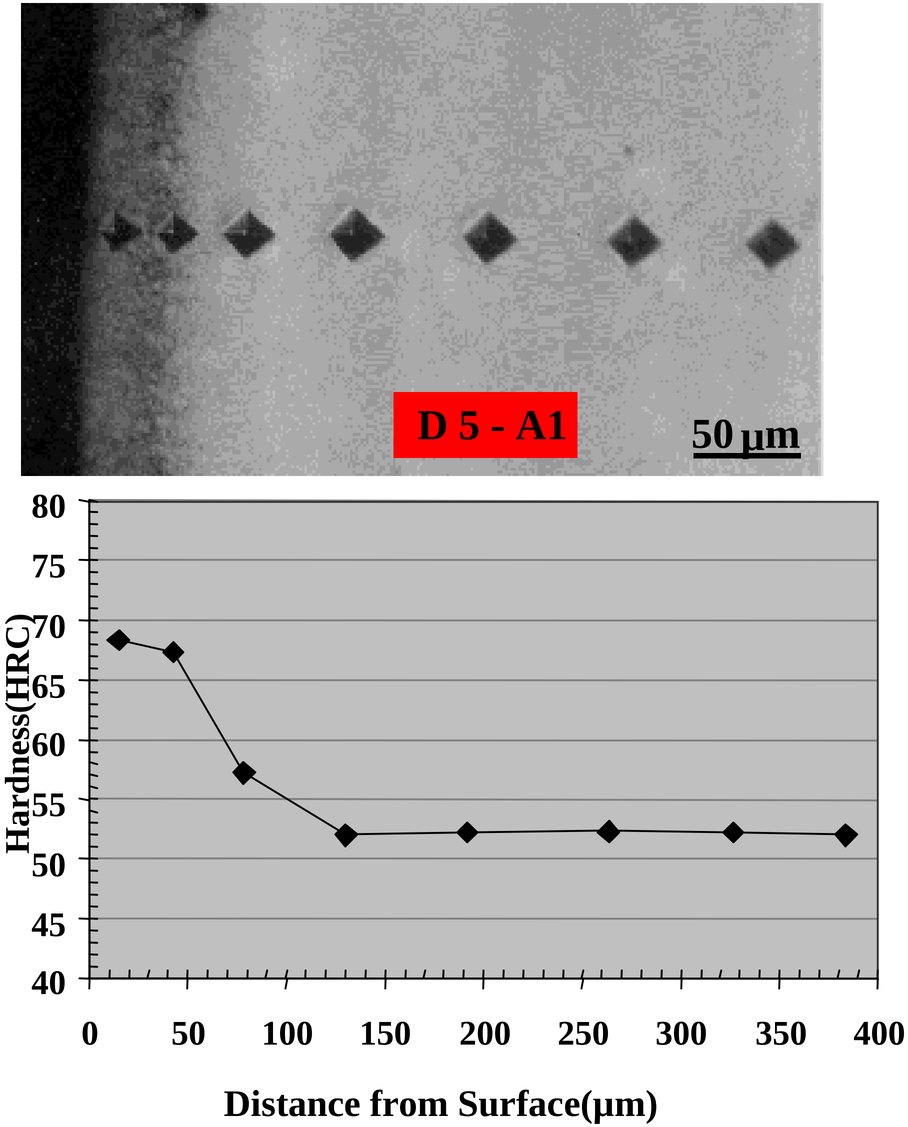


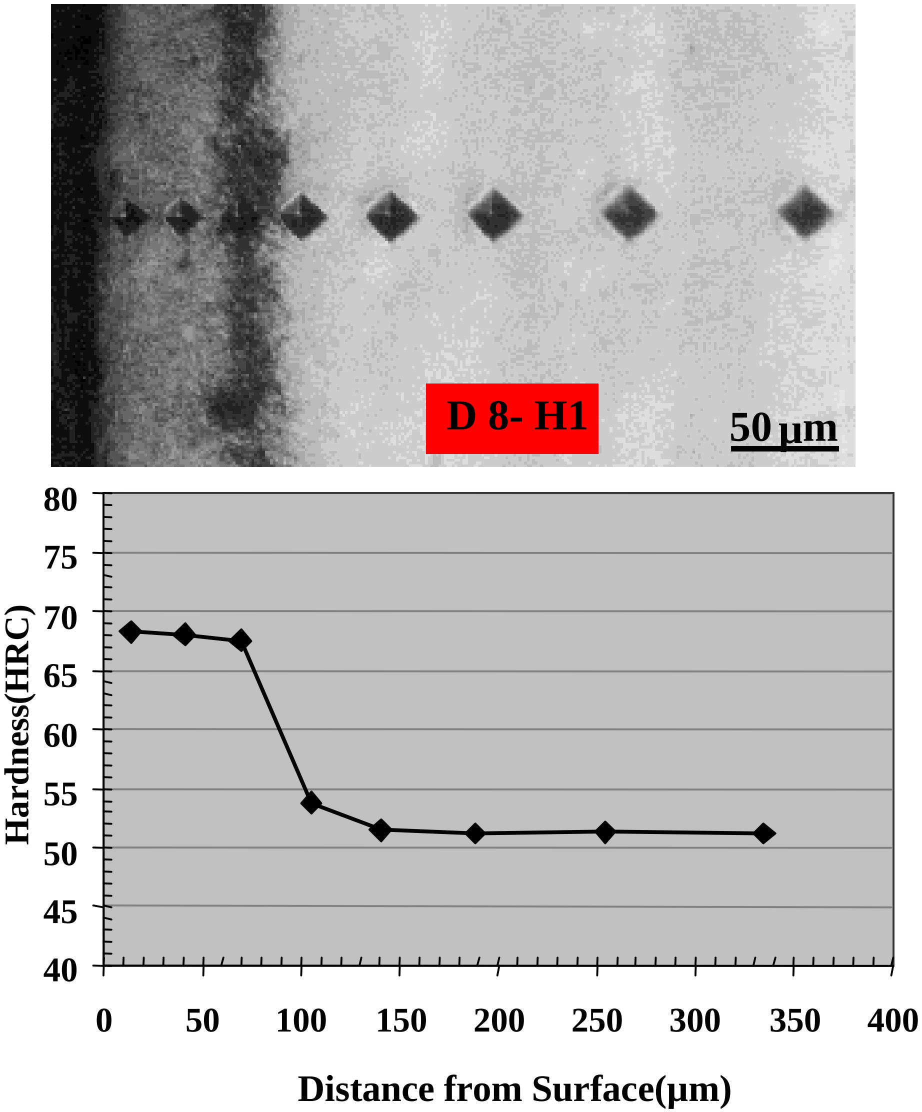




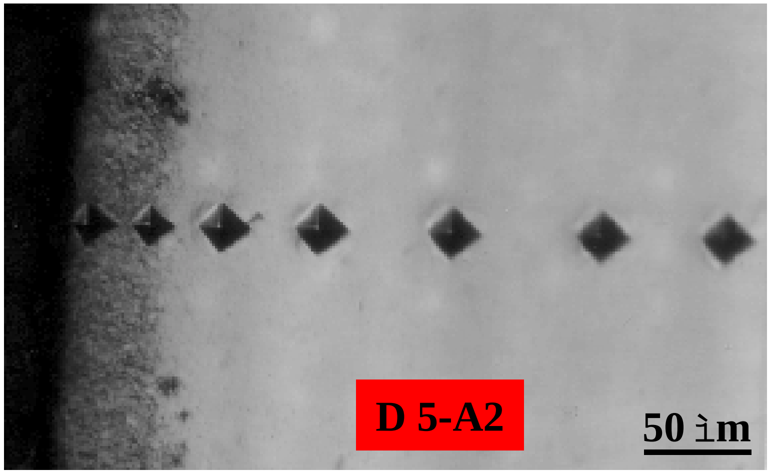


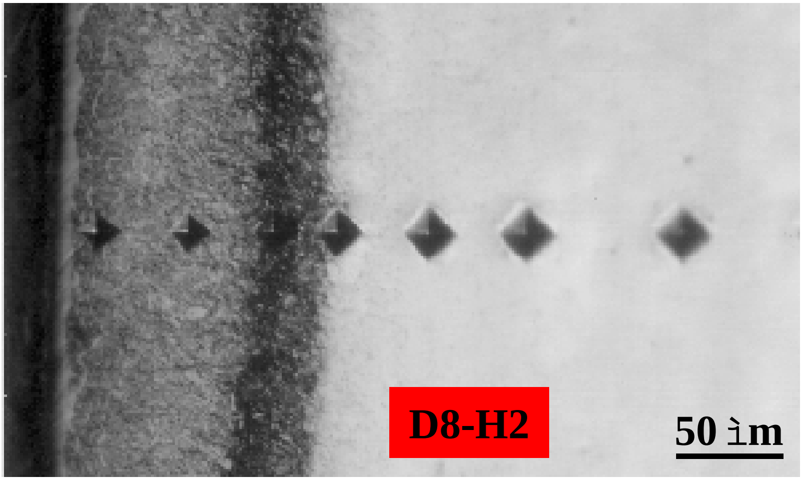
Hardness Distribution in Nitro-Carburized Diffusion Layers

