The Effects of Deep Cryogenic Treatment on PVD – TiN coated AISI M2 high speed steel

Name: Christian Chiadikobi[a]

Supervisors: Prof. Rob Thornton[a,b], Dr Dave Weston[a], Dr Dimitrios Statharas[a]

[a]School of Engineering, University of Leicester; [b]Warwick Manufacturing Group (WMG), The University of Warwick

Outline

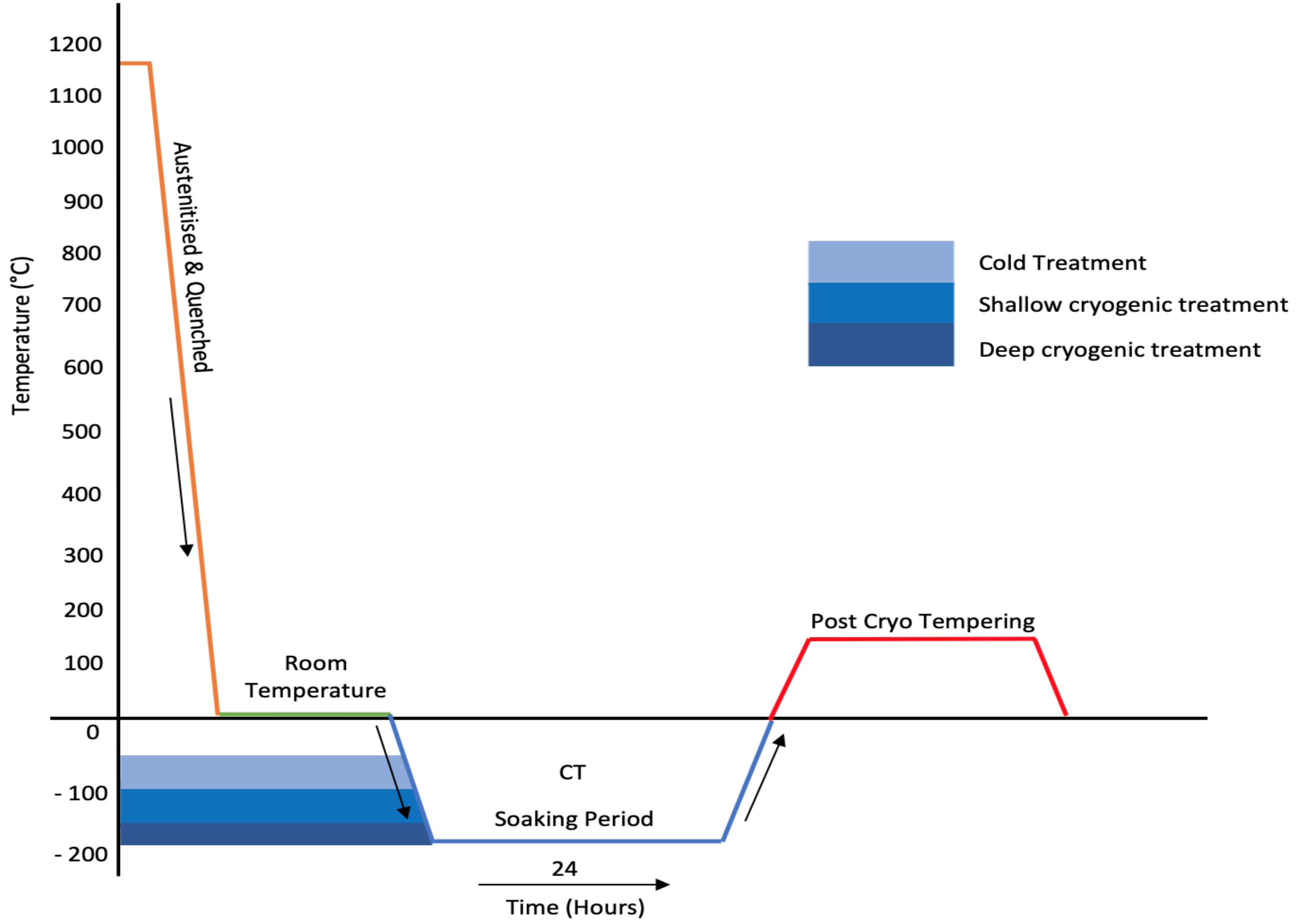
- *Background*
- *Motivation*
- *PhD Approach*
- *Experimental workflow*
- *Results*
- *Next step*
- *Conclusion*
- *References and Acknowledgement*

