MACQUARIE University

Microstructural Characterisation and Surface Examination of Coining Dies

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Introduction:

Coin dies are a highly specialized class of forming tools. In general, the requirements on PVD coating of forming tools are similar to the coatings designed for cutting tools. The quality and reliability of coin dies depend on the chosen material, the thermal treatment used during the production of the die and finalization and the treatment of its functional surfaces. The use of the dies during stamping affects its intrinsic material properties and a degradation occurs after some time. If they are changed in any way, the result will appear on the coins struck from them. Any abrasion marks from refinishing, impact contact, or pits from rust will go into its surface and leave a raised mark on the struck coin.

One common defect occurs when the obverse and reverse dies come together without a planchet between them. This causes part of each die's design to be impressed into the opposite die. The finished coin will exhibit unusual marks that do not belong on it (from the damage) that we call a die clash.

On occasion, a die will break or chip. This allows planchet metal to flow into the newly made void in the die, producing a raised defect on our coin. Chips and die breaks often occur at stressed and weak points in the die's design. Ruling out a massive, catastrophic failure, most die breaks start out small and progress. Thus, the understanding of crack development mechanisms in the die material is necessary to improve their durability. Developing a comprehensive understanding of crack initiation and propagation is therefore of great engineering importance.

Project aim:

The aim of characterization tests is to investigate the root cause of failure in dies. Specifically, to identify if the failure is beginning in the die steel OR is being propagated from the coating layer OR from the interface of the die steel and coating.

Experimental results:

Microstructural Characterisation and Surface Examination of Coining Dies

We have received 6 dies for this project, as follows:

1- D-9999DU026L
2- D-6356DU007K
3- D-2185DU062L
4- D-6172DC083L
5- D-9108DU015L
6- D-9108DU011L

To minimised the potential destruction on the received dies, two dies were selected to conduct the initial tests.

Scanning electron microscopy analysis:

Scanning electron microscopy is a powerful technique applied in microimaging of surface. This technique can be used to explore the surface structure and morphology of the samples. Here, surface morphology of the samples was investigated using PHENOM XL Benchtop SEM at a voltage of 15 KV. **Note:** <u>No sample preparation process was conducted on the samples, as they have been used as received.</u>

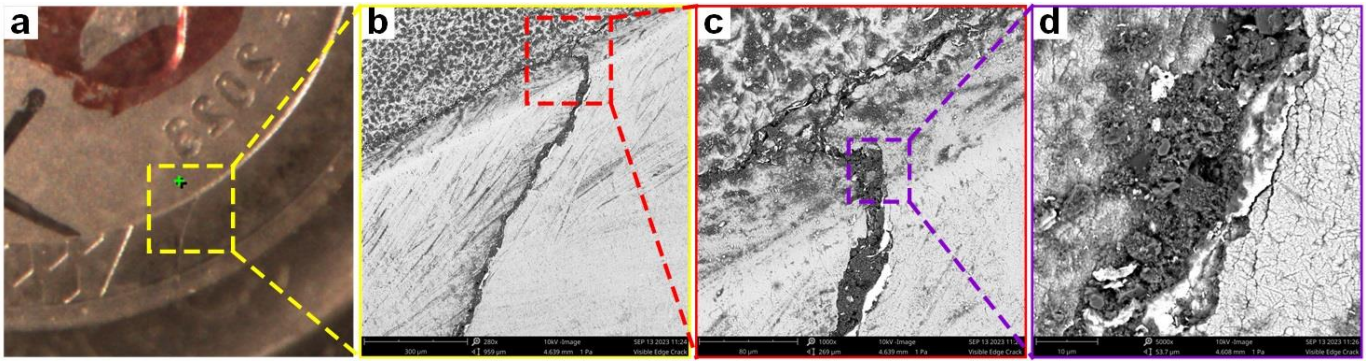


Figure 1. SEM images of sample 1 (#D-9108DU015L) at different magnification (low to high).