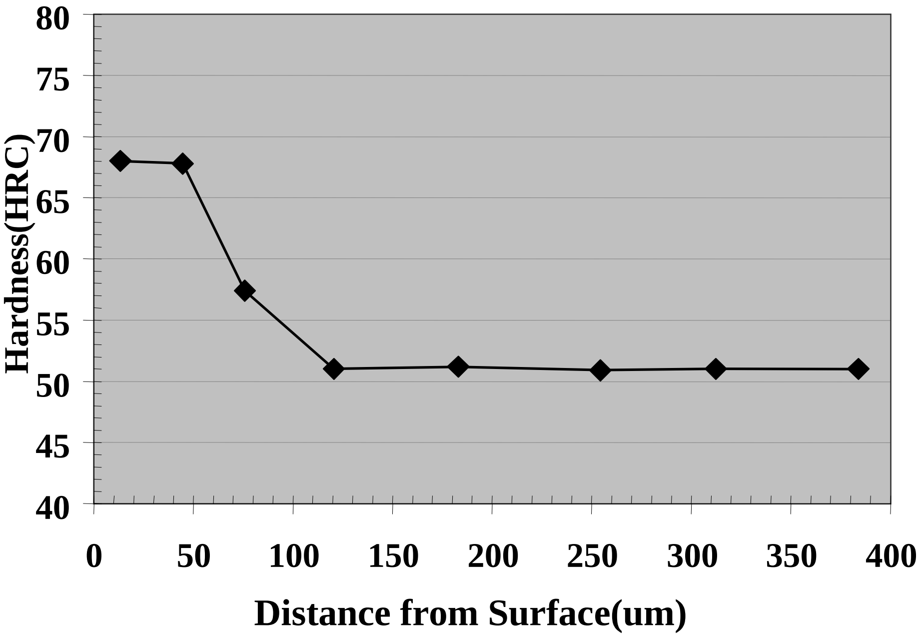


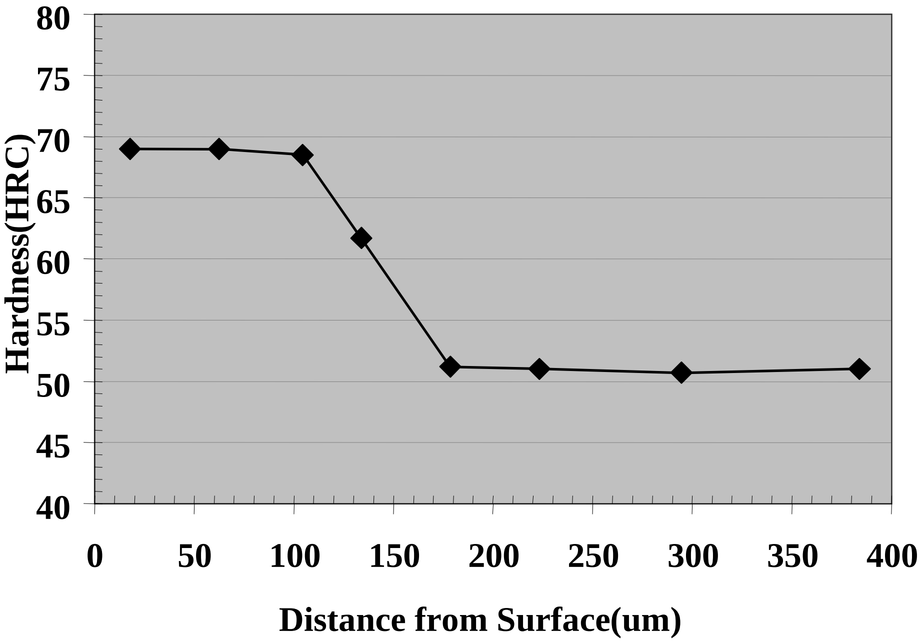


Hardness Distribution in Nitro-Carburized Diffusion Layer

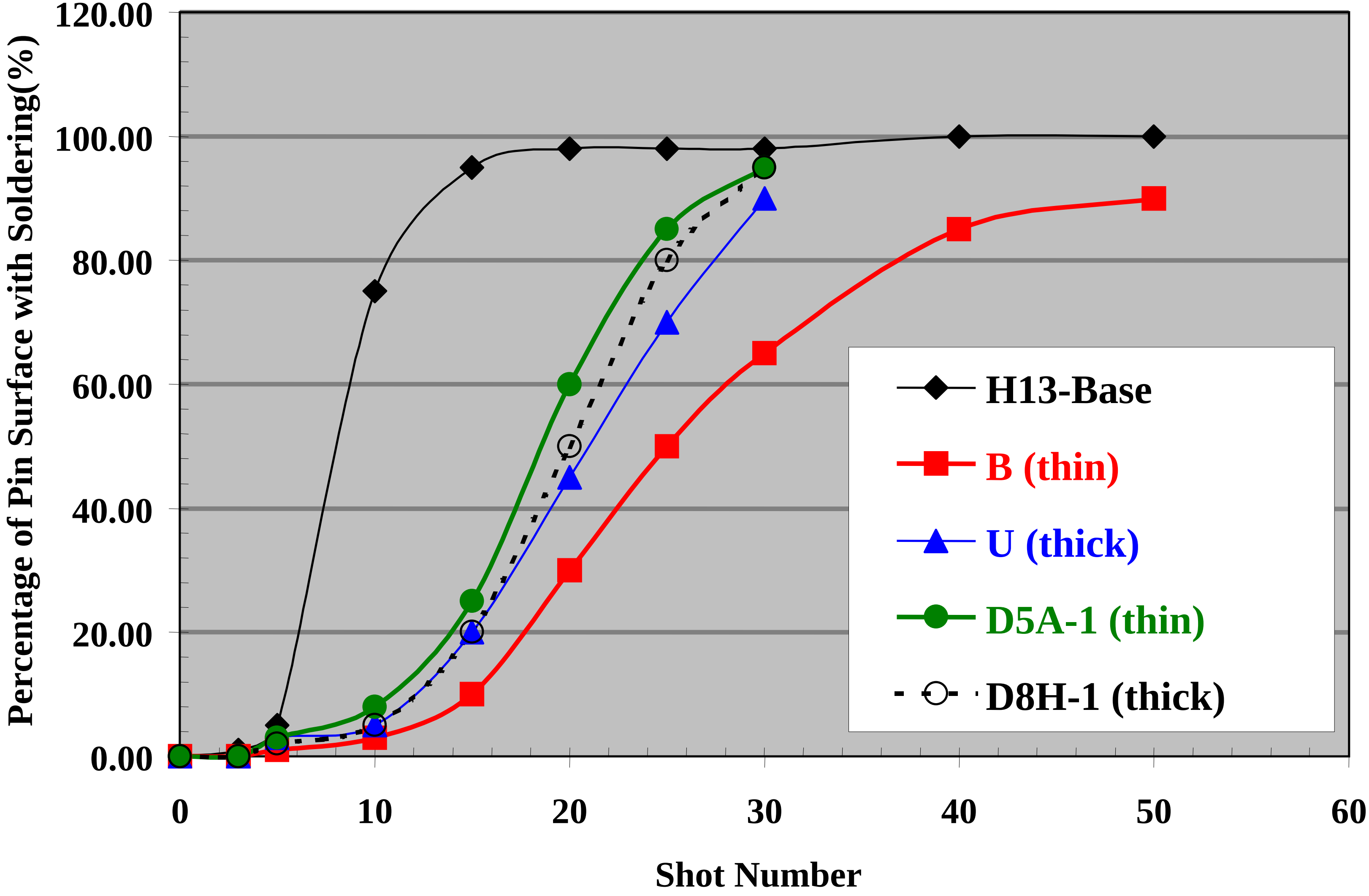




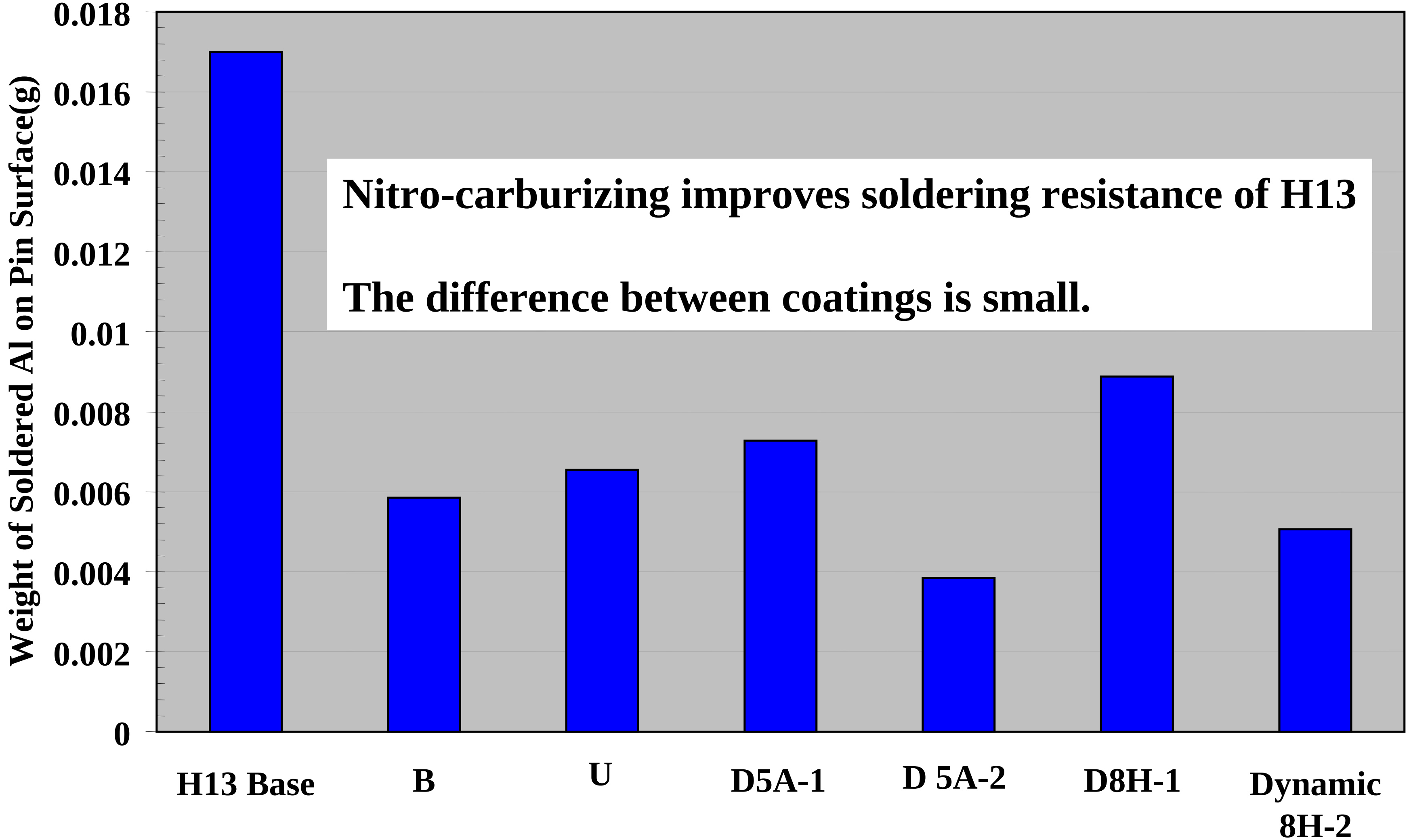




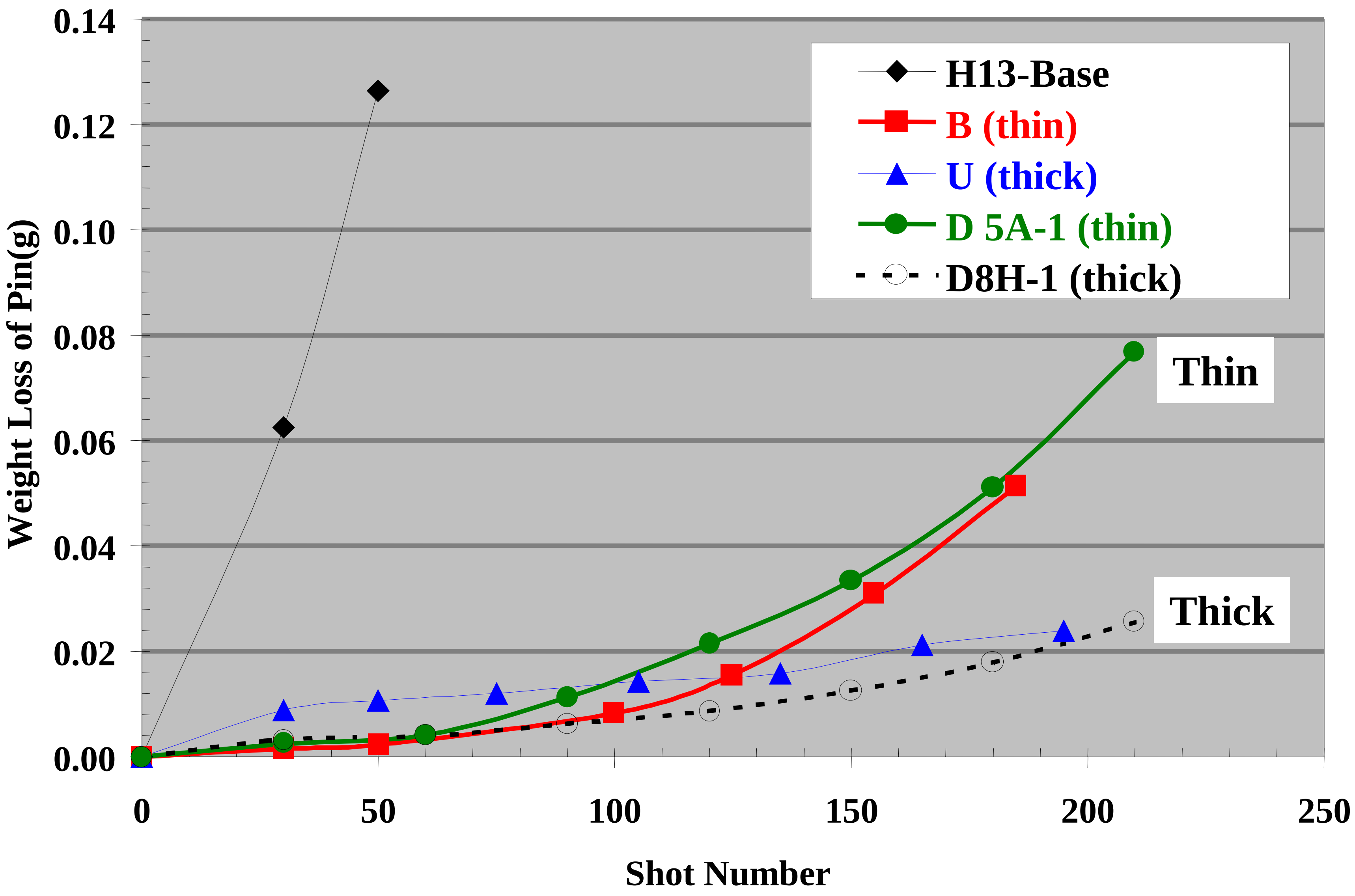
Effect of the Nitro-Carburizing Treatment on Soldering



Effect of Nitro-carburizing on Soldering After 50 Shots

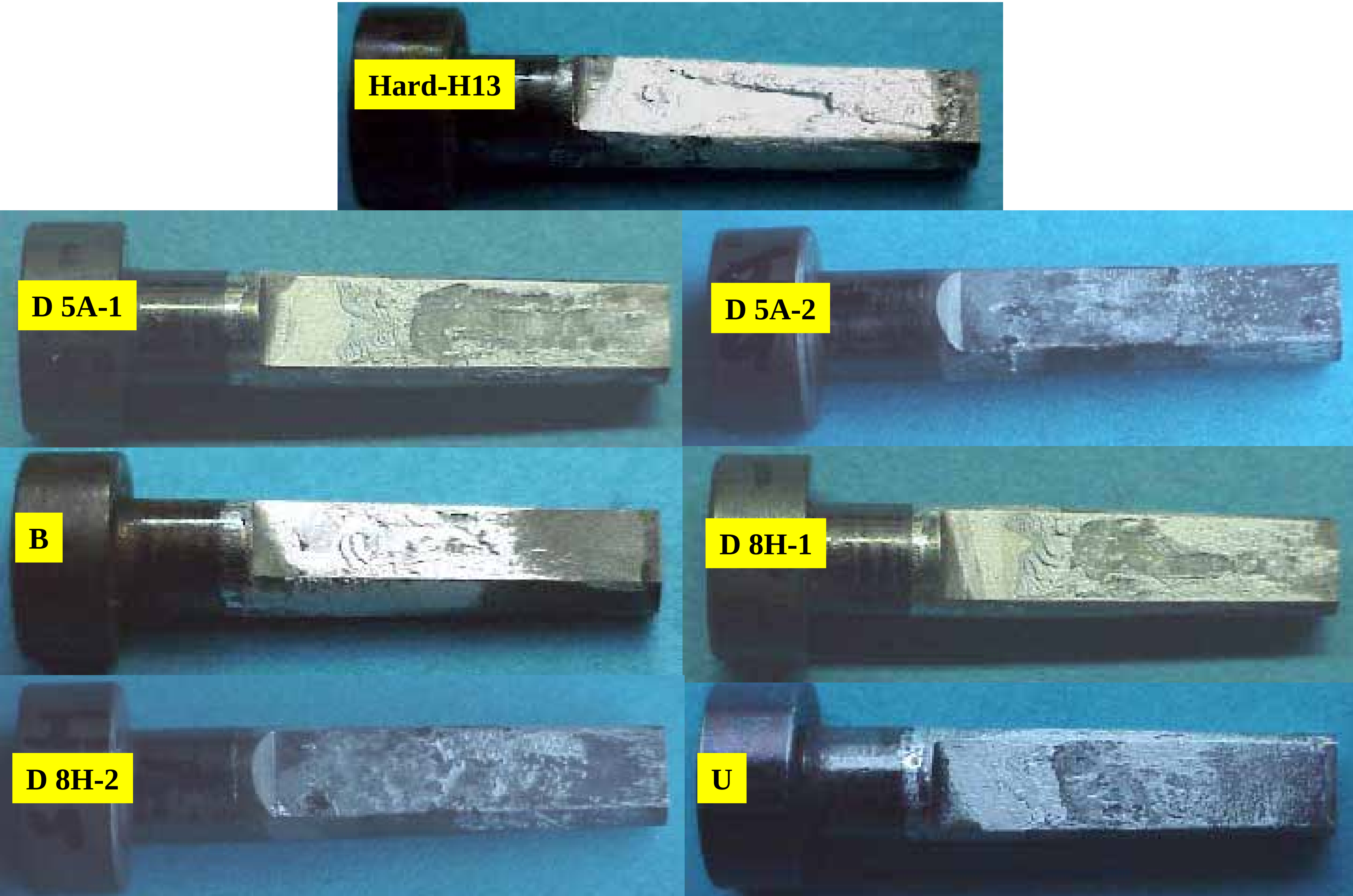


Effect of Nitro-Carburizing on Washout

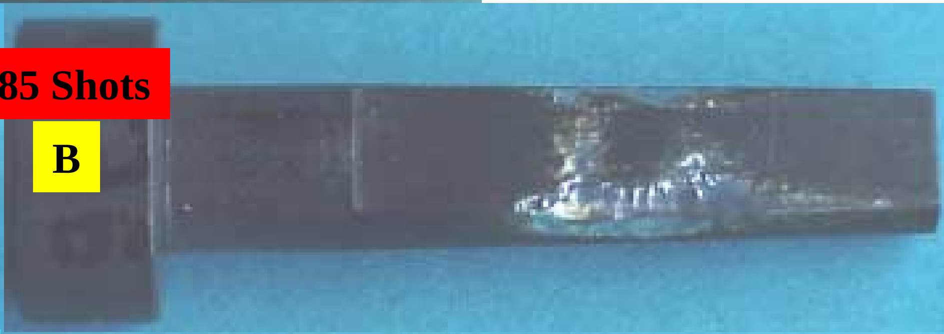


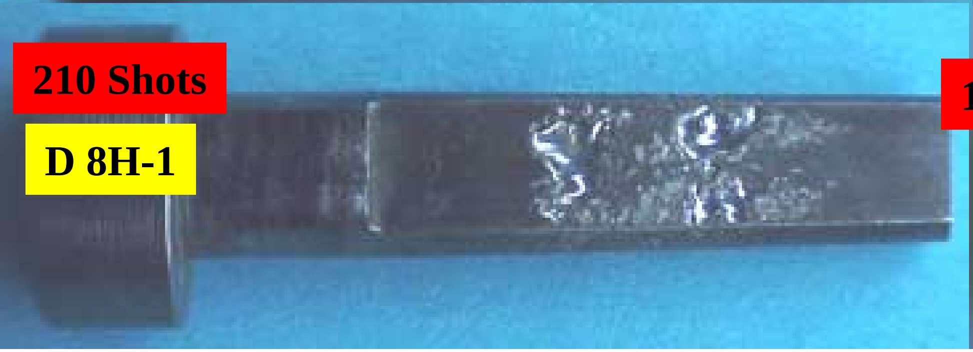
Thick nitro-carburized layers are better for washout than thin layers

Appearance of H13 and Nitro-Carburized Pins after 30 Shots

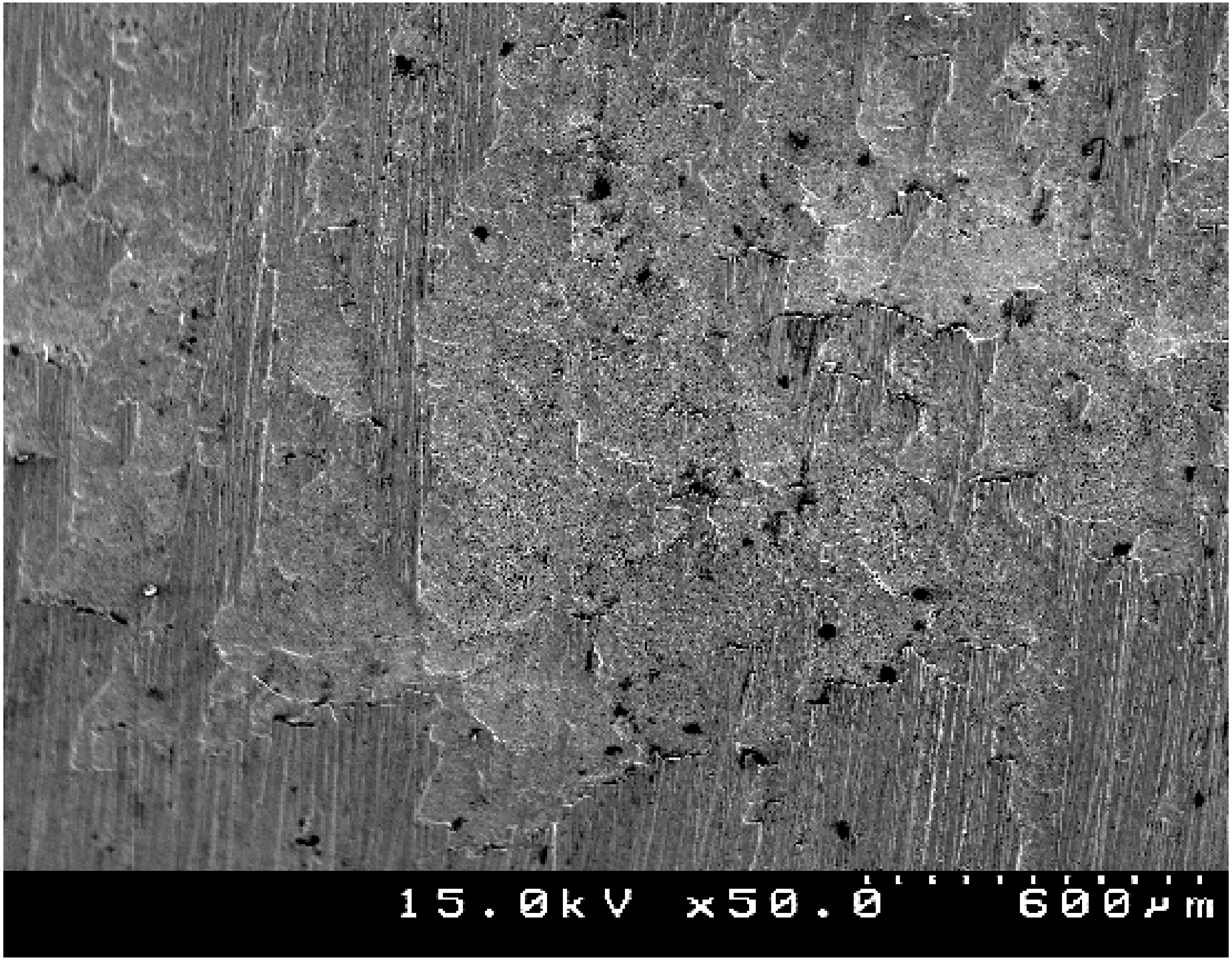


Appearance of H13 and Nitro-Carburized Pins after the Test (Soldered Al Dissolved in NaOH)