Background

- *How we understand it*
- *213K to 193K (-60 to -80 ° C)*
- *193 to 113 K (-80 to -160 ° C)*
- *113 to 77 K (-160 to -196 ° C)*
- *CT practice*

Industry: Restricted parameters, one cycle, varying practice – LN_2 or N_2, large batches

Research: Entire process, small quantities, N_2 cooling medium



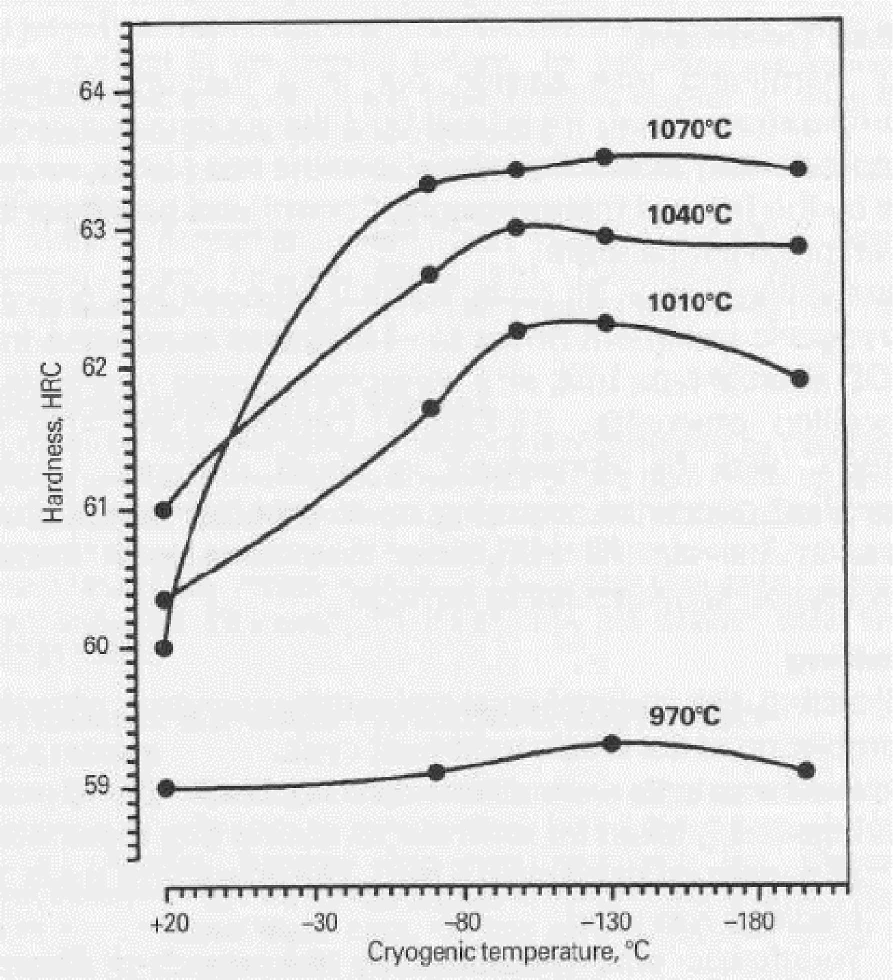
Background

CT Applications (Not limited to)

- *Manufacturing – Cutting tools*
- *Automobile - Brake Discs, Gears*
- *Medical - knives*