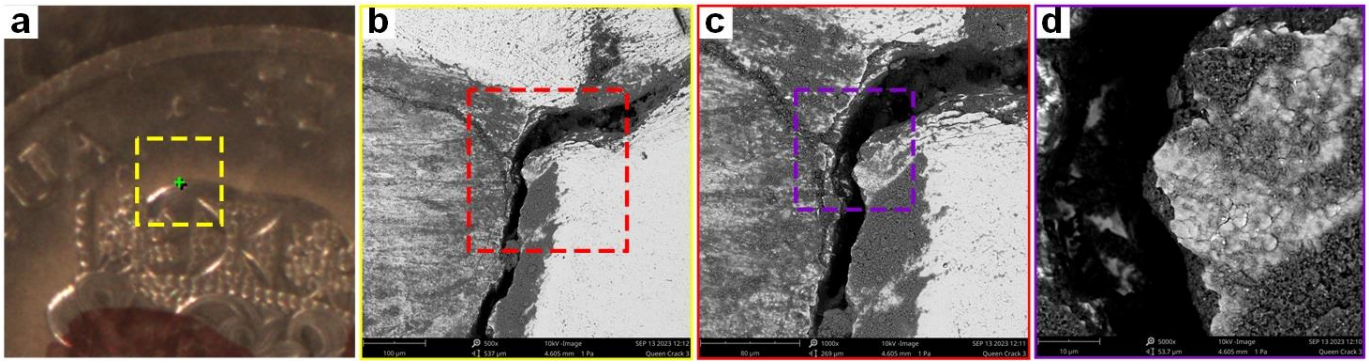


Figure 2. SEM images of sample 2 (#D-9999DU026L) at different magnification (low to high).

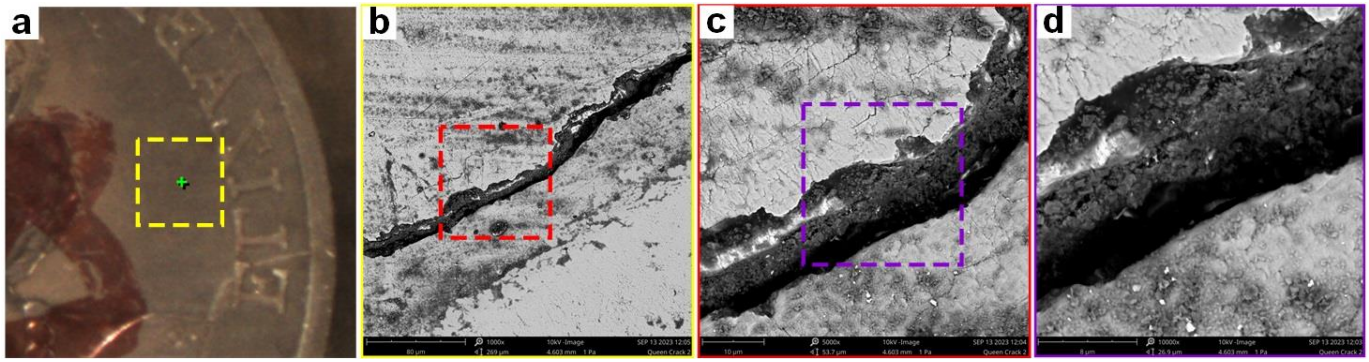


Figure 3. SEM images of sample 2 (#D-9999DU026L) at different magnification (low to high).