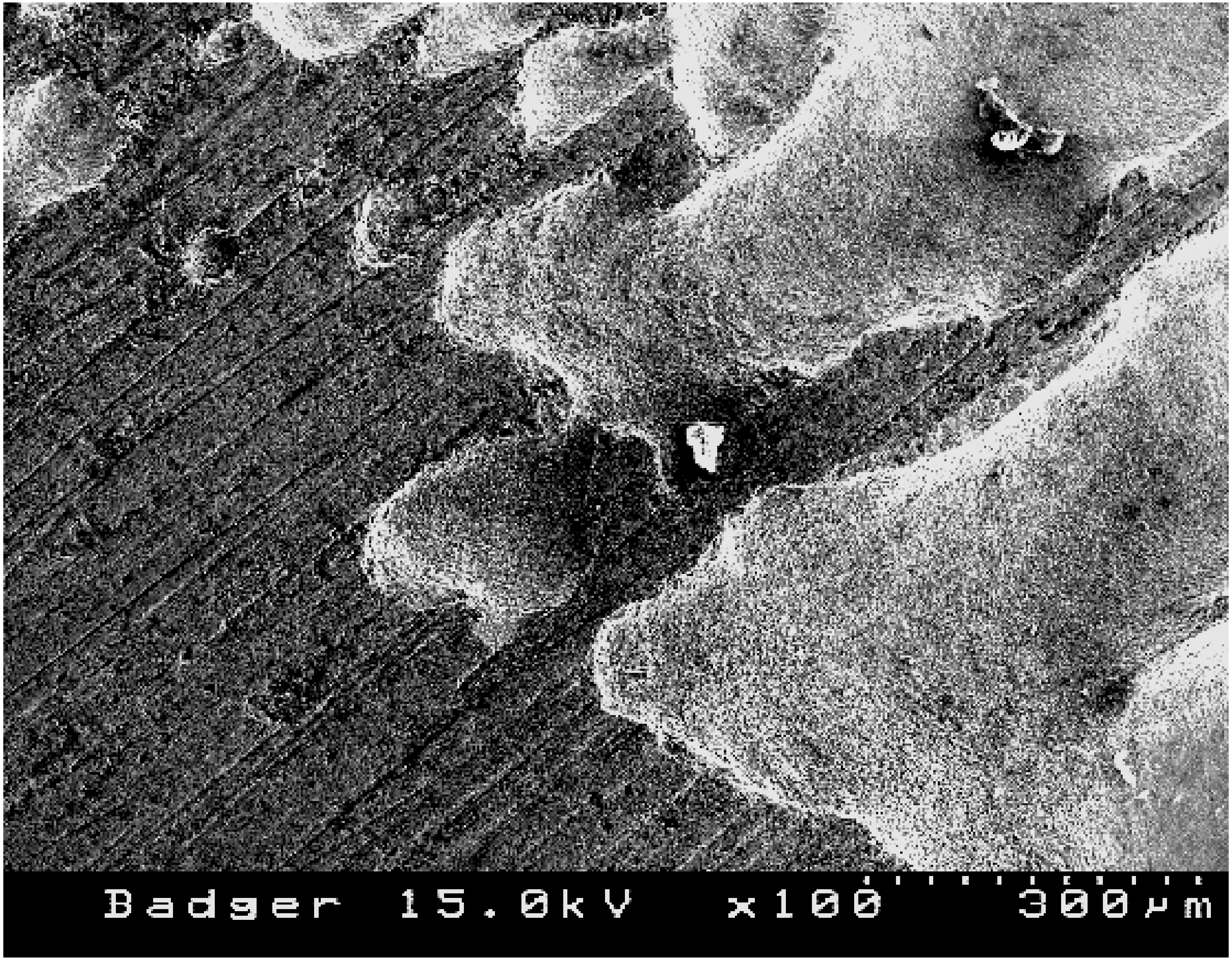




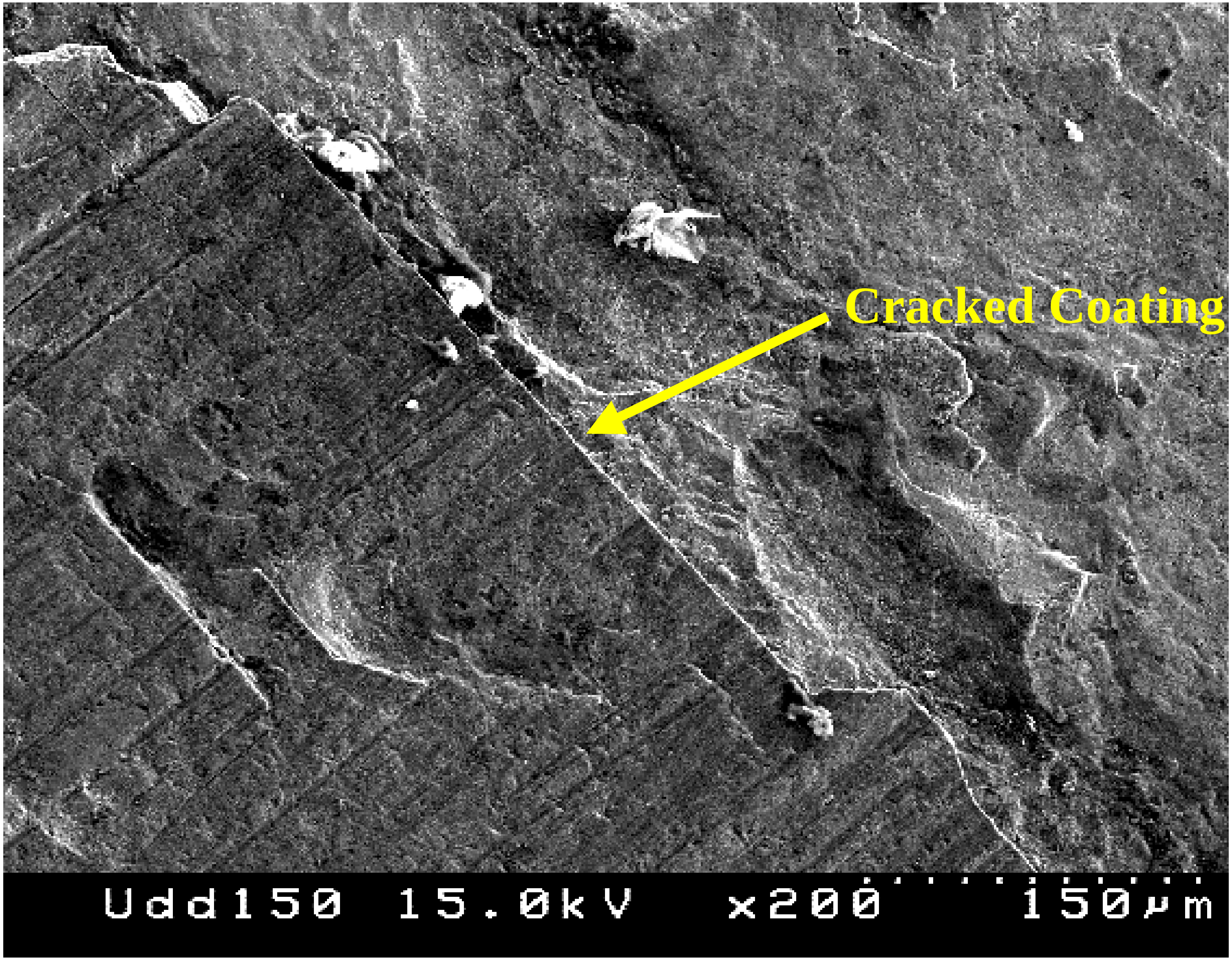
Impingement Surface of <u>Thin</u> Nitro-Carburized layer in "B" Pin (after 50 Shots - Soldered Al Dissolved in NaOH)



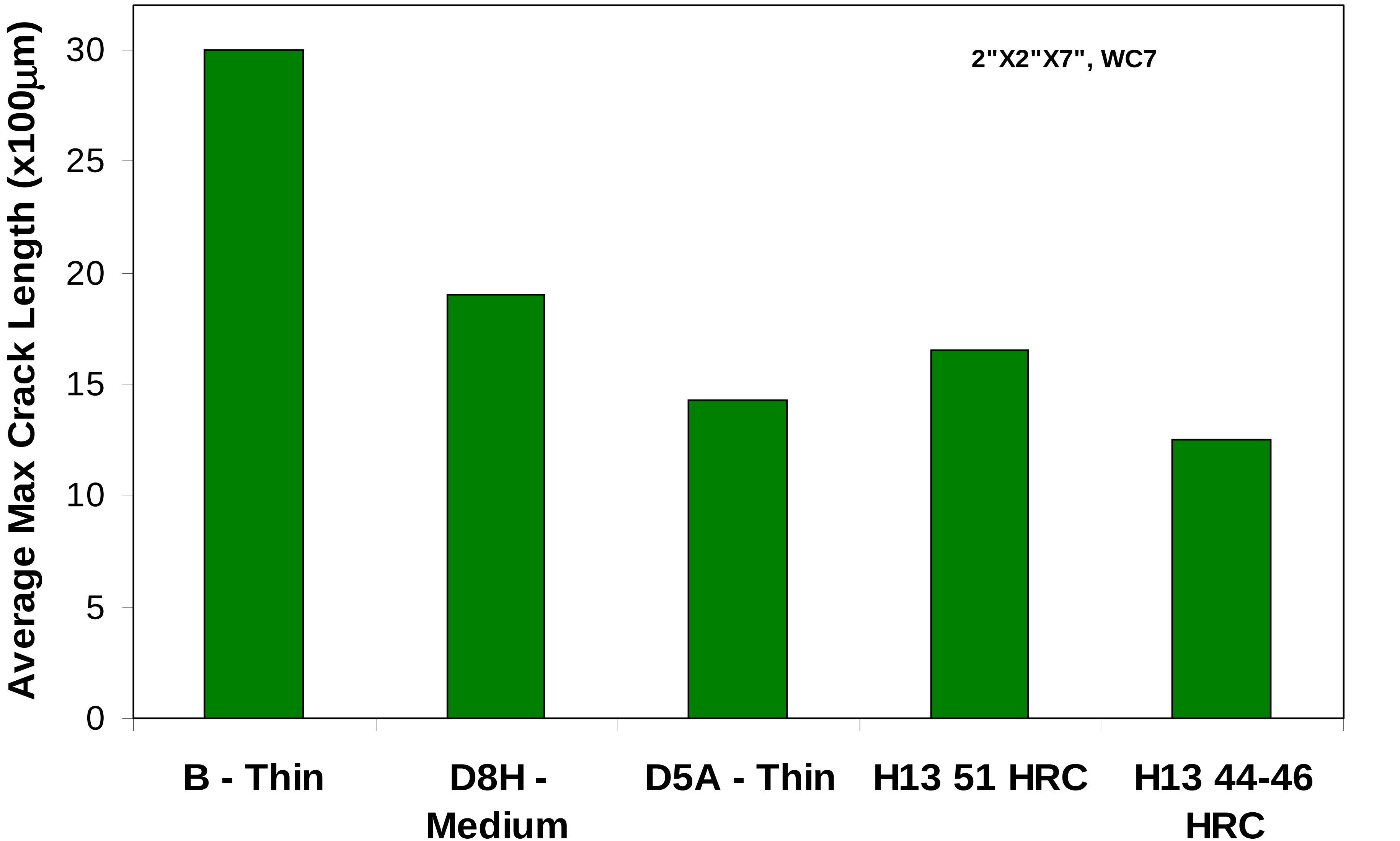
Impingement Surface of <u>Thin</u> Nitro-Carburized layer in "B" Pin (after 150 Shots-Soldered Al Dissolved in NaOH)



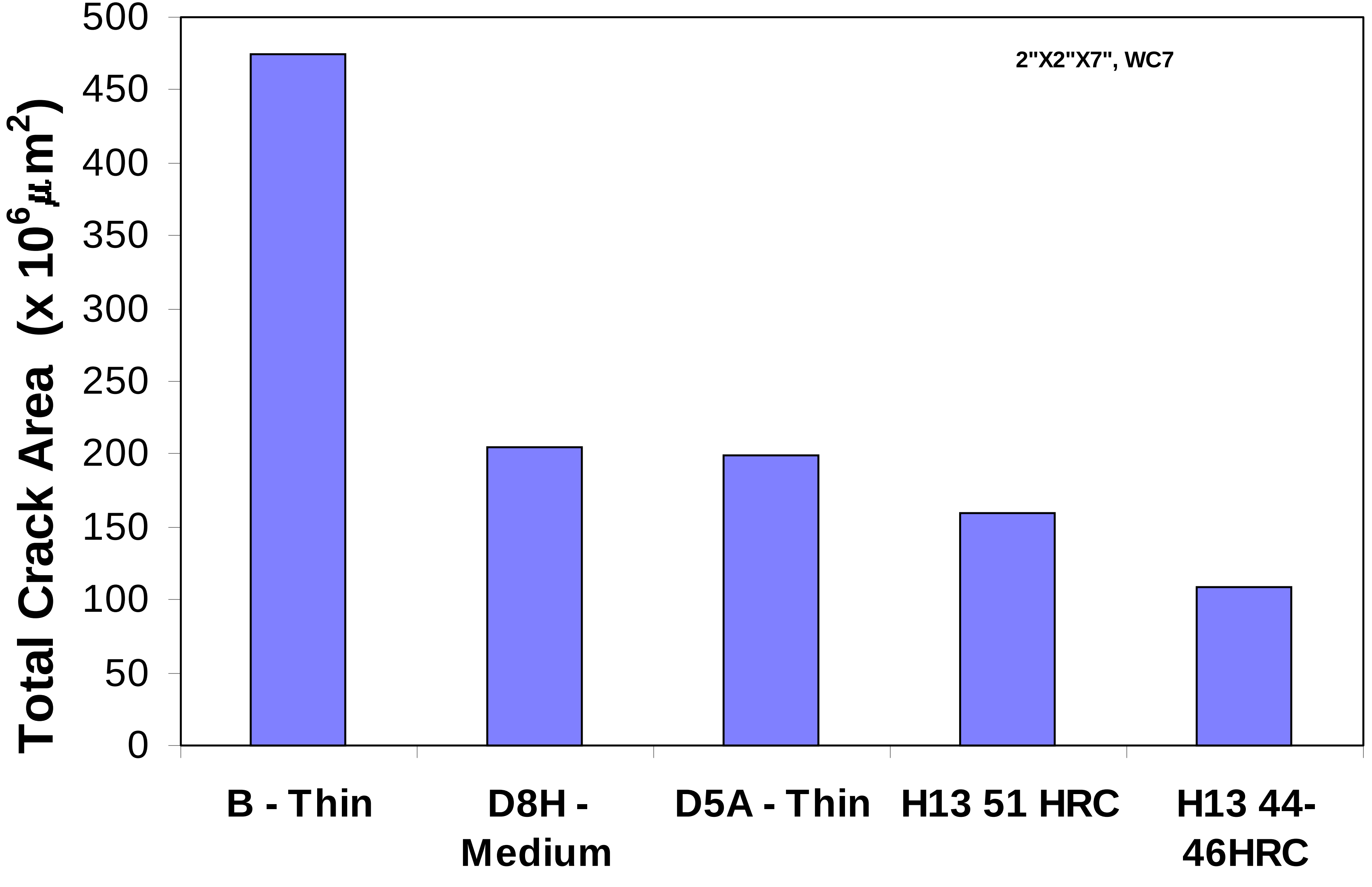
Impingement Surface of <u>Thick</u> Nitro-Carburized Layer in "U" Pin (after 150 Shots and Soldered Al Dissolved in NaOH)



Average Max Crack Length of Nitrocarburized H13



Total Crack Area of Nitrocarburized H13



CONCLUSIONS

1. **Nitro-carburizing improves somewhat the resistance of H13 pins to soldering (sticking of Al) but does not prevent it.**

2. **Nitro-carburizing improves significantly the resistance of H13 pins to washout (loss of base metal).**

3. **Thick nitro-carburized layers provide better resistance to washout. However, they tend to crack more readily than thin coatings.**

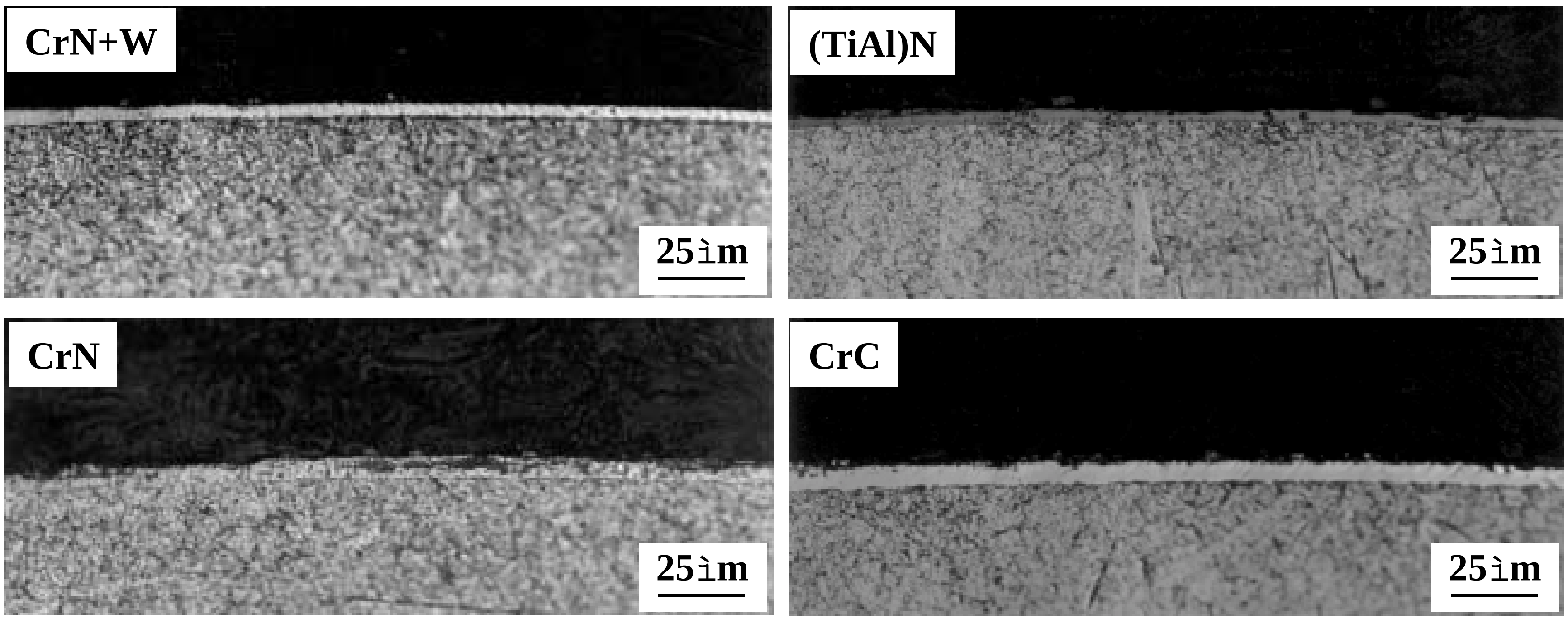
4. **Based on these observations, thick nitro-carburized coatings are desirable in "soldering intensive" applications; thin nitro-carburized coatings may be desirable in "thermal fatigue intensive" applications that can induce cracking.**

Characteristics of PVD Coatings

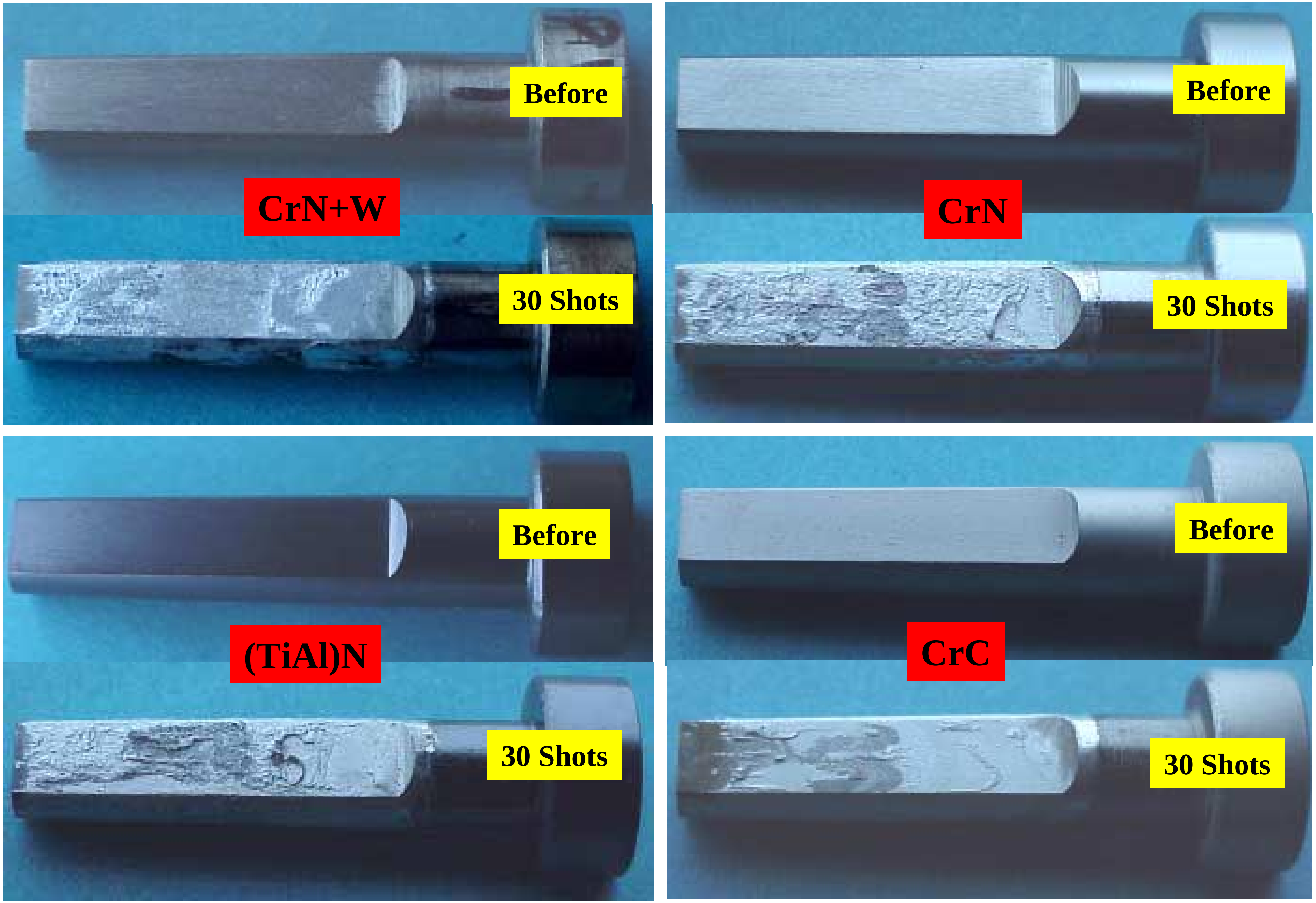
Coating	Coating Process	Thickness* (μm)	Hardness** (Hv -kg/mm^2)	Oxidation Temperature (F)**
CrN+W (I)	PVD	3.5	2500±400	2,278
CrN (P)	PVD	5.0	2500±400	2,278
(TiAl)N (B)	PVD	2.0	2600±400	2,278-2,368
CrC (B)	PVD	6.25	1850±200	2,278