Some applications CT materials – coated

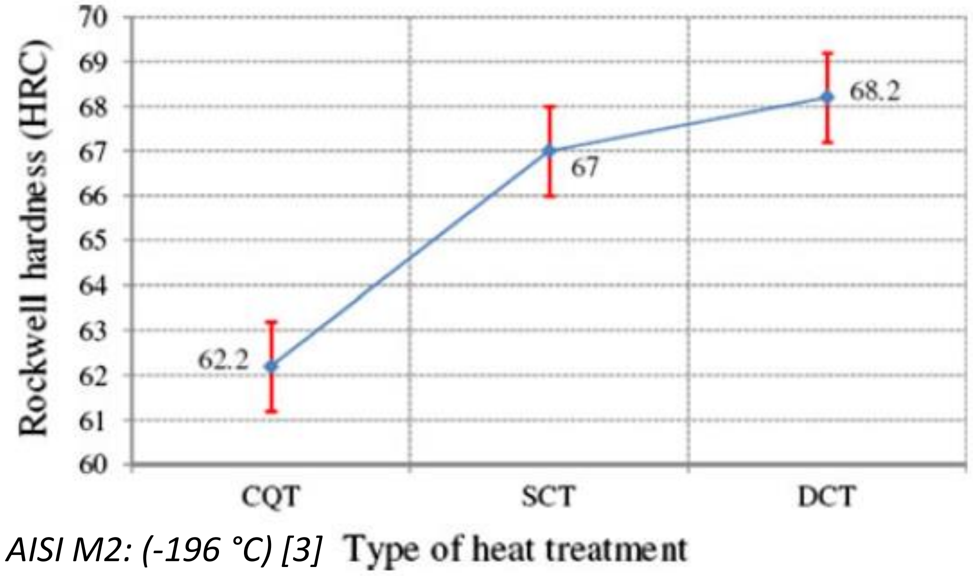
Motivation



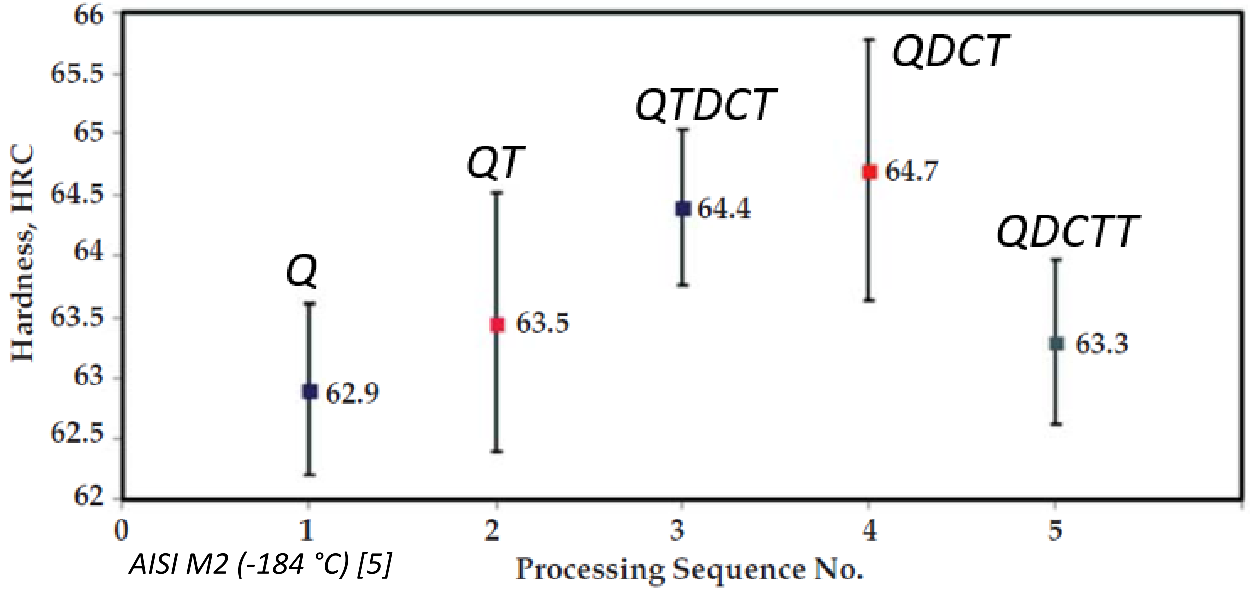
AISI D2	**-80**	**63.4 HRC**	**Reference [1]**
AISI D2	-196°C	63.7 HRC	Reference [2]

AISI D2: Max Hardness (-80 to -110 °C) [1]

Motivation



AISI M2: (-196 °C) [3]



AISI M2 (-184 °C) [5]