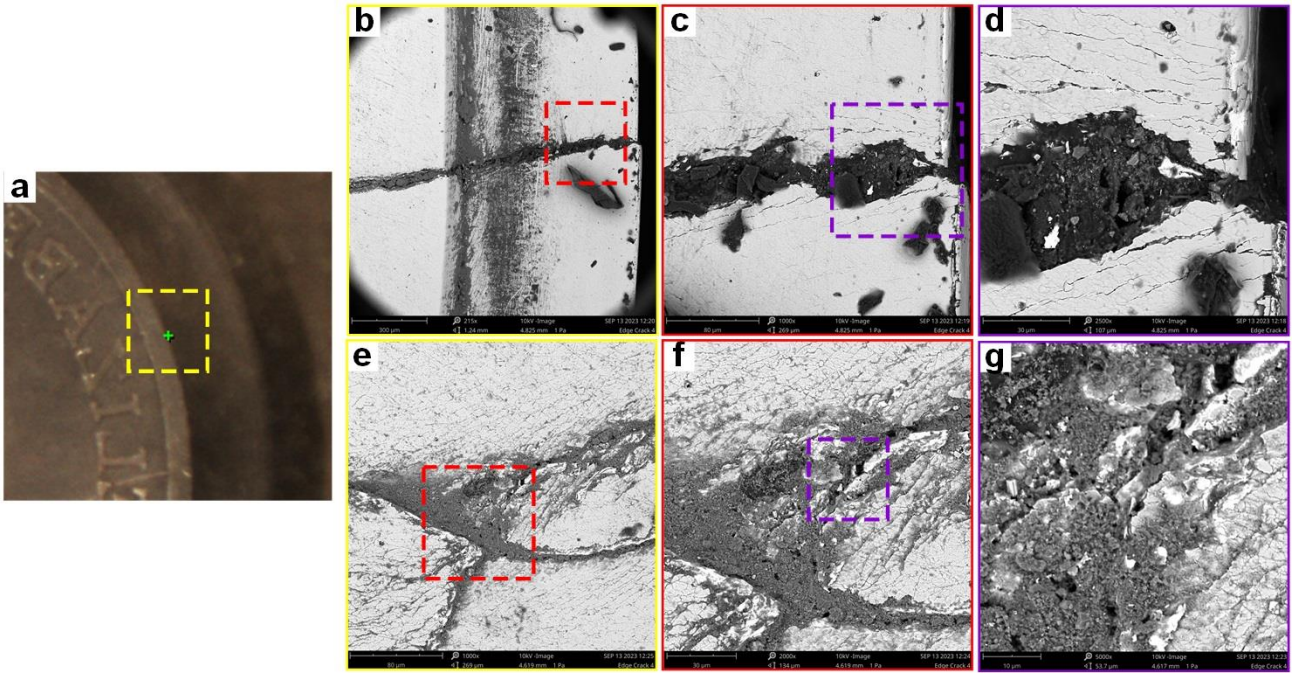


Figure 4. SEM images of sample 2 (#D-9999DU026L) at different magnification (low to high).

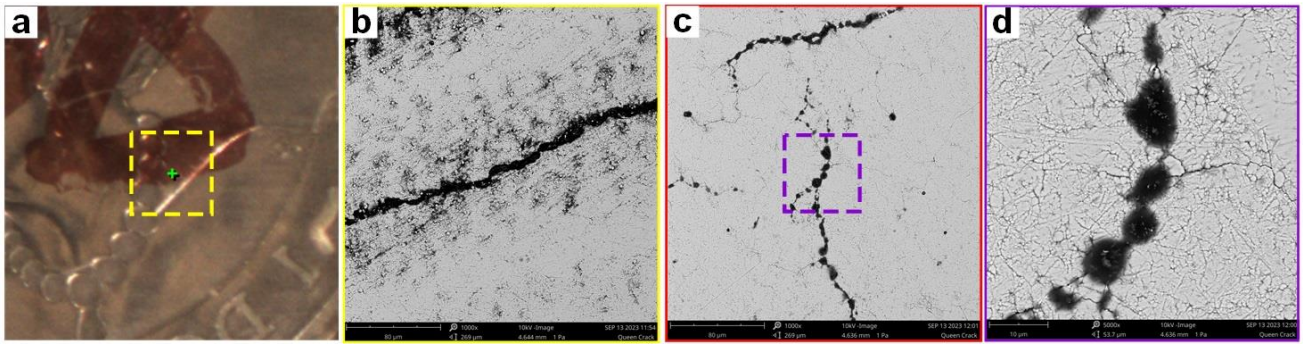


Figure 5. SEM images of sample 2 (#D-9999DU026L) at different magnification (low to high).

Etching process:

Etching is a common practice in metallurgy to reveal the microstructure of metal samples. Microstructure refers to the arrangement of the grains and phases within the material. It creates contrast variations on the surface of the material, making it easier to distinguish different phases, grains, and other microstructural features under a microscope. This enhanced contrast helps in identifying various constituents and defects. Etching makes grain boundaries more visible. The grain boundaries are the interfaces between individual crystals (grains) in the steel. Examining these boundaries can provide information about the quality of the steel, such as its grain size and the presence of any abnormalities.