* Measured
** Literature

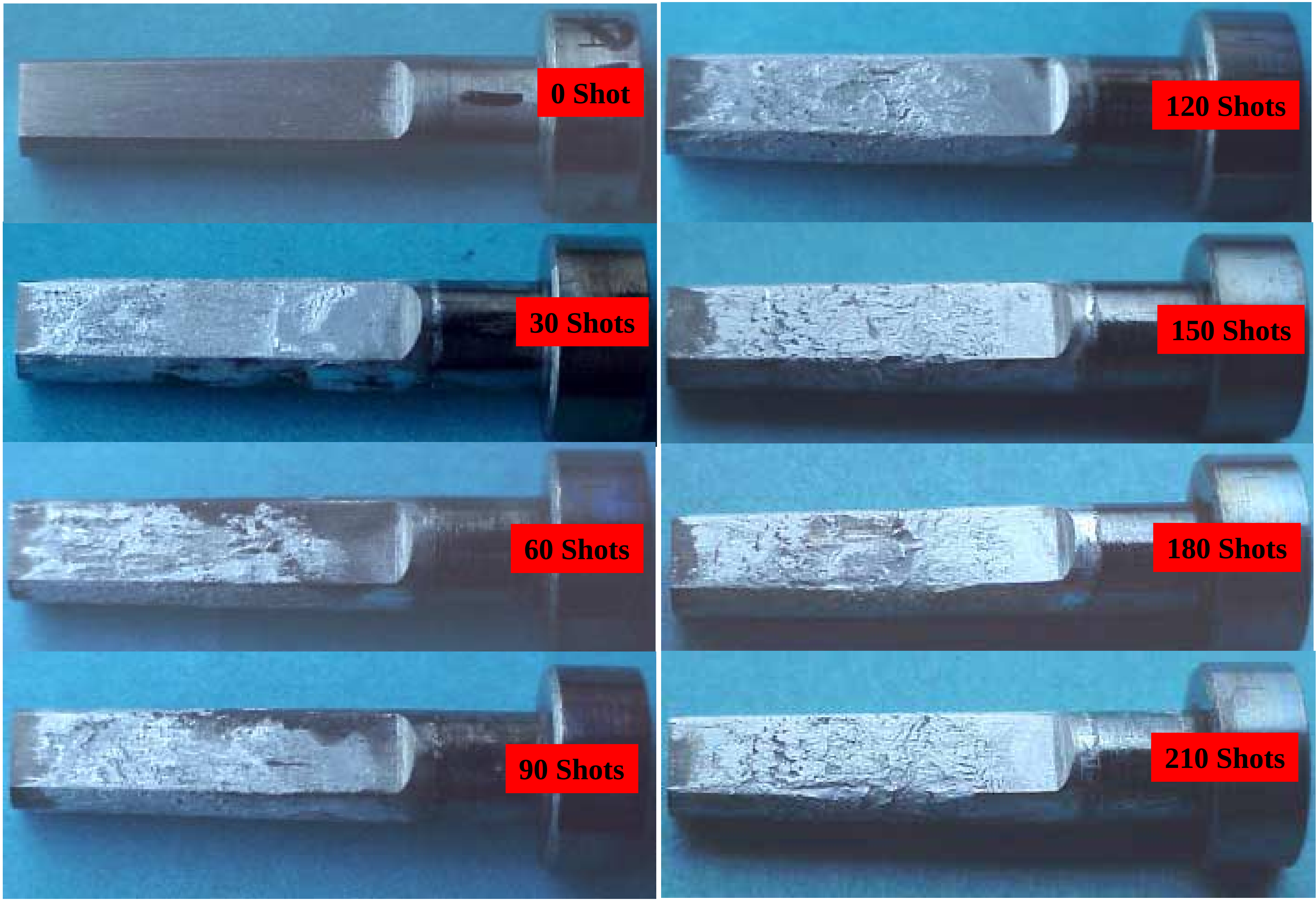
Cross- Section of PVD Coatings and Pin Substrates



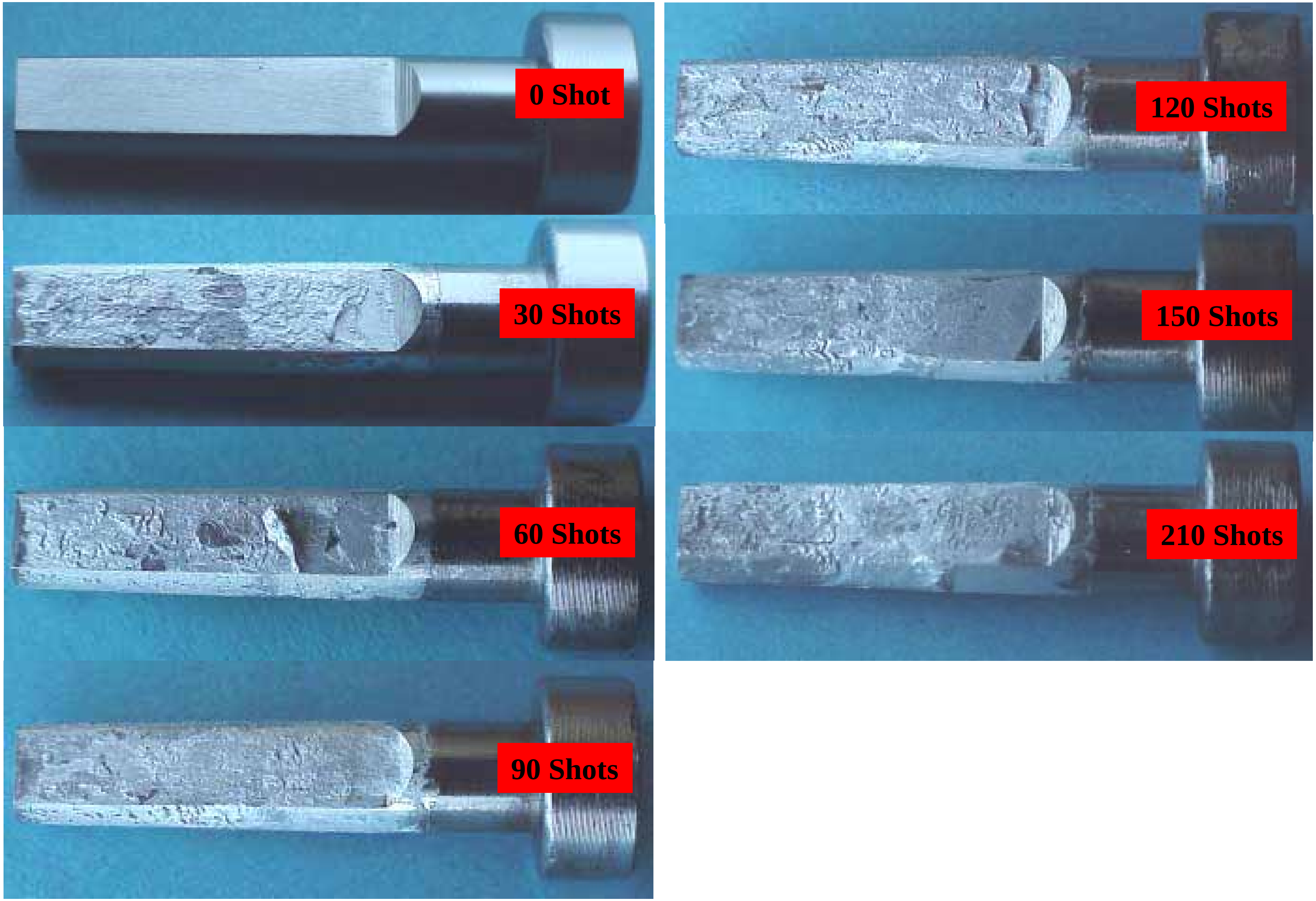
Appearance of PVD Coated Pins before Testing & after 30 Shots



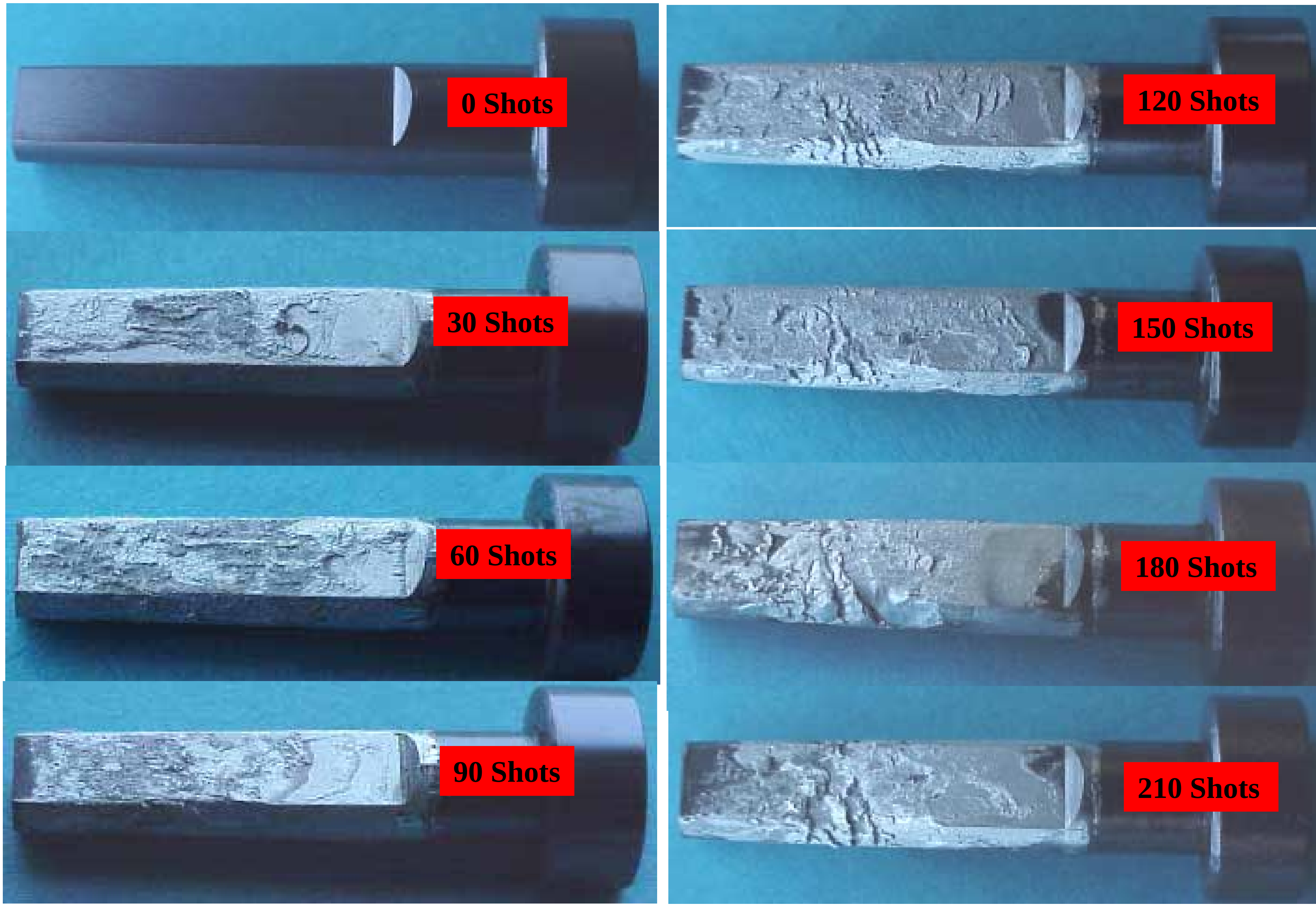
Soldering Appearance of H13 Pin with CrN+W PVD Coating



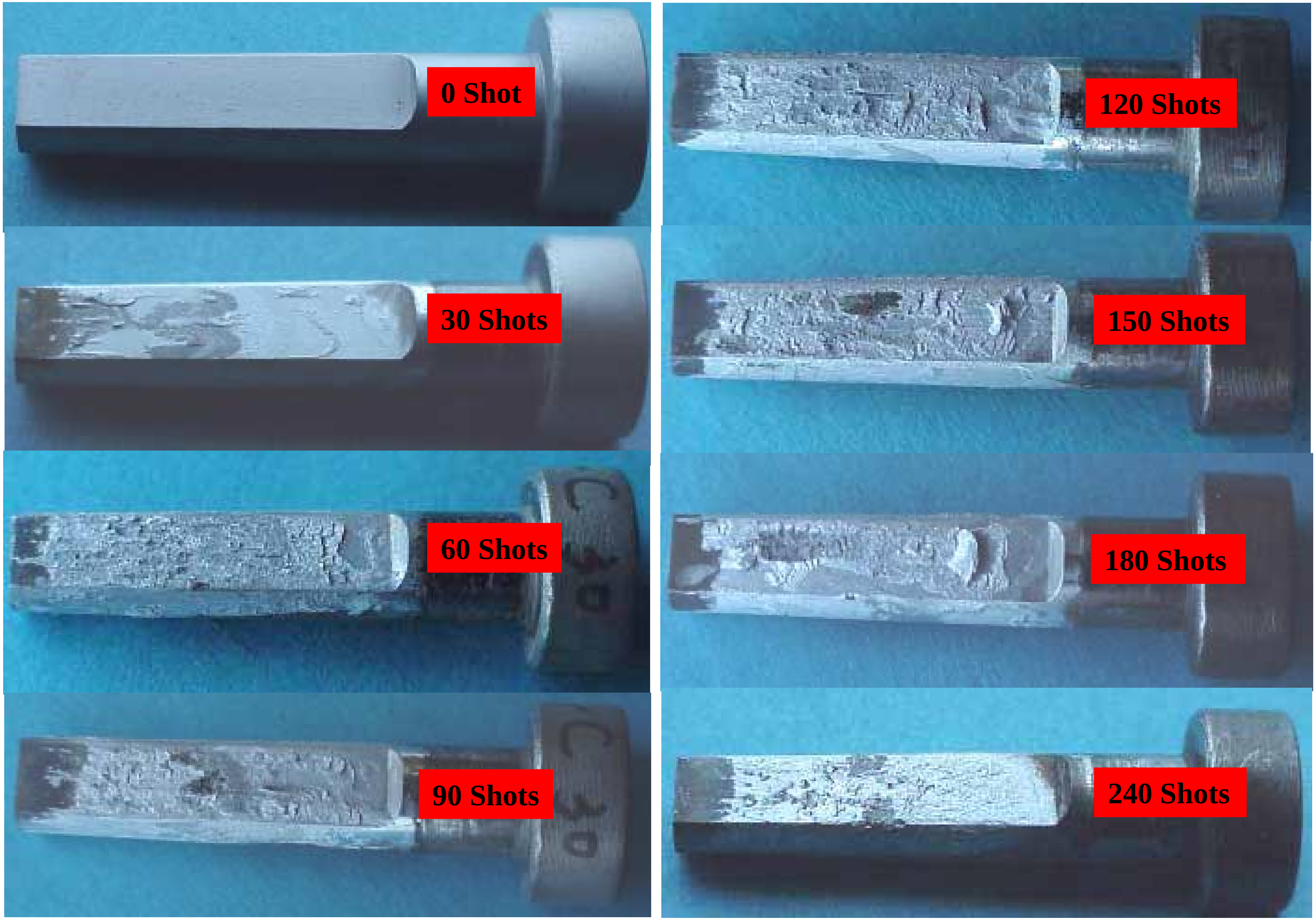
Soldering Appearance of H13 Pin with CrN PVD Coating



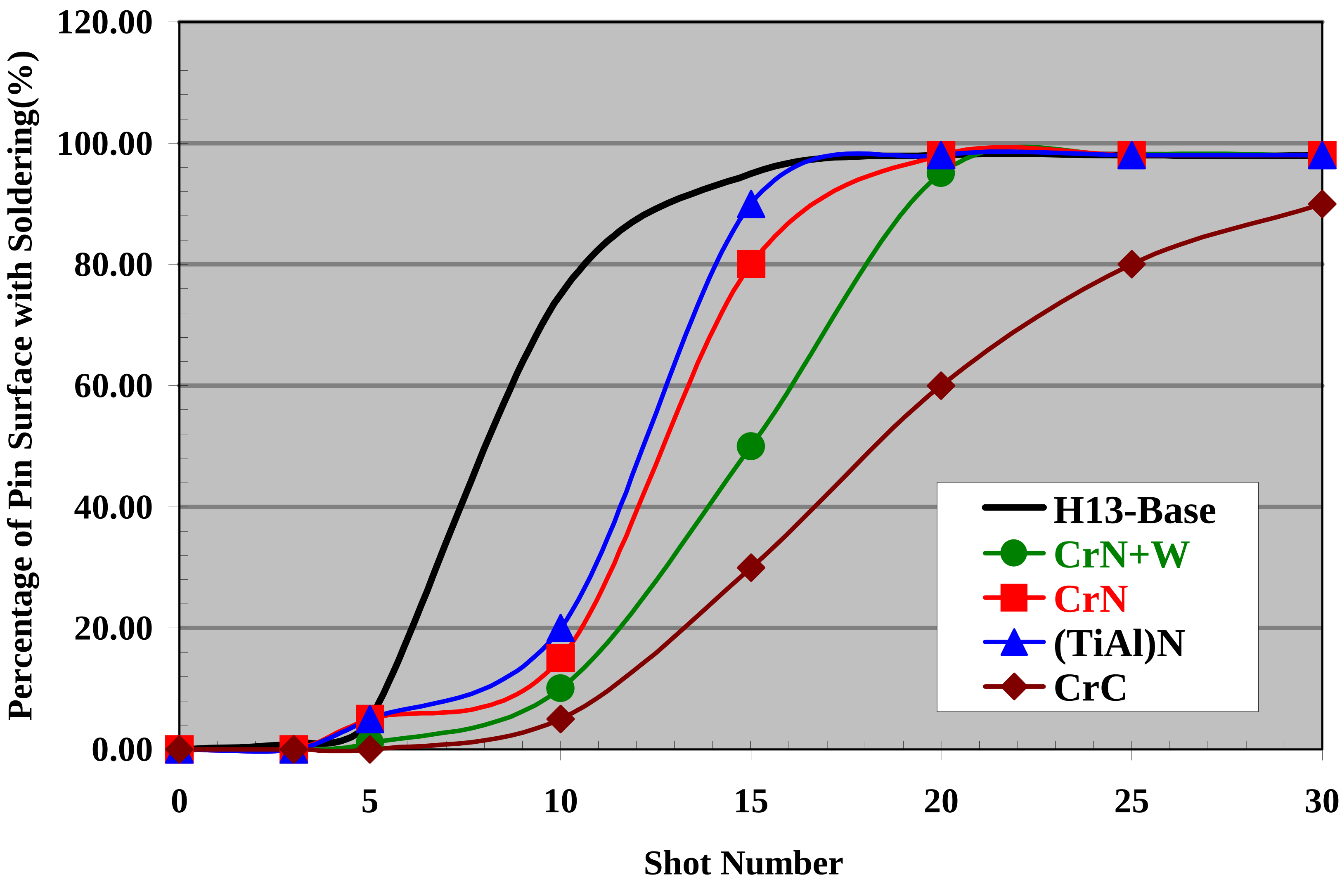
Soldering Appearance of H13 Pin with (TiAl)N PVD Coating