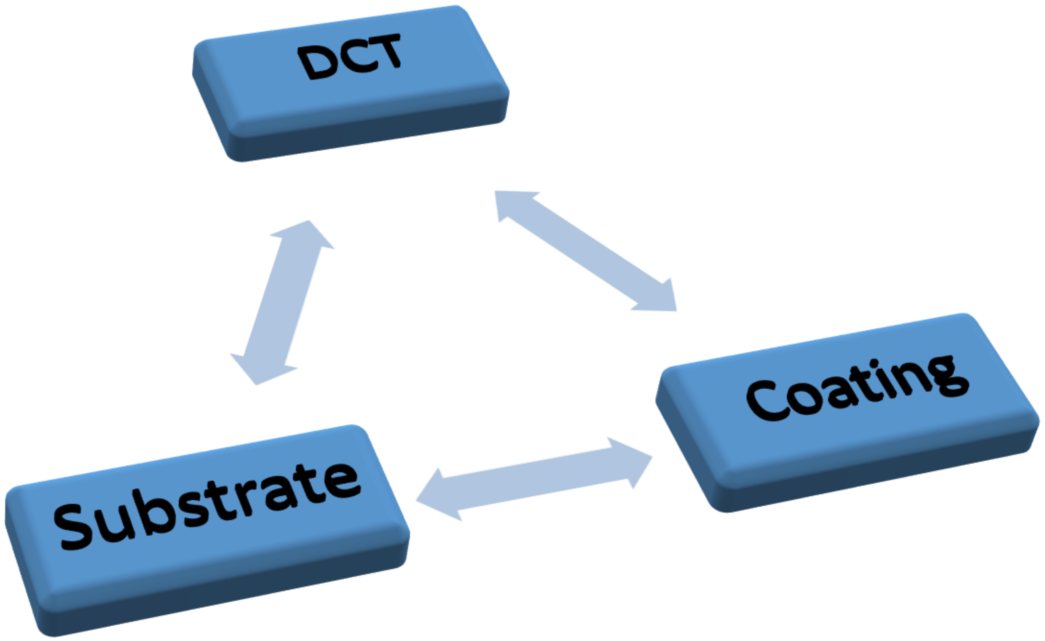
Effect of the cryogenic treatment on the tool hardness *AISI M2: (-196 °C) [4]*

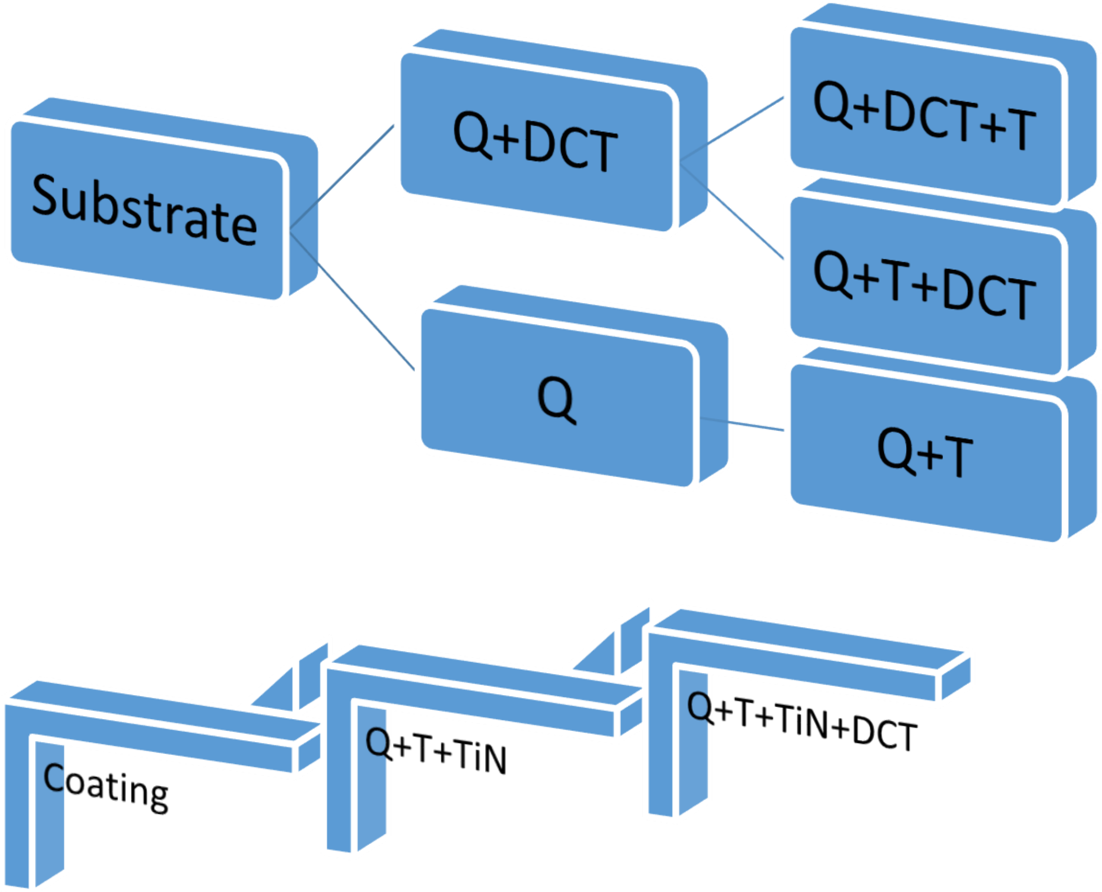
Treated tools	HRc	Untreated tools	HRc
A	66	D	66
B	65	E	65
C	66	F	66