Here, we used 2% nitol as an etchant to etch the dies to study their microstructure. After applying the etchant to the substrate, the material underwent a controlled corrosion process to achieve the

desired pattern. To regulate the etching time, the substrate was dipped in water after intervals of 5, 10, 15, and 20 seconds. Through this systematic approach, different exposure times were tested to determine the optimal duration for achieving the finest results. In this specific case, it was found that the 10-second interval yielded the best outcome. Consequently, this precise timing was adopted as the standard for imaging, ensuring consistent and high-quality results in the etching process.

SEM analysis of etched sample:

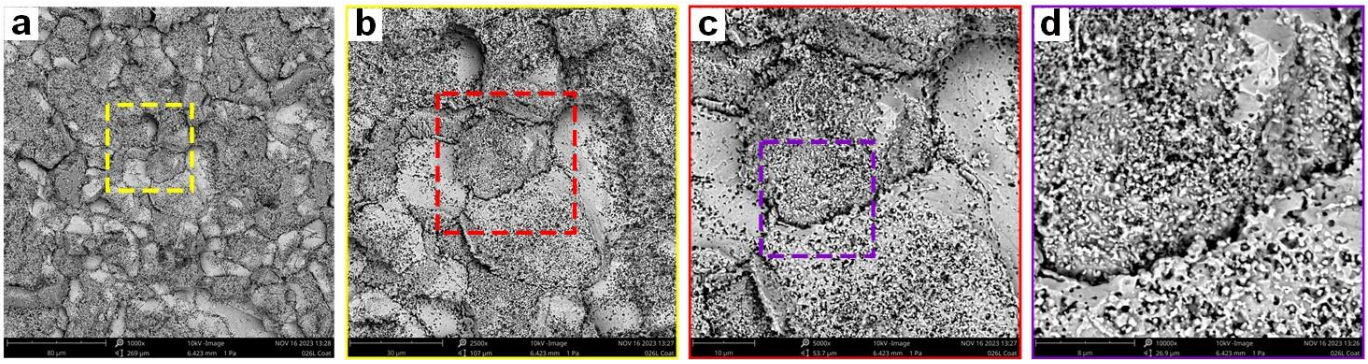


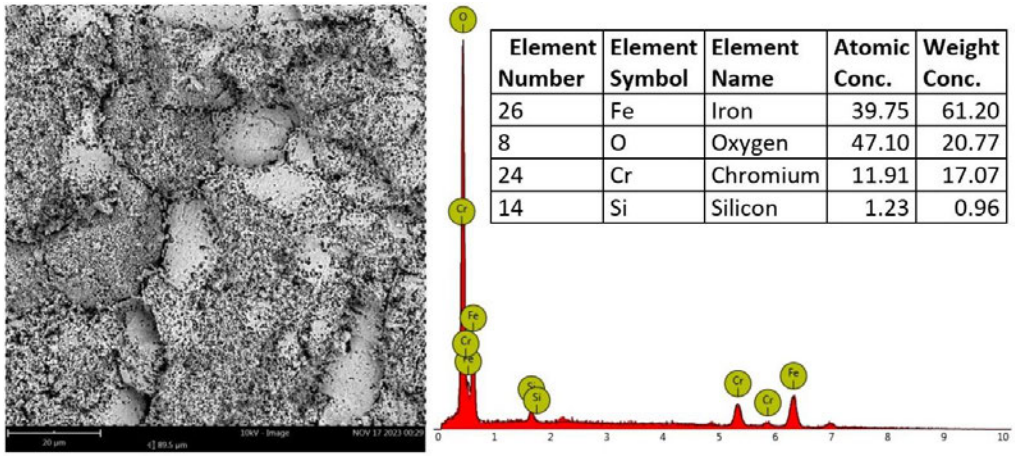
Figure 6. SEM images of etched sample 2 (#D-9999DU026L) at different magnification (low to high).

Energy Dispersive X-ray (EDX) analysis:

EDX, also known as Energy Dispersive X-ray Spectroscopy (EDS) is a technique used to analyse the elemental composition of a material. It is commonly employed in various scientific and industrial fields.