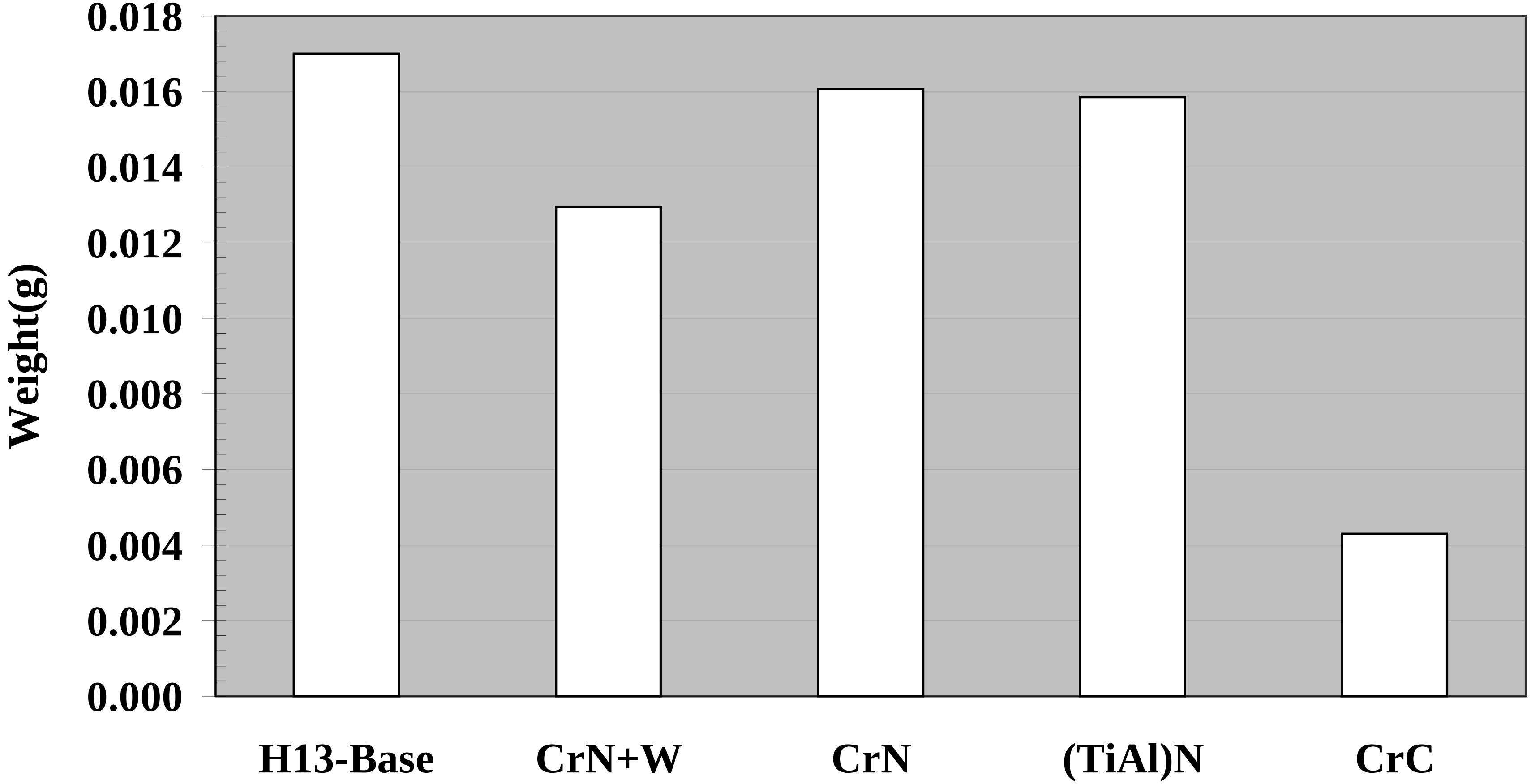
Soldering Appearance of H13 Pin with CrC PVD Coating



Effect of PVD Coating on Soldering



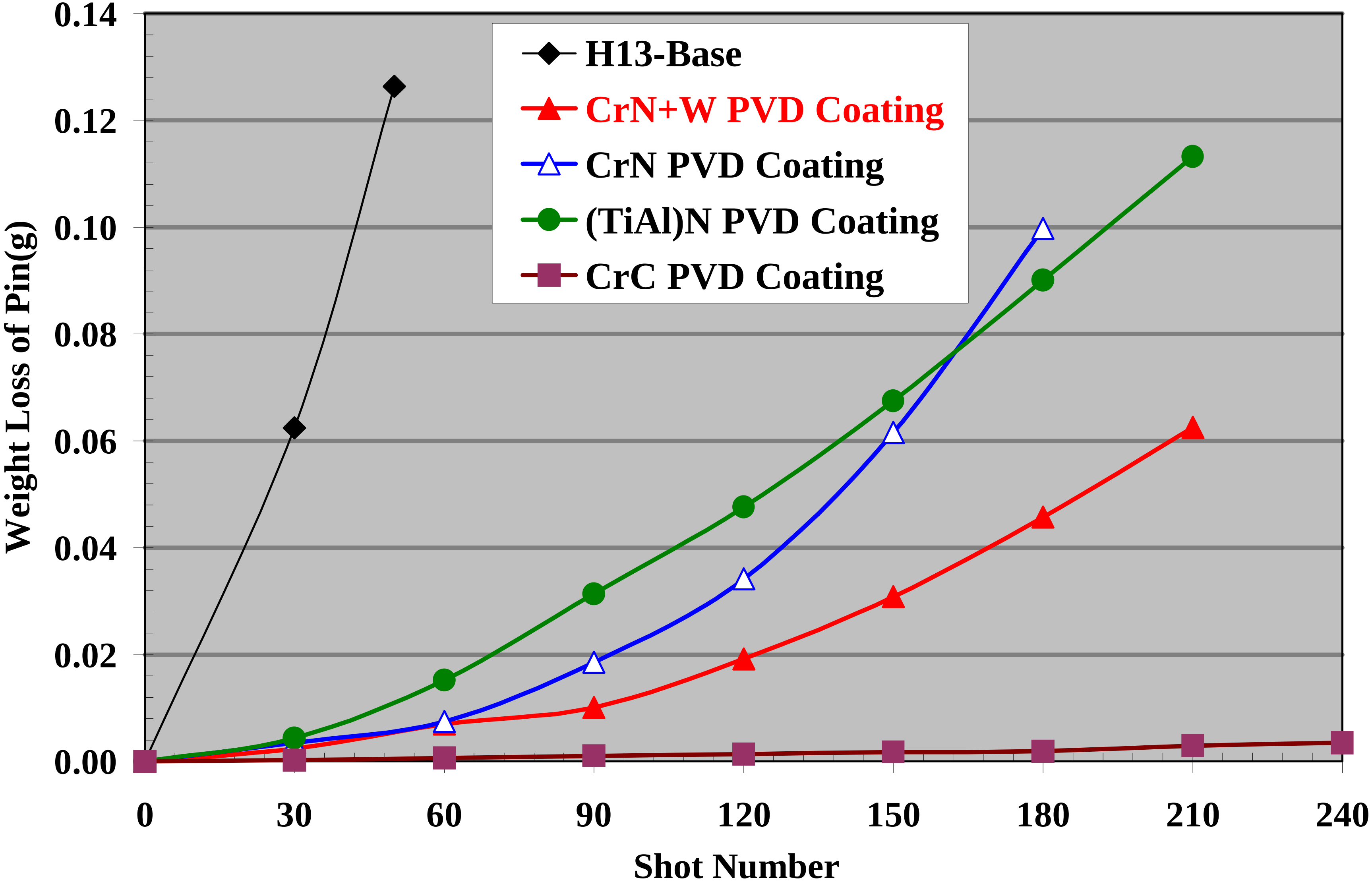
Effect of PVD Coating Materials on Soldering (after 30 shots)

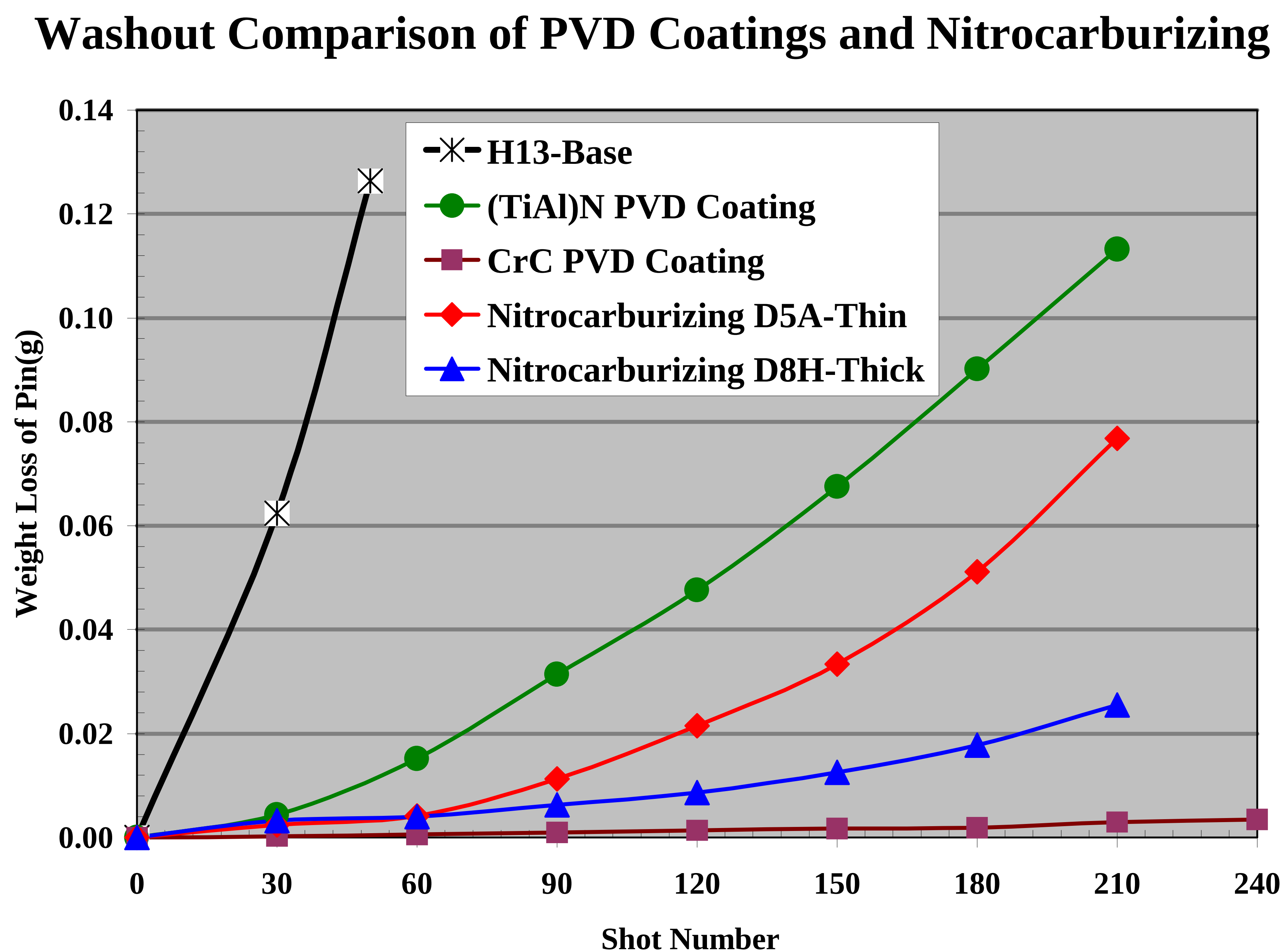


1.Weight of Soldered Al on Pin Surface Is Determined by Weighting Pins after 30 Shots with and without the Soldered Aluminum

2.Soldered Aluminum Cleaned with Sodium Hydroxide

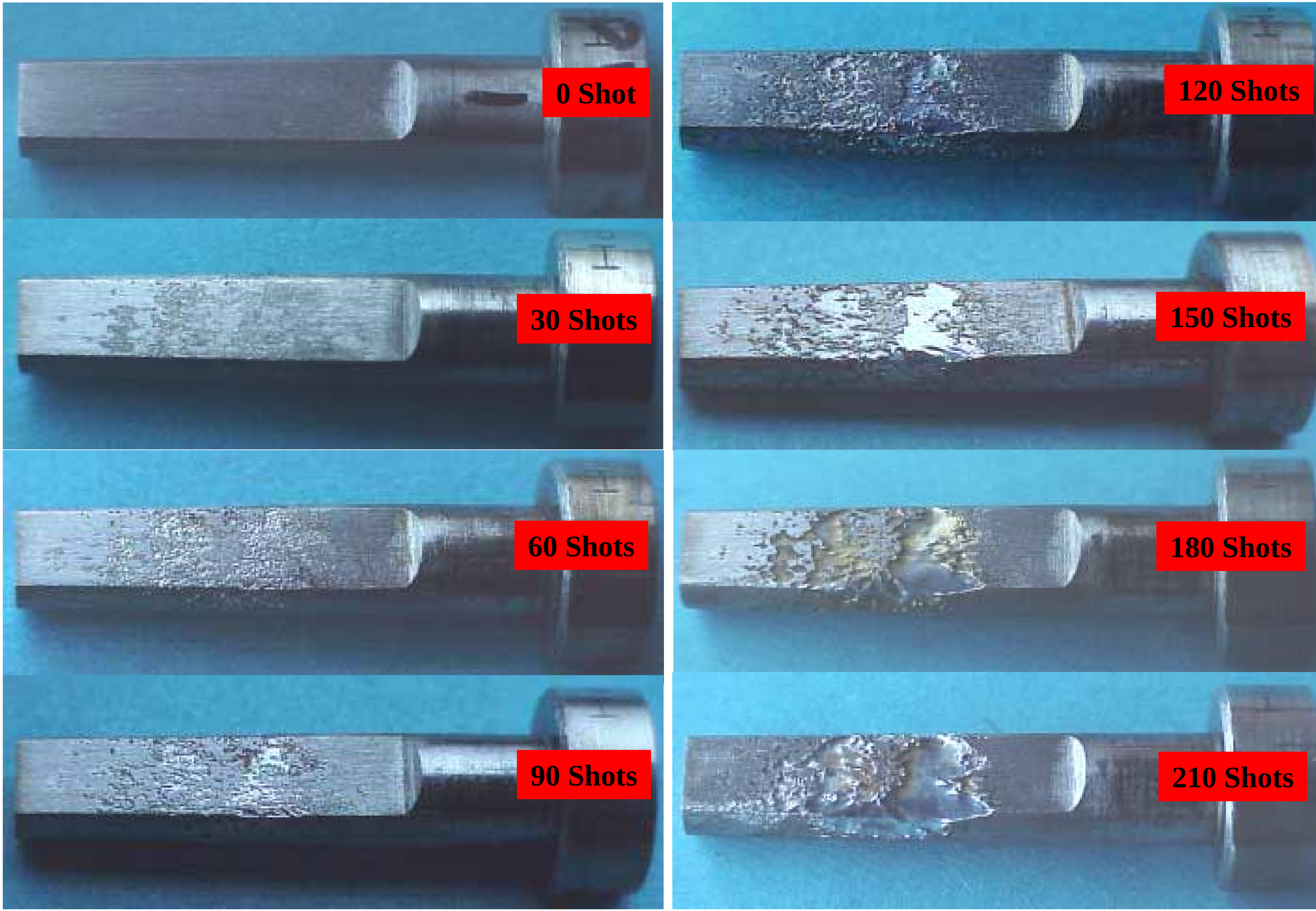
Effect of PVD Coatings on Washout Resistance



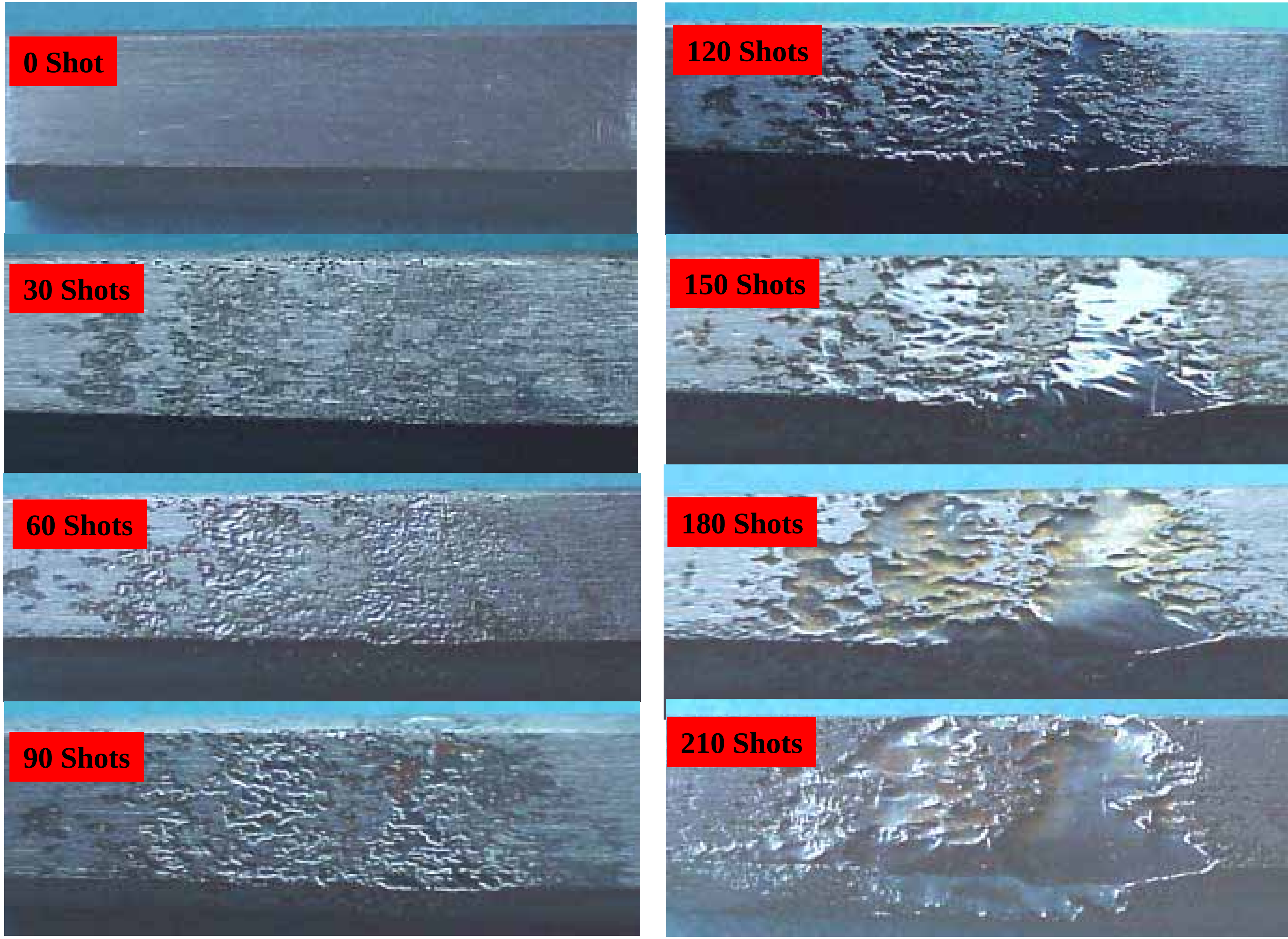


Washout Comparison of PVD Coatings and Nitrocarburizing
H13-Base
(TiAl)N PVD Coating
CrC PVD Coating
Nitrocarburizing D5A-Thin
Nitrocarburizing D8H-Thick
Weight Loss of Pin(g)
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
0
30
60
90
120
150
180
210
240
Shot Number