(-196 °C/24 h) [6]

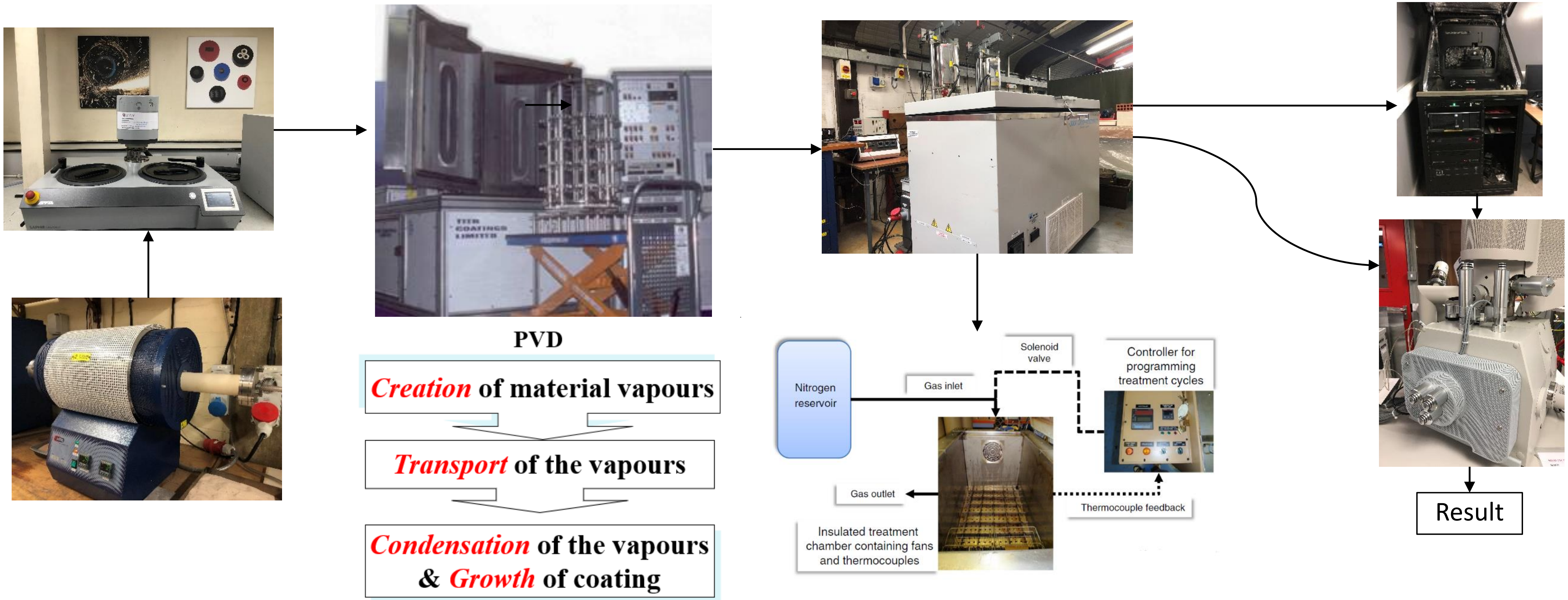
AISI M2 + TiN	DCT + TiN	153% tool life improvement
AISI M2 + TiN	TiN + DCT	109 % tool life improvement

Approach


DCT
Coating
Substrate


Substrate
Q+DCT
Q+DCT+T
Q+T+DCT
Q
Q+T
Coating
Q+T+TiN
Q+T+TiN+DCT

Experimental workflow



Coating set up/ process [Ref 8]

CT set up /technology [9]