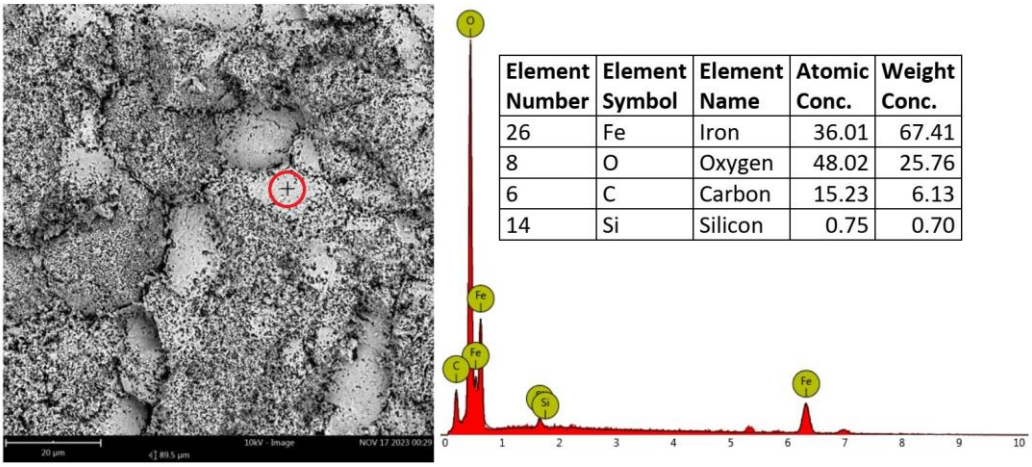
EDX analysis of etched sample:

EDX (point and region) of the samples were performed using PHENOM XL Benchtop SEM to analyse the material compositions in each sample.



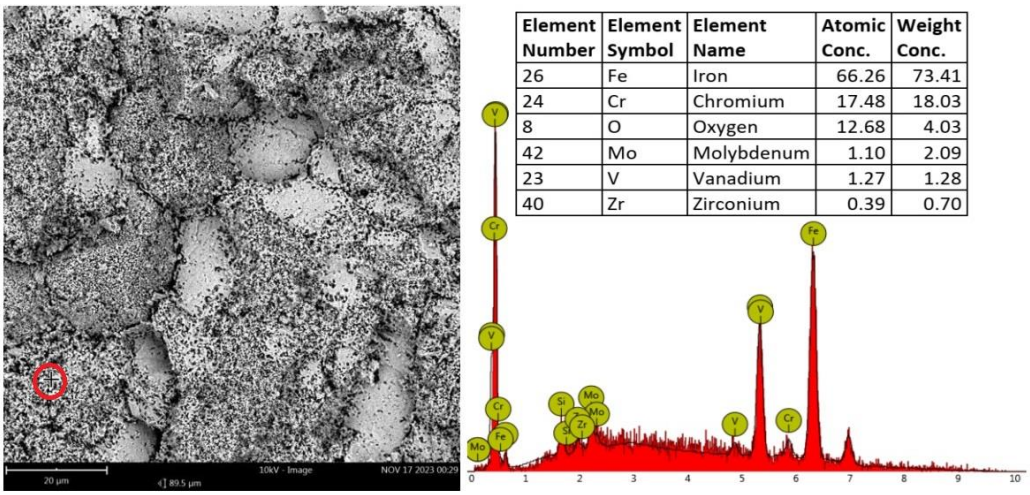
Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
26	Fe	Iron	39.75	61.20
8	O	Oxygen	47.10	20.77
24	Cr	Chromium	11.91	17.07
14	Si	Silicon	1.23	0.96

Figure 7. EDX analysis of selected region in sample 2 (#D-9999DU026L)



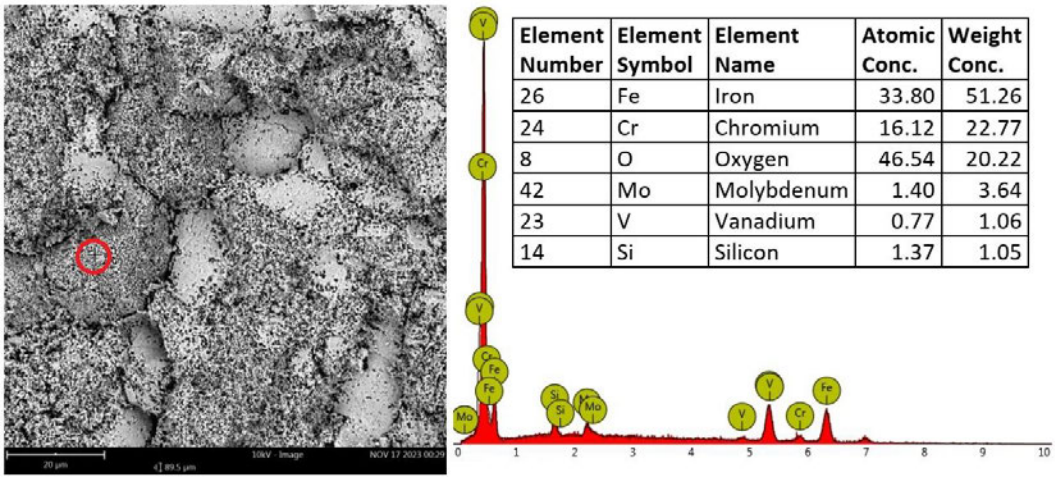
Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
26	Fe	Iron	36.01	67.41
8	O	Oxygen	48.02	25.76
6	C	Carbon	15.23	6.13
14	Si	Silicon	0.75	0.70