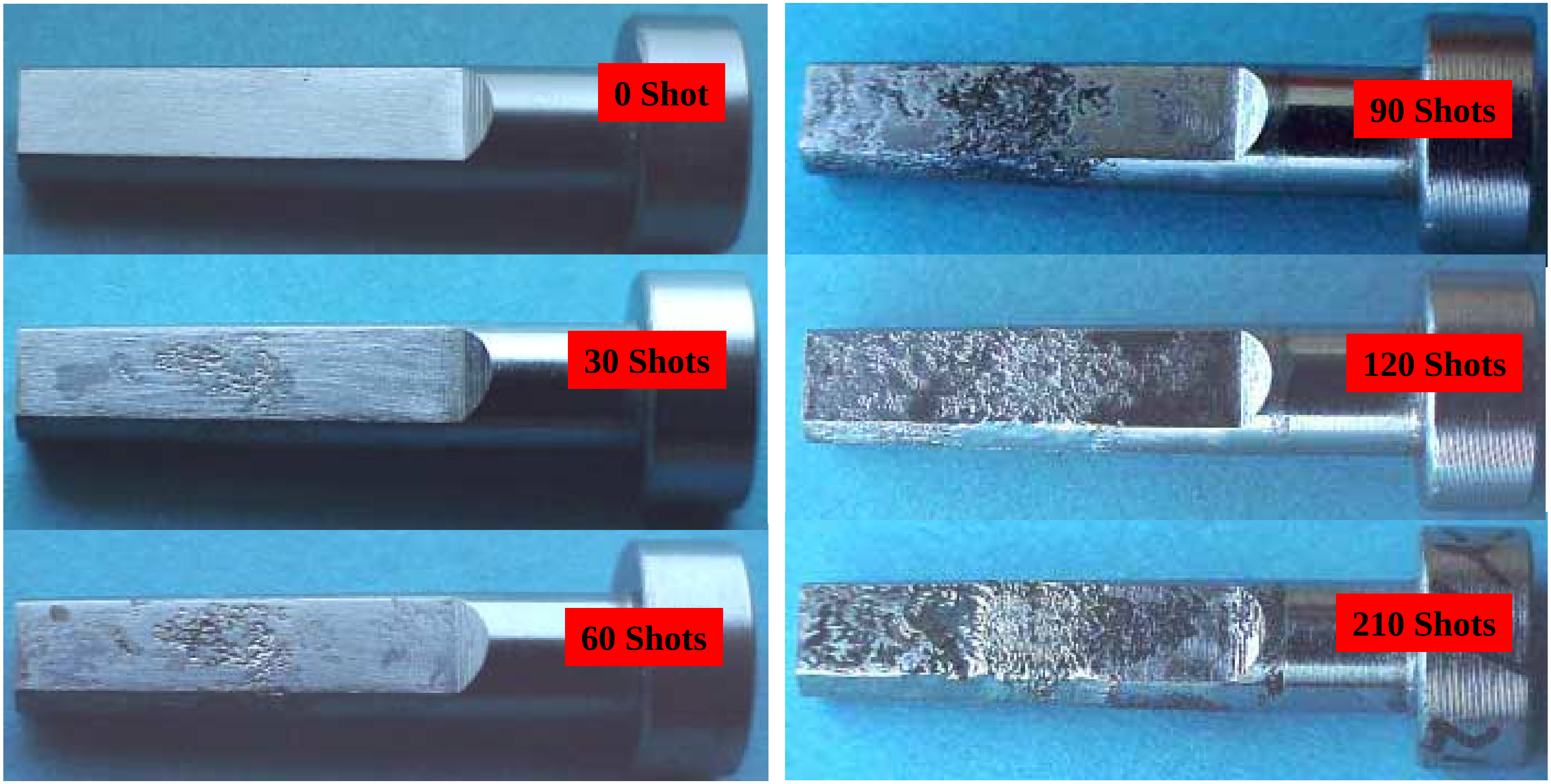
Degradation of H13 Pin with CrN+W PVD Coating



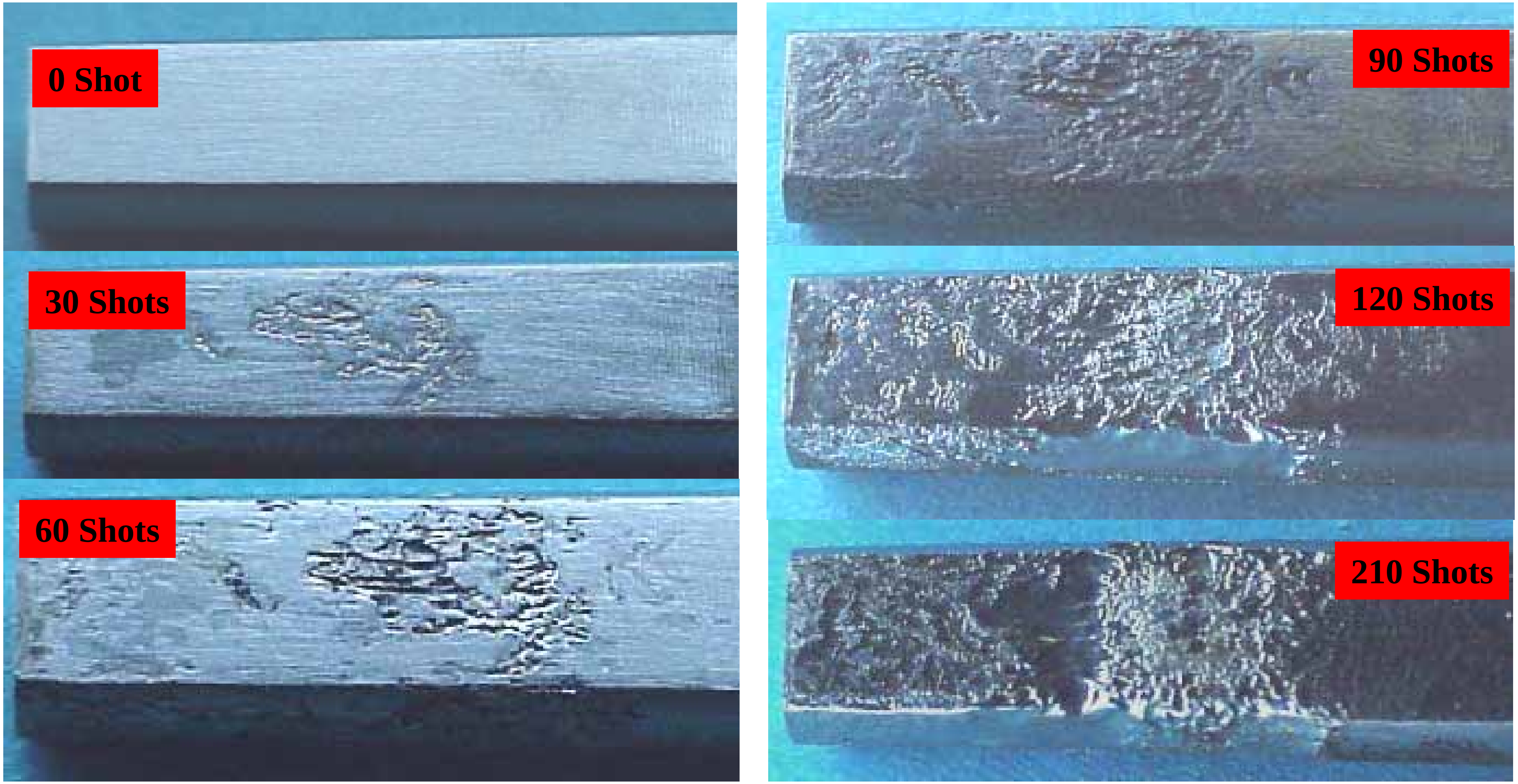
Degradation of H13 Pin with CrN+W PVD Coating (magnified)



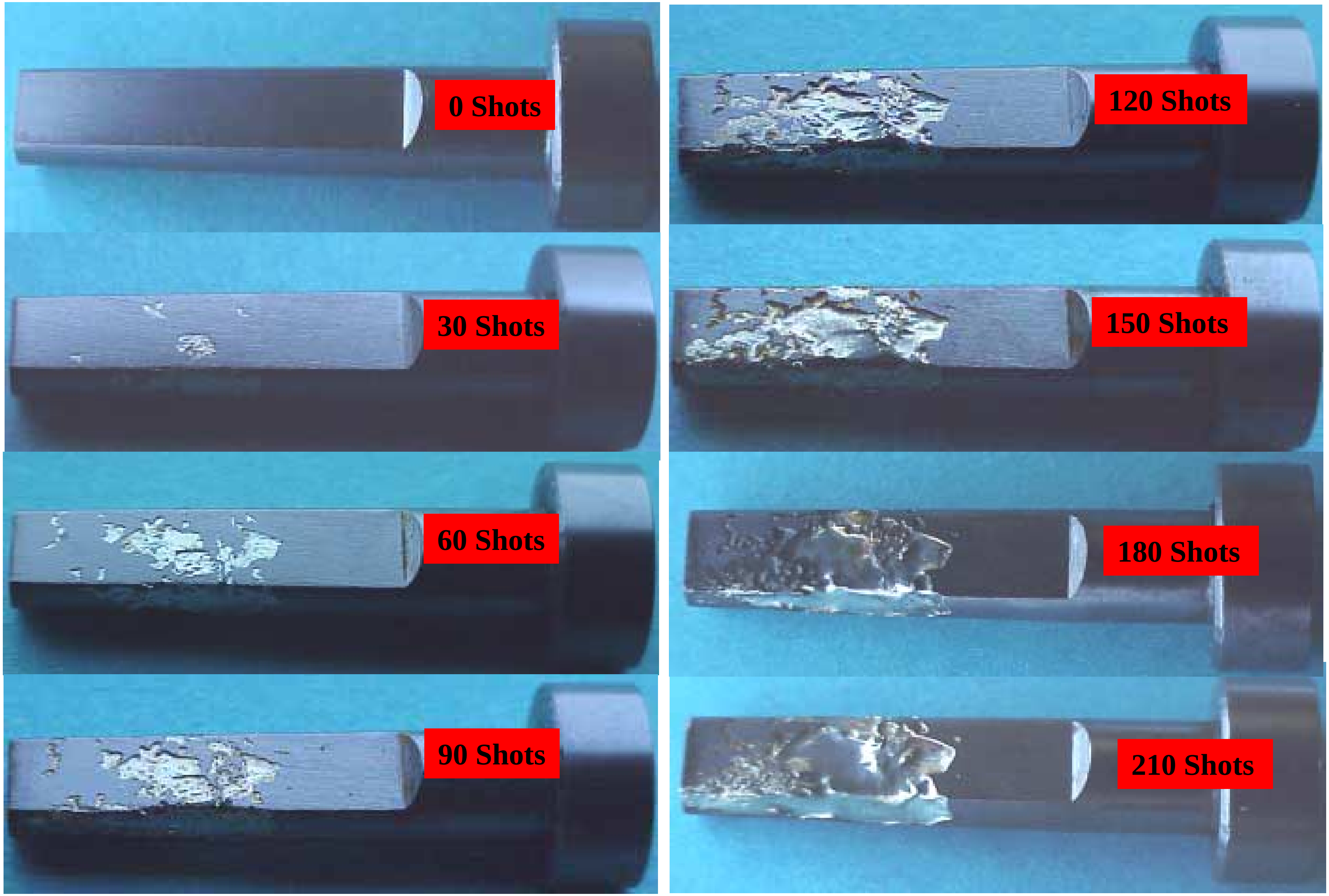
Degradation of H13 Pin with CrN PVD Coating



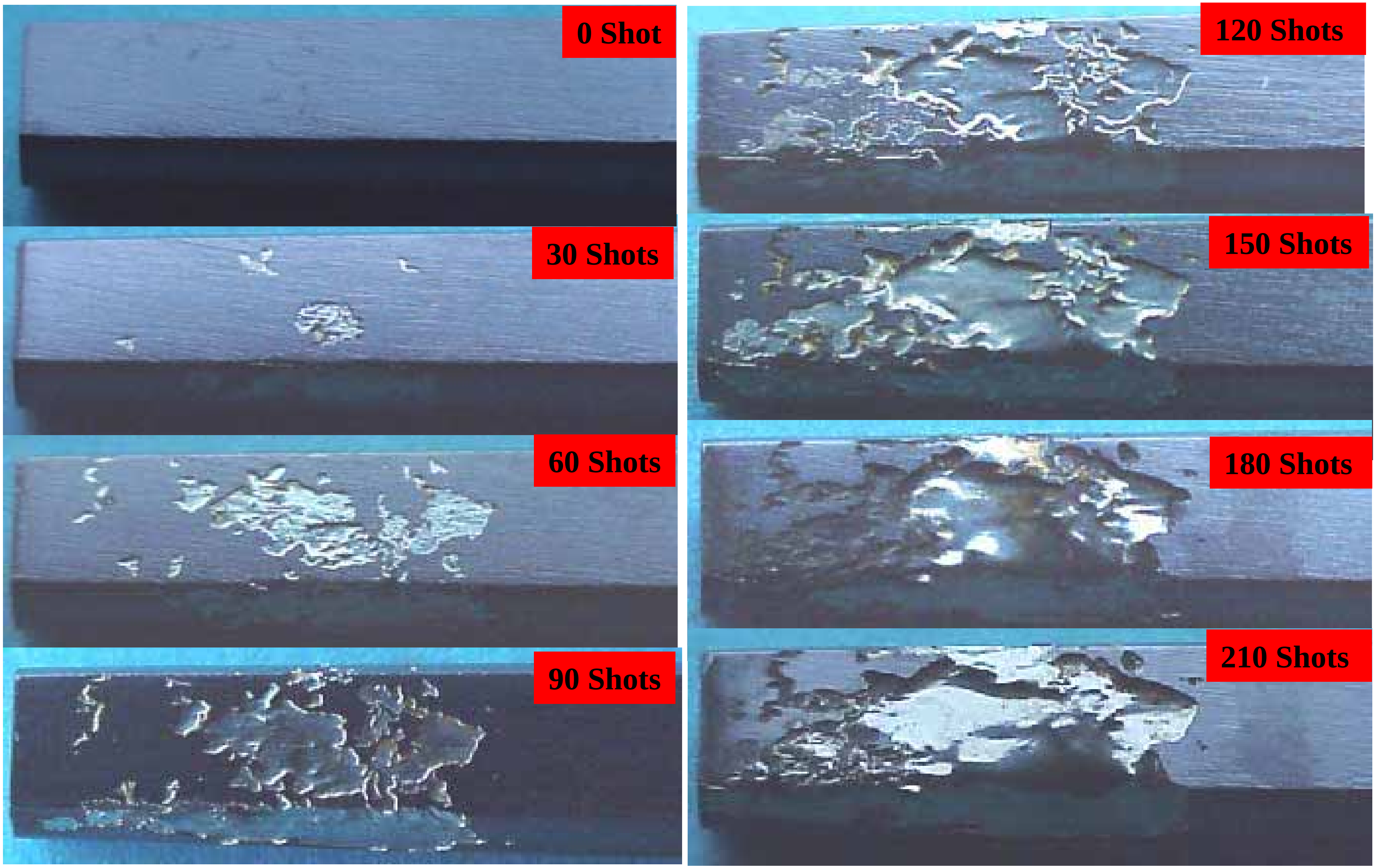
Degradation of H13 Pin with CrN PVD Coating (magnified)



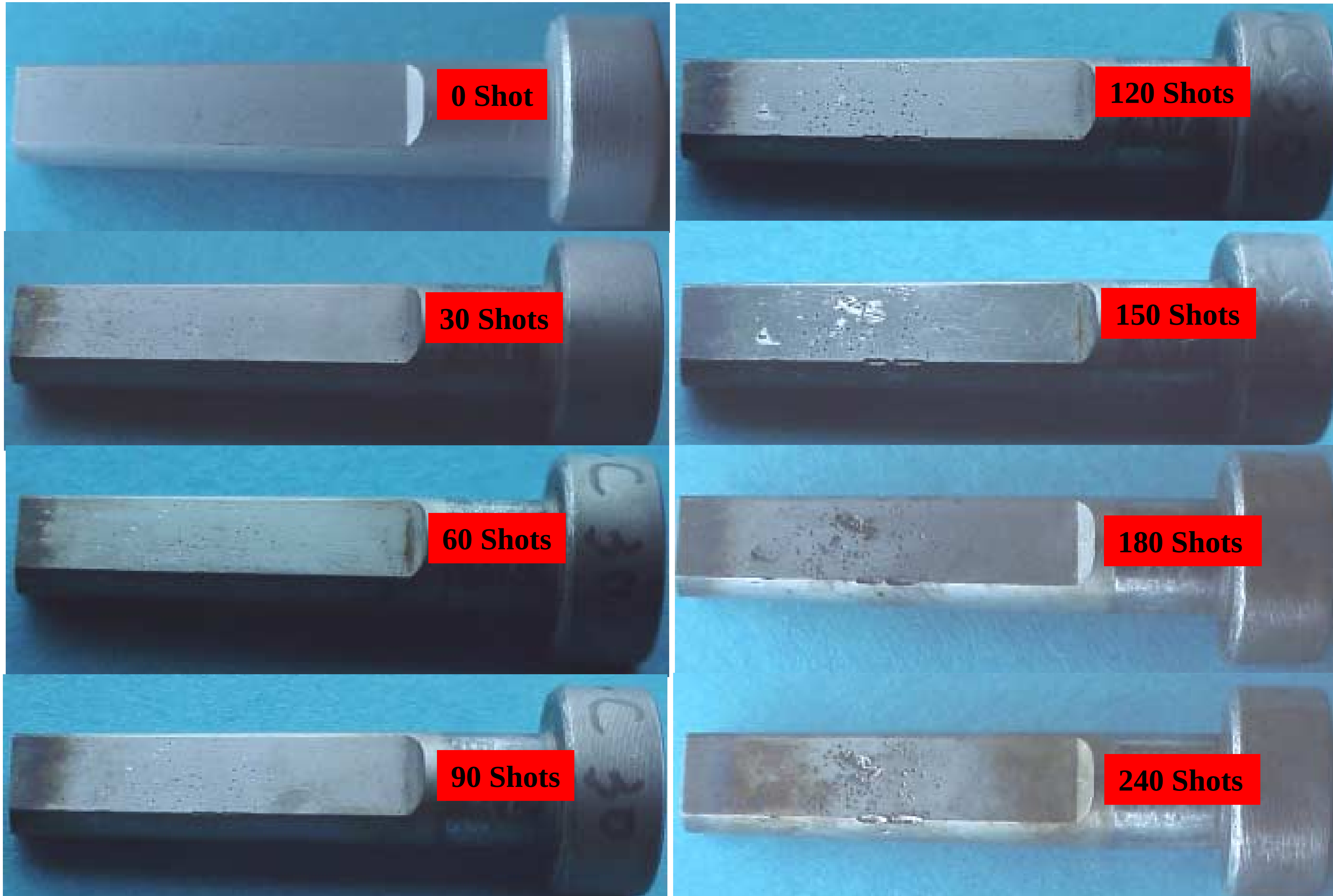
Degradation of H13 Pin with (TiAl)N PVD Coating