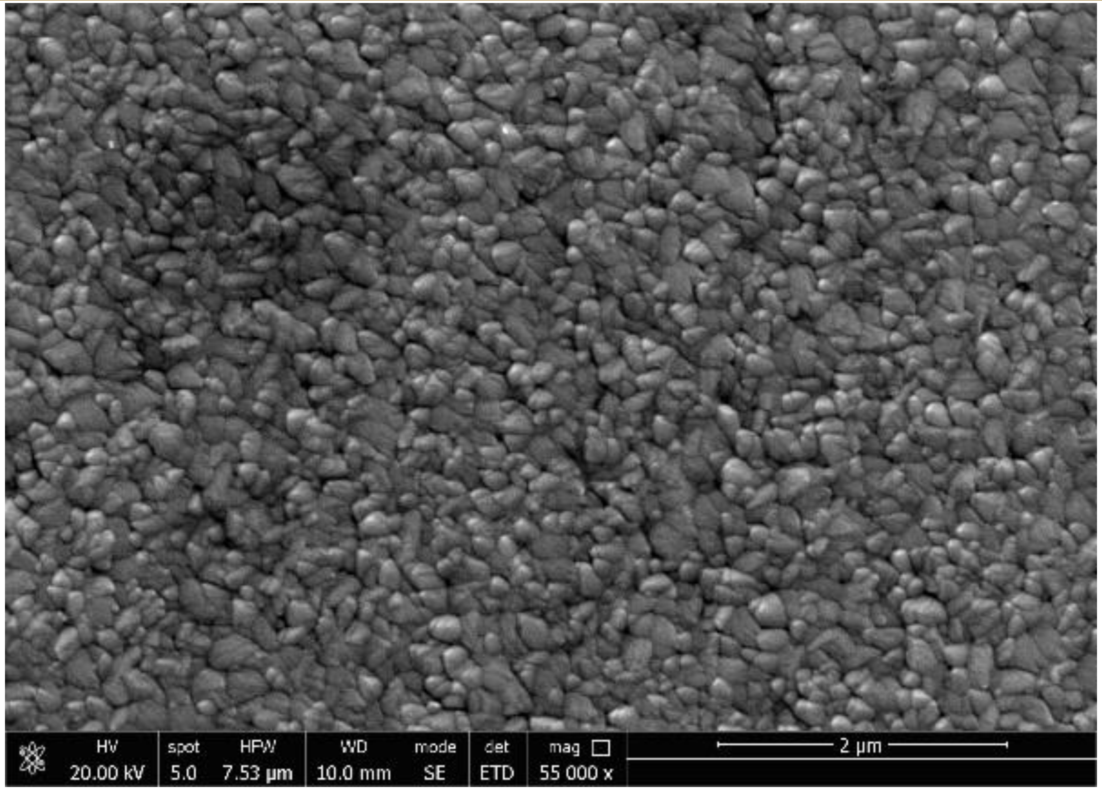
EPSRC
Engineering and Physical Sciences Research Council