Figure 8. EDX analysis of selected point (highlighted in red circle) in sample 2 (#D-9999DU026L). This point is selected from the uncoated area of the sample.



Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
26	Fe	Iron	66.26	73.41
24	Cr	Chromium	17.48	18.03
8	O	Oxygen	12.68	4.03
42	Mo	Molybdenum	1.10	2.09
23	V	Vanadium	1.27	1.28
40	Zr	Zirconium	0.39	0.70

Figure 9. EDX analysis of selected point (highlighted in red circle) in sample 2 (#D-9999DU026L). This point is selected from the coated area of the sample.



Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
26	Fe	Iron	33.80	51.26
24	Cr	Chromium	16.12	22.77
8	O	Oxygen	46.54	20.22
42	Mo	Molybdenum	1.40	3.64
23	V	Vanadium	0.77	1.06
14	Si	Silicon	1.37	1.05

Figure 10. EDX analysis of selected point (highlighted in red circle) in sample 2 (#D-9999DU026L). This point is selected from the coated area of the sample.

Note: Carbon concentration determination in EDX can be challenging and is often not as accurate as other elements. The low sensitivity of the EDX technique to light elements like carbon makes it inherently less accurate in quantifying their concentrations. Additionally, the presence of other elements in the sample matrix, such as oxygen and nitrogen, can interfere with the carbon signal, leading to inaccuracies. Furthermore, the complex nature of carbon compounds and their variable stoichiometry can introduce uncertainties in the calibration process. Despite these challenges, EDX remains a valuable tool for elemental analysis, but users should be aware of its limitations when assessing carbon concentrations.

Samples preparation process for hardness testing:

The samples were cut to size using a Struers Secotom-50 Precision cutting machine. The cut-off wheel was 10S20, turning at 2200rpm at a speed between 1 and 2mm/s (depending on the height and thickness of the sample). The samples were then mounted using a Struers CitoPress-30 Mounting Press and PolyFast medium for hardness analysis. Prior to hardness testing, the samples were ground using 1200 grit SiC foil paper and then polished with a 6-micron diamond abrasive and oxide polishing alumina based Mol-B3.