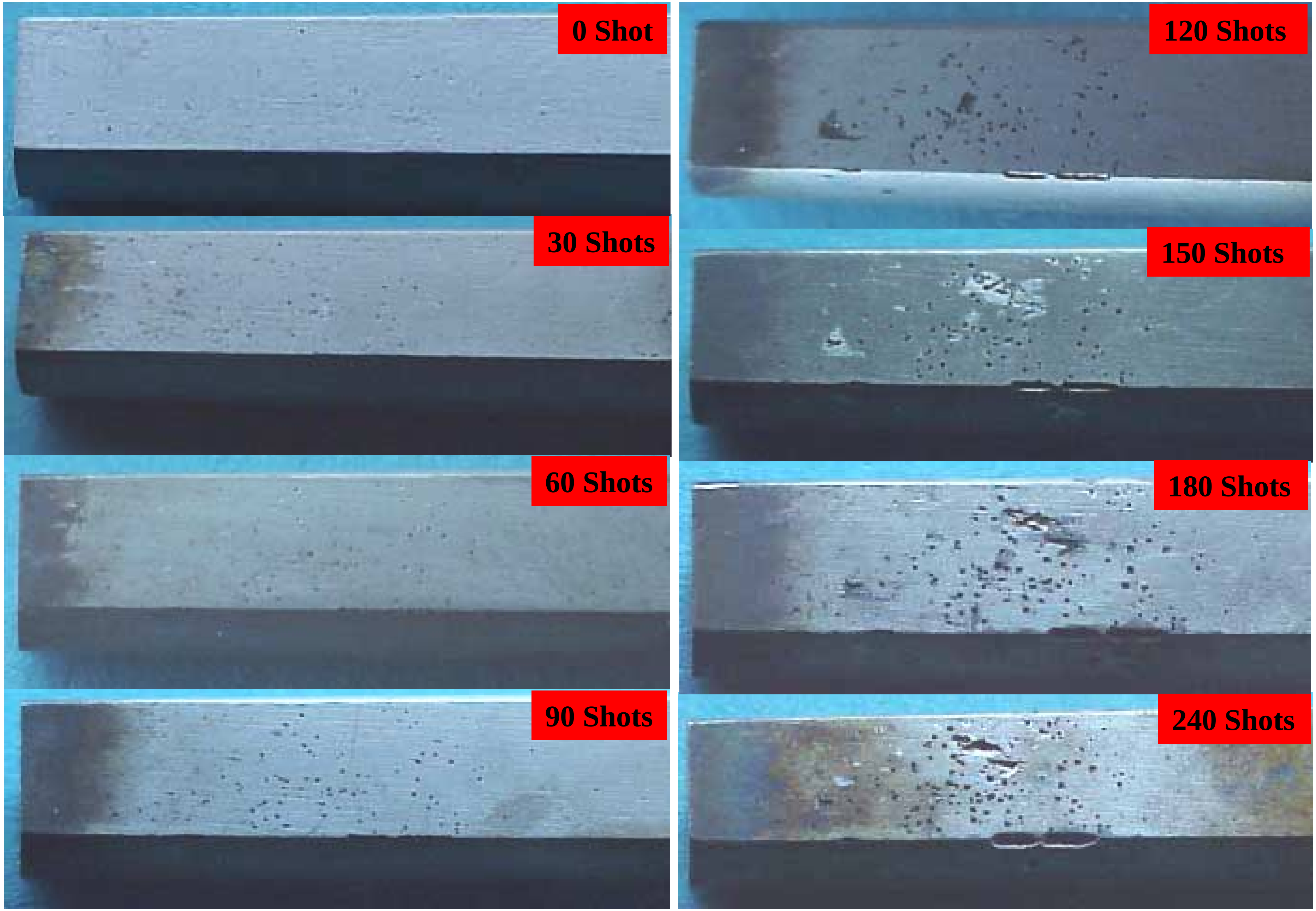
Degradation of H13 Pin with (TiAl)N PVD Coating (magnified)



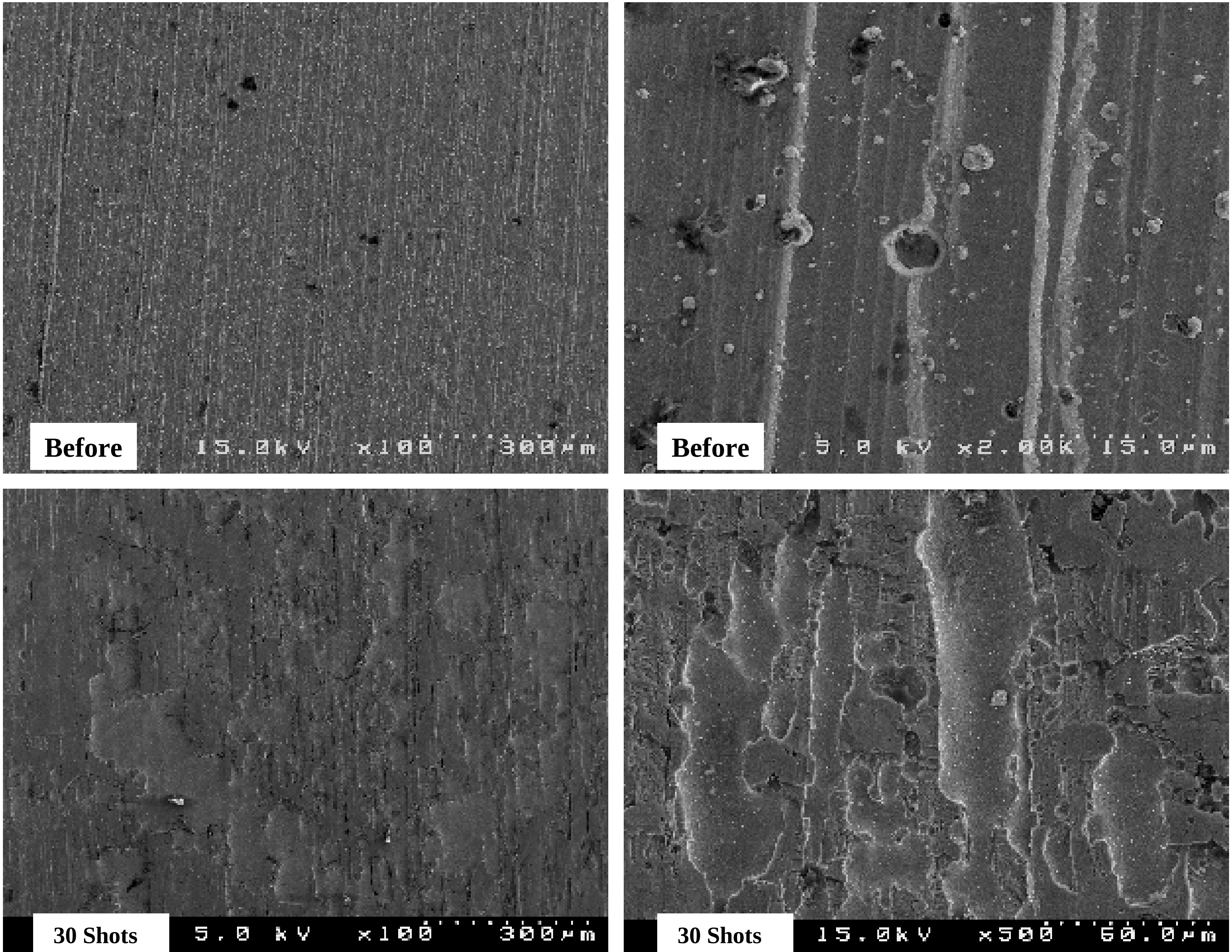
Degradation of H13 Pin with CrC PVD Coating



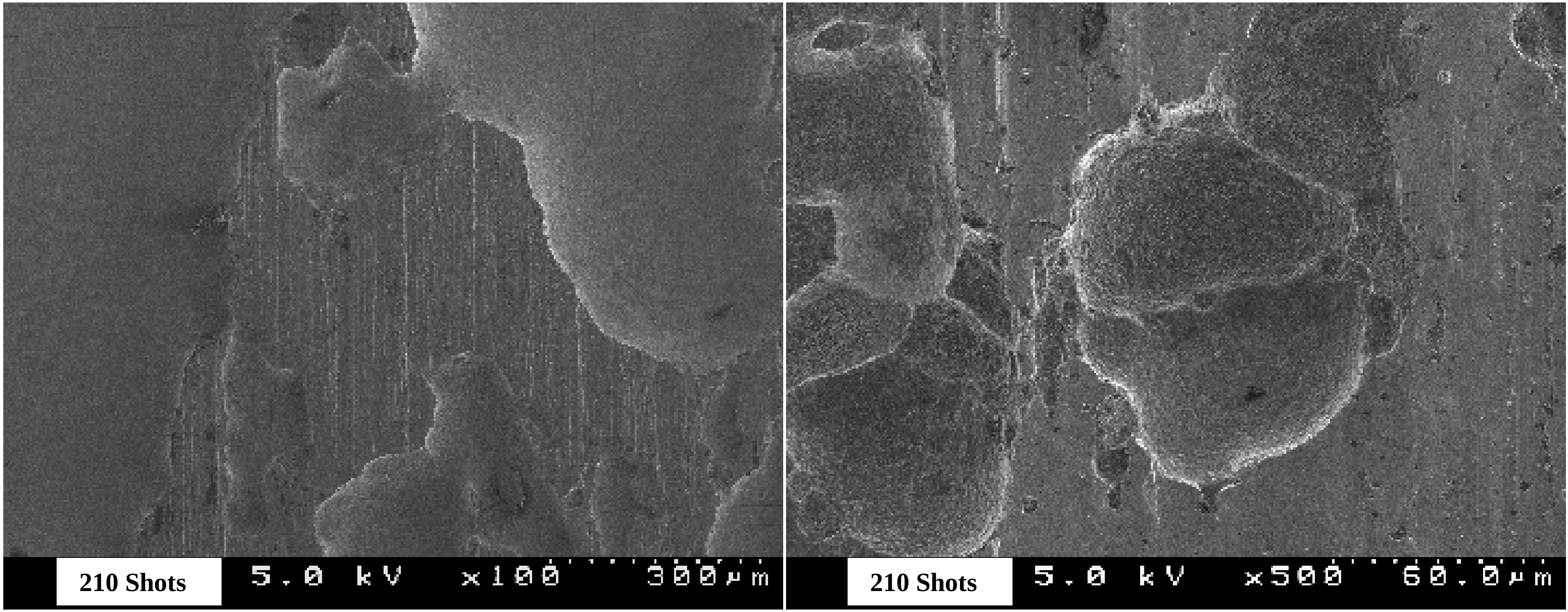
Degradation of H13 Pin with CrC PVD Coating (magnified)



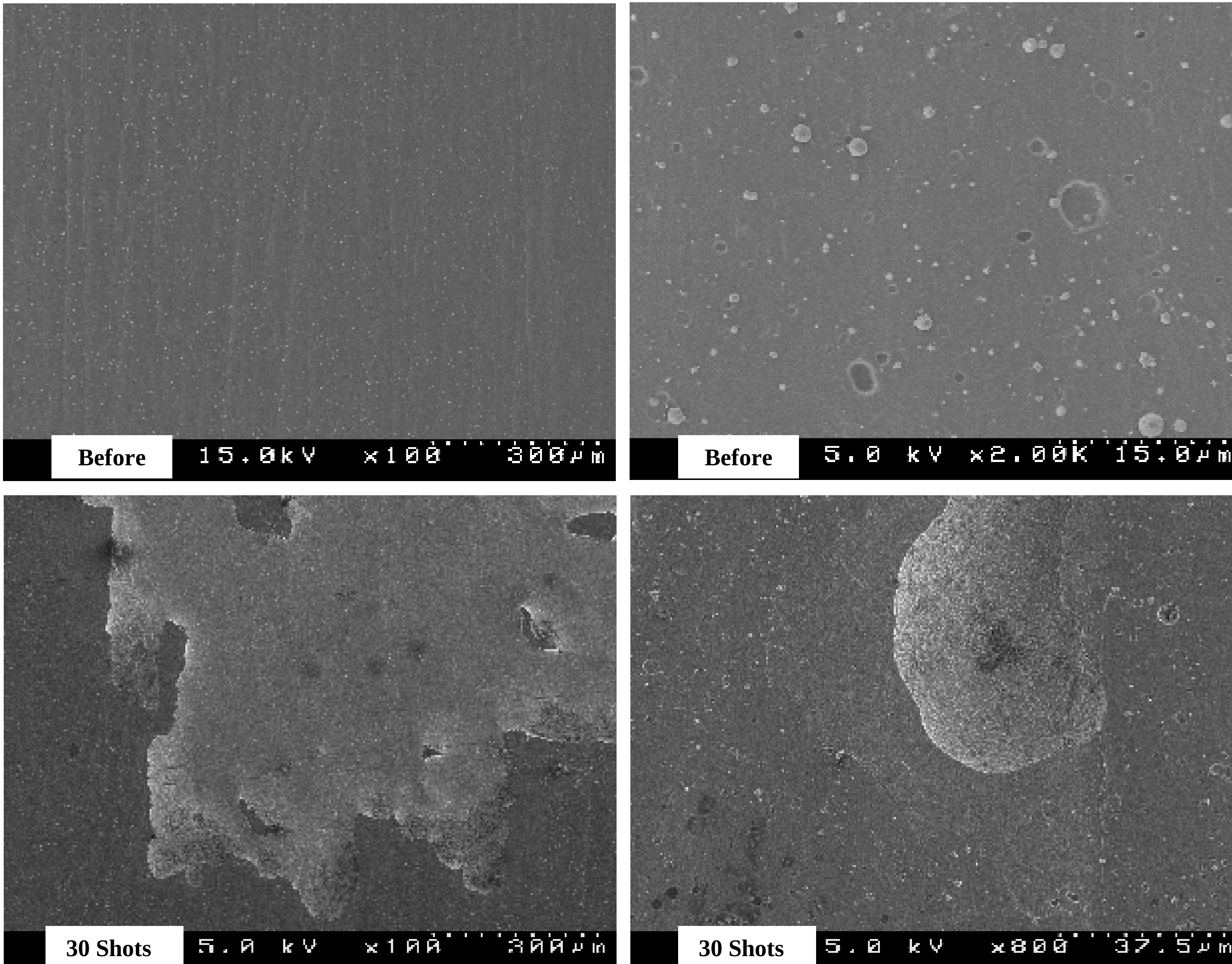
Degradation of H13 Pin with CrN+W PVD Coating (Pin Holes and Worn off)



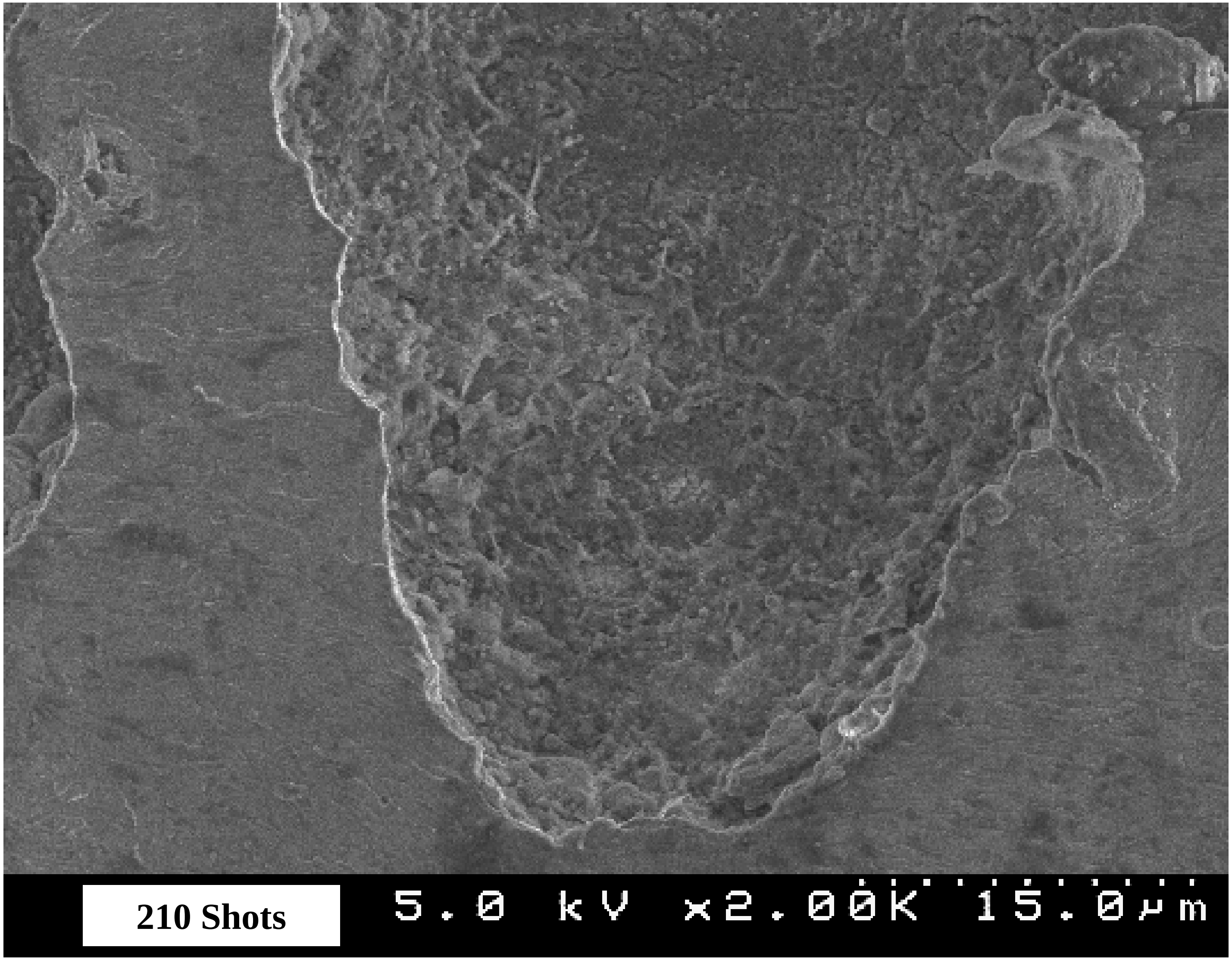
Degradation of H13 Pin with CrN+W PVD Coating after 210 Shots (Pin Holes and Worn off)



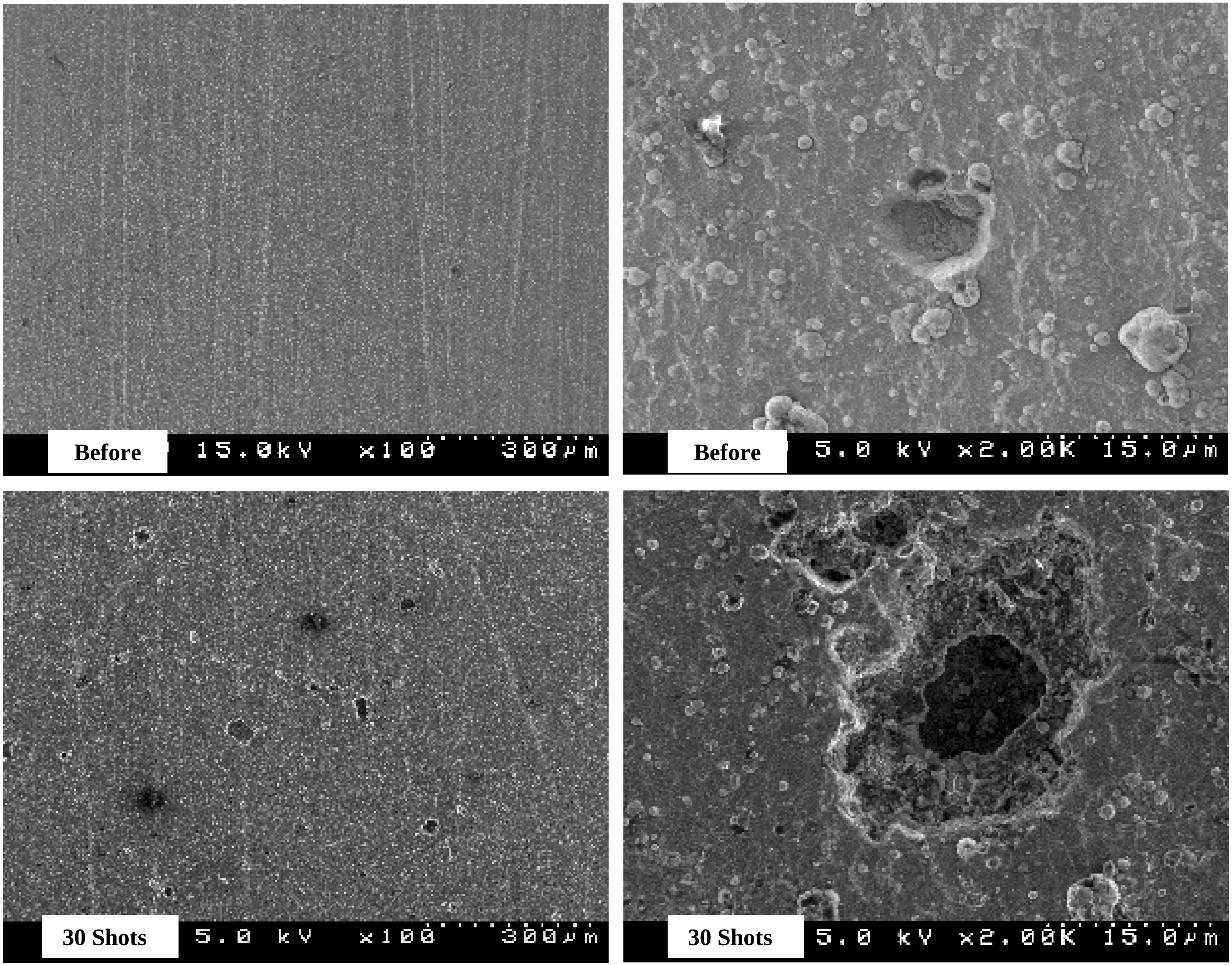
Degradation of H13 Pin with (TiAl)N PVD Coating (Pin Holes and Worn off)



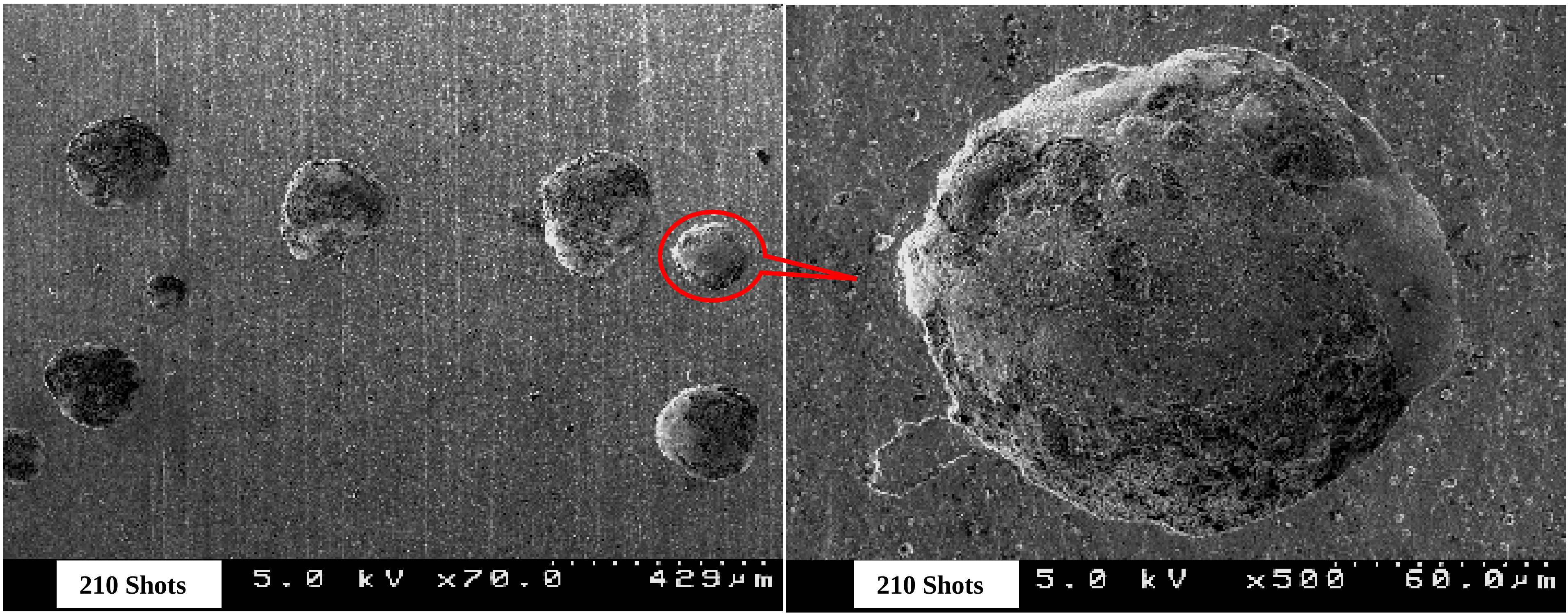
Degradation of H13 Pin with (TiAl)N PVD Coating after 210 Shots (Pin Holes and Worn off)



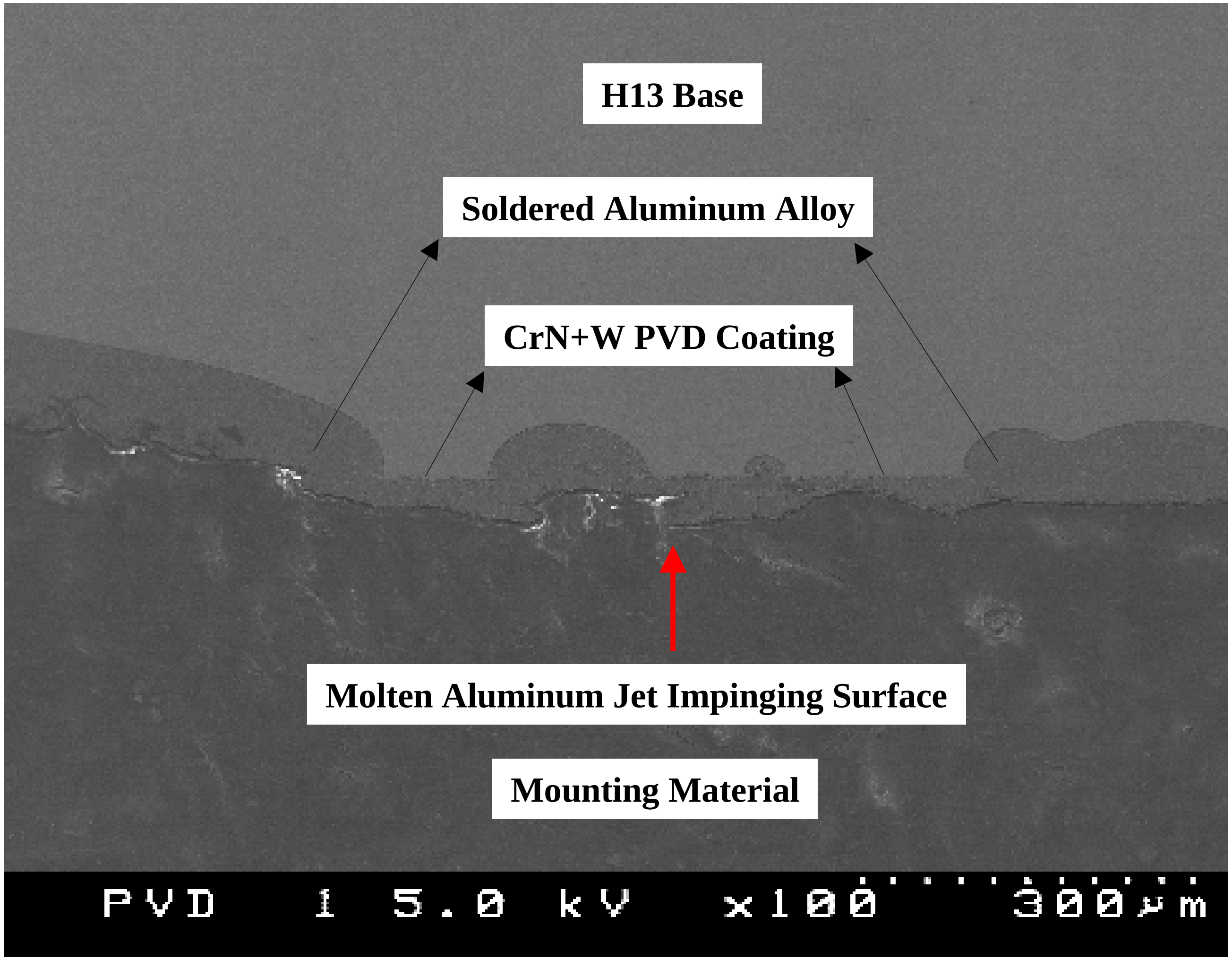
Degradation of H13 Pin with CrC PVD Coating(Only Pin Holes)



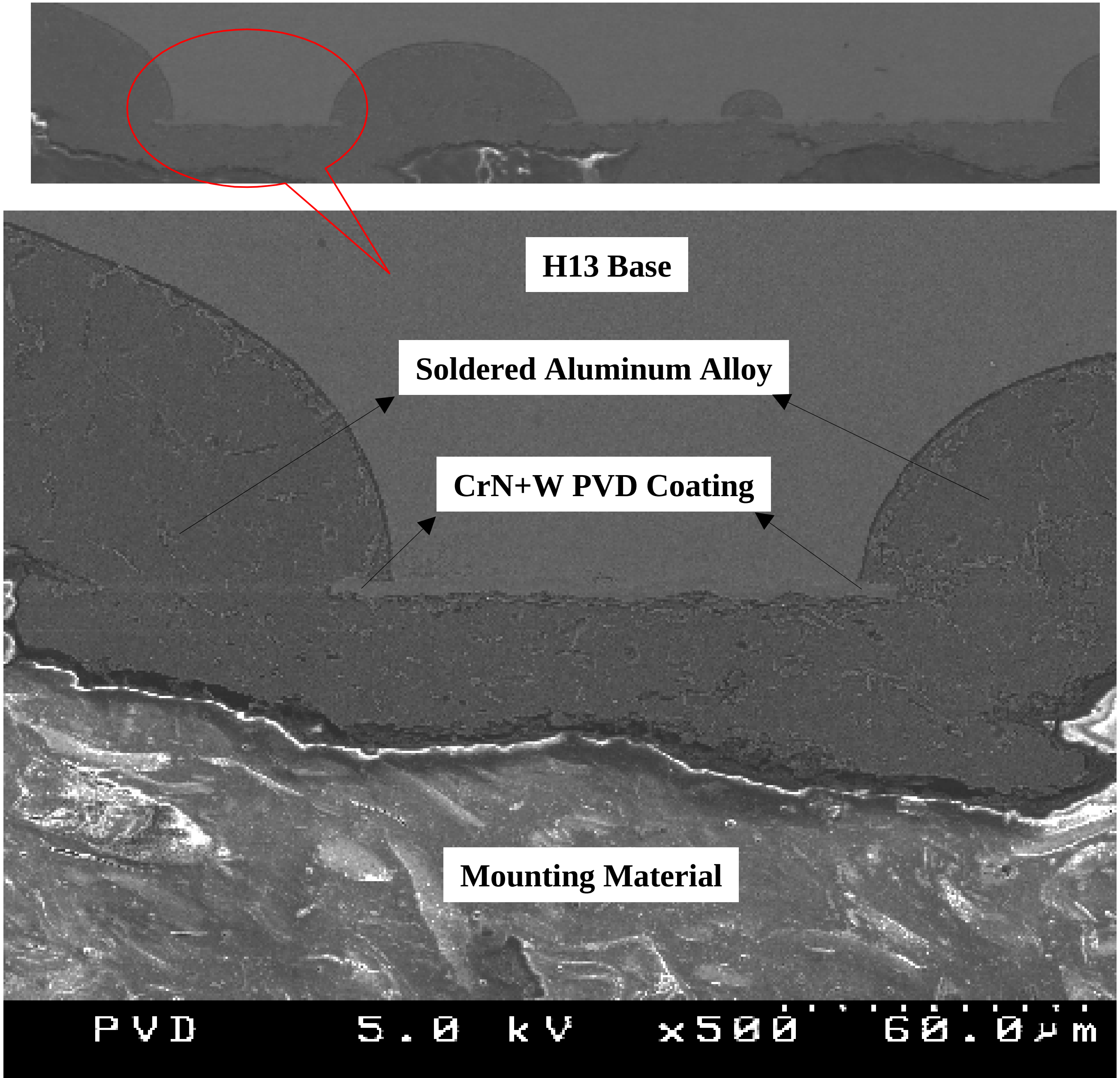
Degradation of H13 Pin with CrC PVD Coating after 240 Shots (Only Pin Holes)



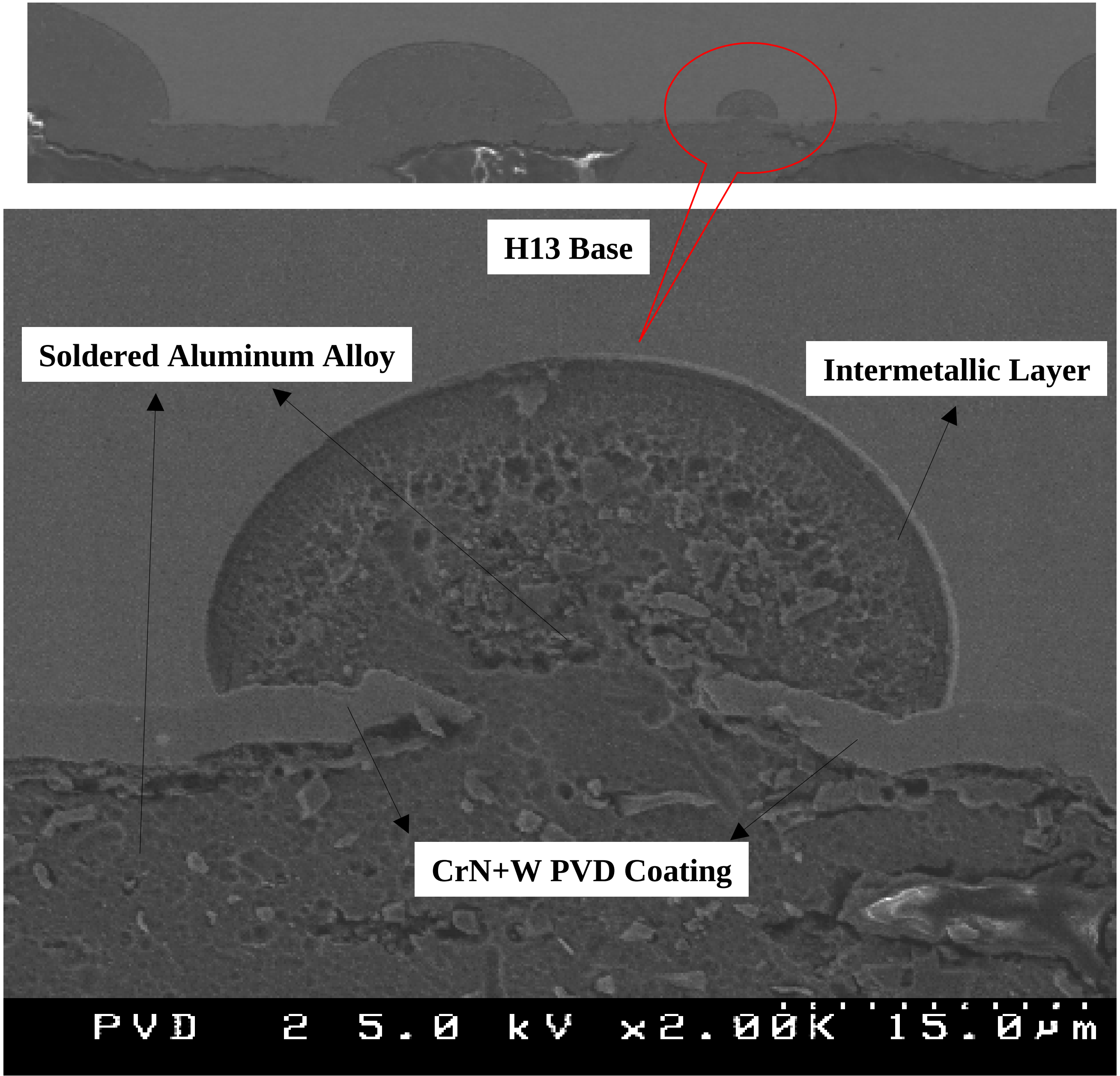
Cross Section View & Failure Mode of CrN+W PVD Coated H13 Pin after 120 Shots



Cross Section View & Failure Mode of CrN+W PVD Coated H13 Pin after 120 Shots



Cross Section View & Failure Mode of CrN+W PVD Coated H13 Pin after 120 Shots



CONCLUSIONS

- **The "thick" CrC PVD coating was the best performer among the PVD coatings evaluated so far.**
- **However, this coating is primarily recommended for small cores; It is more susceptible to thermal fatigue cracking than the thin coatings.**
- **The thin PVD coatings fail at surface imperfections in the substrate and at defect sites in the coating (pin-holes). Good coatings practices are essential in ensuring a high performance coating.**