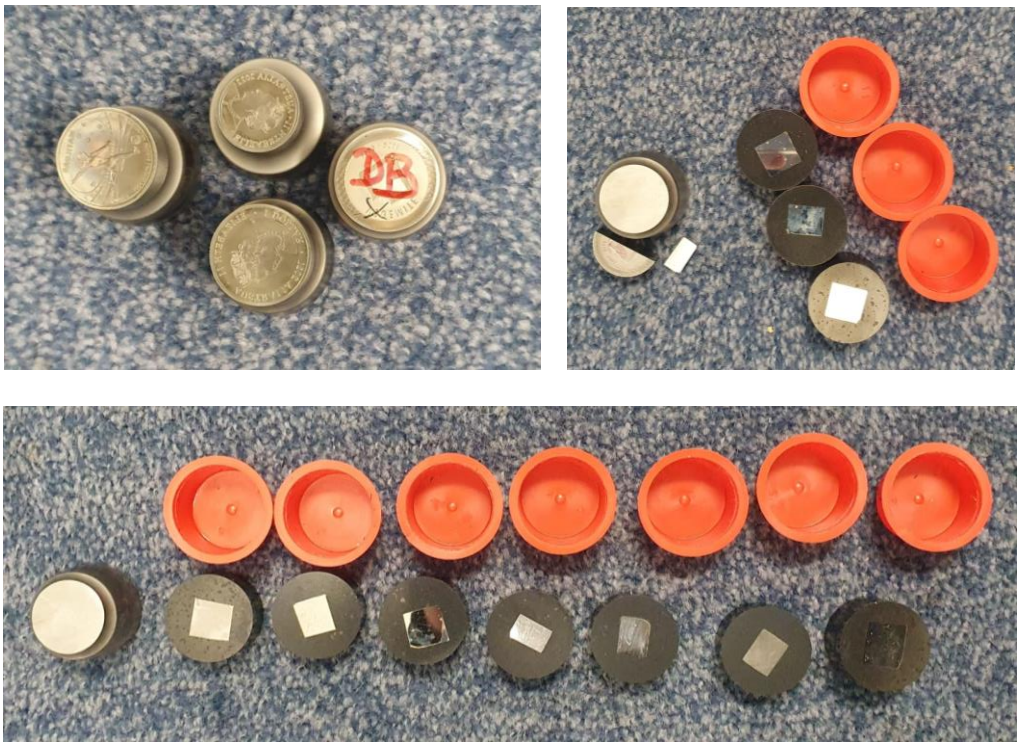


Figure 11. Images taken from all dies at the end of the project.
Dies #D-6356DU007K, #D-2185DU062L, #D-6172DC083L and #D-9108DU011L (top left), Die #D-9108DU015L (top right), Die #D-9999DU026L (bottom)

Hardness test:

A Struers DuraScan Vickers hardness tester was used to measure the microhardness of each polished sample. The diamond indenter was set to 0.5-3HV of force with a 10 s dwell time used to take every measurement. 10 indentations were made for each sample at randomly selected areas to increase the accuracy of the results. The indents were made at least six (6) diagonals apart to avoid interference of one measurement with the other.

Here, an average hardness value of 700HV and 705HV were recorded for sample 1 (#D-9108DU015L) and sample 2 (#D-9999DU026L), respectively. More details about the hardness data can be find in the following tables.

Table 1. Hardness analysis of sample 1 (#D-9108DU015L) after surface polishing.

	A	B	C	D	E	F	G	H
1	NR.	Hardness	Method	Diag	Diag1	Diag2	HMin	HMax
2	1	695	HV 3	0.089483416	0.089220287	0.089746545	80	200
3	2	705	HV 3	0.088835005	0.088703454	0.088966555	80	200
4	3	701	HV 3	0.089096403	0.088446262	0.089746545	80	200
5	4	715	HV 3	0.088177649	0.088172118	0.08818318	80	200
6	5	691	HV 3	0.089750814	0.089224581	0.090277048	80	200
7	6	699	HV 3	0.089230046	0.089235511	0.089224581	80	200
8	7	701	HV 3	0.089101033	0.088966555	0.089235511	80	200
9	8	697	HV 3	0.08935614	0.089750814	0.088961465	80	200
10	9	697	HV 3	0.089356139	0.089487697	0.089224581	80	200
11	10	699	HV 3	0.089230046	0.089235511	0.089224581	80	200

Table 2. Hardness analysis of sample 2 (#D-9999DU026L) after surface polishing.

	A	B	C	D	E	F	G	H
1	NR.	Hardness	Method	Diag	Diag1	Diag2	HMin	HMax
2	1	695	HV 3	0.089483416	0.089746545	0.089220287	80	200
3	2	699	HV 5	0.115149276	0.114761397	0.115537155	80	200
4	3	785	HV 0,5	0.034371541	0.034368213	0.034374869	80	200
5	4	721	HV 2	0.071724229	0.071724229	0.071724229	80	200
6	5	693	HV 3	0.089619636	0.089765849	0.089473415	80	200
7	6	683	HV 3	0.090282066	0.089229656	0.091334476	80	200
8	7	695	HV 3	0.089483416	0.089220287	0.089746545	80	200
9	8	689	HV 3	0.089884887	0.090018961	0.089750814	80	200
10	9	693	HV 3	0.089619637	0.089755859	0.089483416	80	200
11	10	693	HV 3	0.089619665	0.089229656	0.090009674	80	200

Discussions:

1- Below are the heat treatment details provided to Macquarie researchers by RAM:

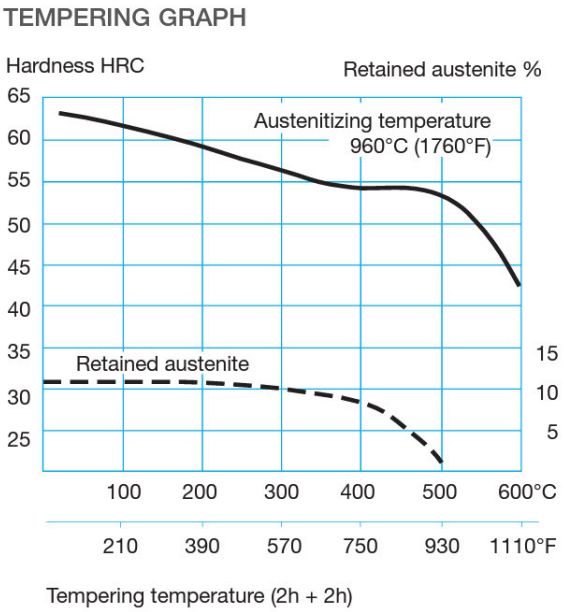
Step	Time (minutes)	Temperature (° Celsius)	Pressure (bar)	
0	0	Room temperature	0.0	
1	60	651	1.0	Austenitization
2	120	651	1.0	
3	60	961	0.0	
4	100	961	0.0	
5	1	40	1.6	Quench?
6	100	40	1.6	Hold
7	1	40	0.0	
8	40	165	1.0	Temper 1
9	150	165	1.0	
10	1	40	1.5	Quench?
11	60	40	1.5	Hold
12	40	150	1.0	Temper 2
13	150	150	1.0	
14	1	40	1.5	Quench?
15	100	40	1.5	
16	1	40	0.0	

The quench is recorded as one minute - but the resolution of the data log (provided by RAM) is one minute. So, the quench time could be 1 sec. In that case, the hardness value expected from the samples after “austenitization + quench” is around 820 HV (first row in the table below):

Cooling curve no.	Hardness HV10	T800-500 (sec)
1	820	1
2	762	107
3	743	423
4	734	1071
5	657	1596
6	455	3228
7	413	4292

At this stage, the tool steel should have martensite structure, which is very hard but brittle (not tough enough). So, RAM applied two tempering steps (temper 1 and 2) to reduce hardness but increase toughness.

According to the following tempering graph (provided by RAM), the tool steel should have a hardness of 59RC (727HV) after the tempering process. However, the hardness data obtained in this project was slightly lower (around 700HV) than the expected hardness from the samples (727 HV). **This could be due to slow quenching process used in the manufacturing of tool steel** (please see the future work for more details).



2- The microstructure analysis of the samples was conducted under SEM. Based on the obtained data, we are concluding that the crack has initiated from the substrate (tool steel) and extended from the substrate to PVD coating. **It is VERY unlikely for the crack to have initiated from the coating and propagated to the substrate.**

3- The EDX data collected in this project **was consistent with the information presented in the technical brochure provided by Udeholm Calmax**. The spectral analysis revealed distinct peaks corresponding to iron, chromium, molybdenum, and vanadium. The relative intensities of these peaks closely mirrored the expected elemental composition as outlined in the brochure.

4- PVD coatings are usually deposited at temperatures between 200°C (390°F) and 500°C (930°F). If a temperature of 200°C (390°F) is used, the substrate hardness will be higher than that obtained at a deposition temperature of 500°C (930°F). However, the adhesion of the coating on the steel is better if a deposition temperature of 500°C (930°F) is used.

The PVD deposition temperature should be approximately 20°C (68°F) lower than the previously used tempering temperature. According to the PVD coating data log provided by RAM, a PVD deposition temperature of 10°C or 48°C was used. **This low PVD temperature could not interfere with the heat treatment process applied on the dies**. *However, the coating could suffer from low adhesion to the surface, due to low deposition temperature.*

Potential future work:

It would be useful to obtain a sample of the Calmax steel that was used in die manufacture. This could be used to repeat the heat treatment process used, to determine the resulting hardness. Our team at MQ University is happy to run the test on the sample without any cost to RAM.