UNIVERSITY OF LEICESTER

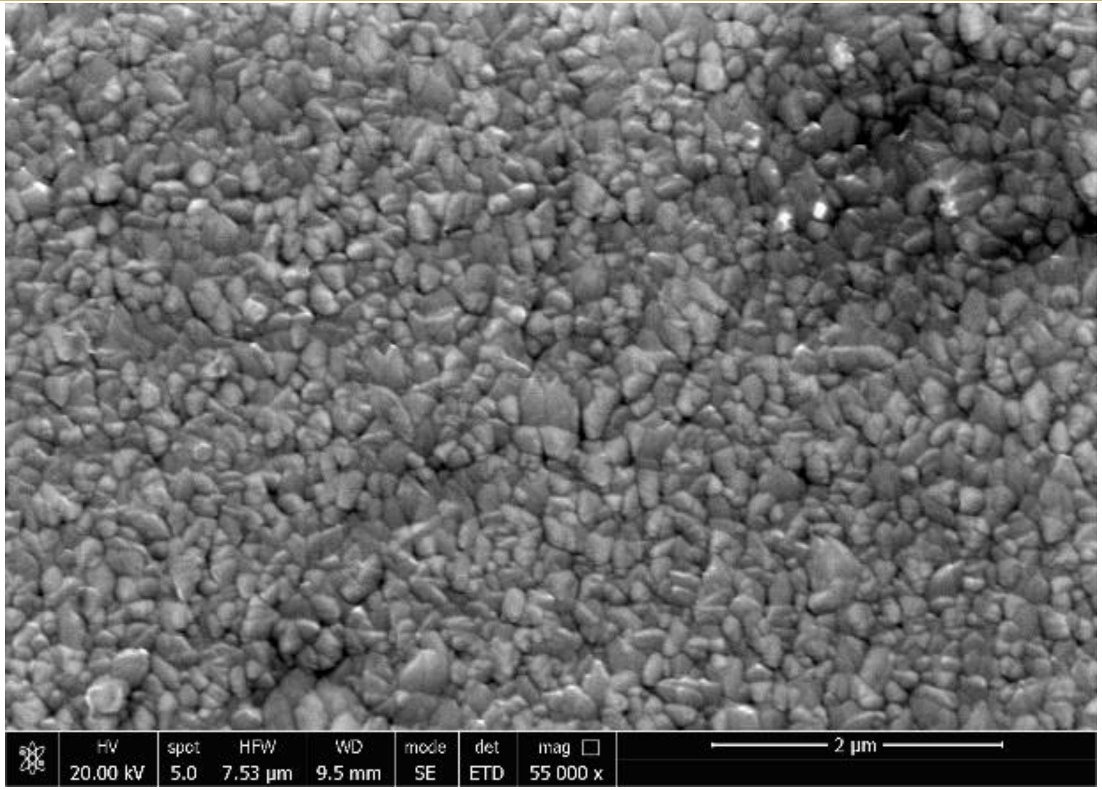
UNIVERSITY OF BIRMINGHAM

The University of Nottingham
UNITED KINGDOM · CHINA · MALAYSIA

Results – Bulk structure

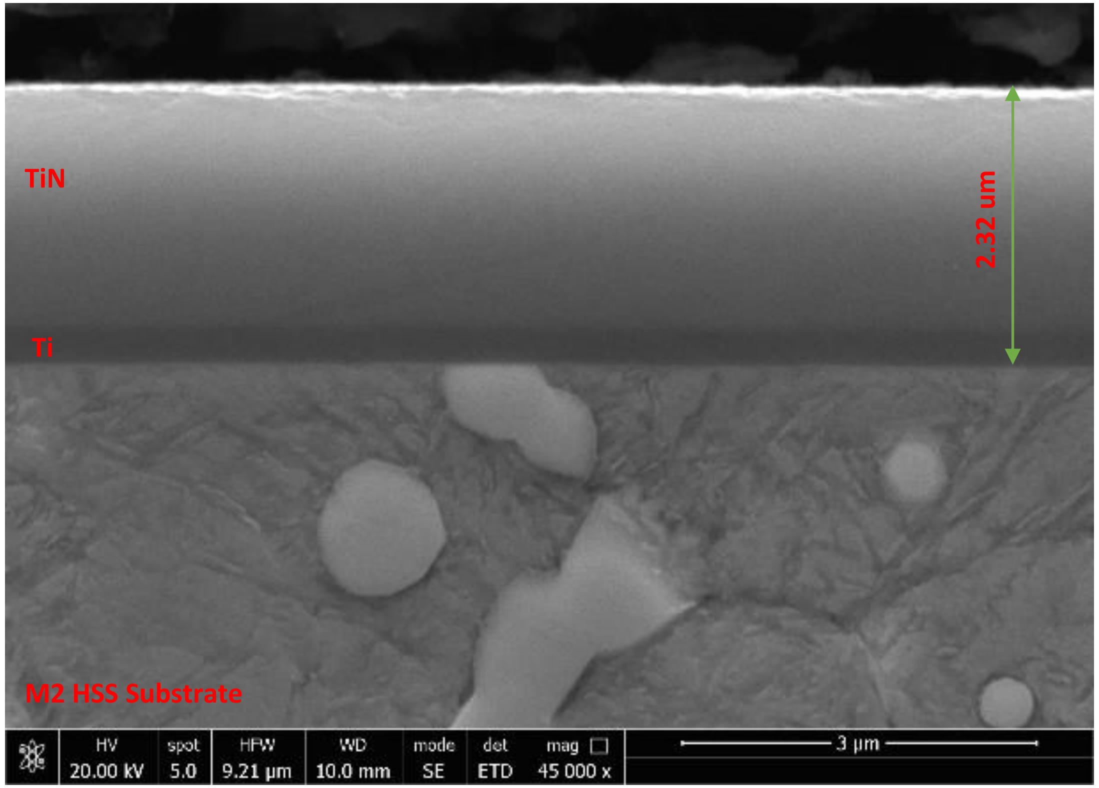


SEM Micrograph : Q+T+Coat – Coating surface

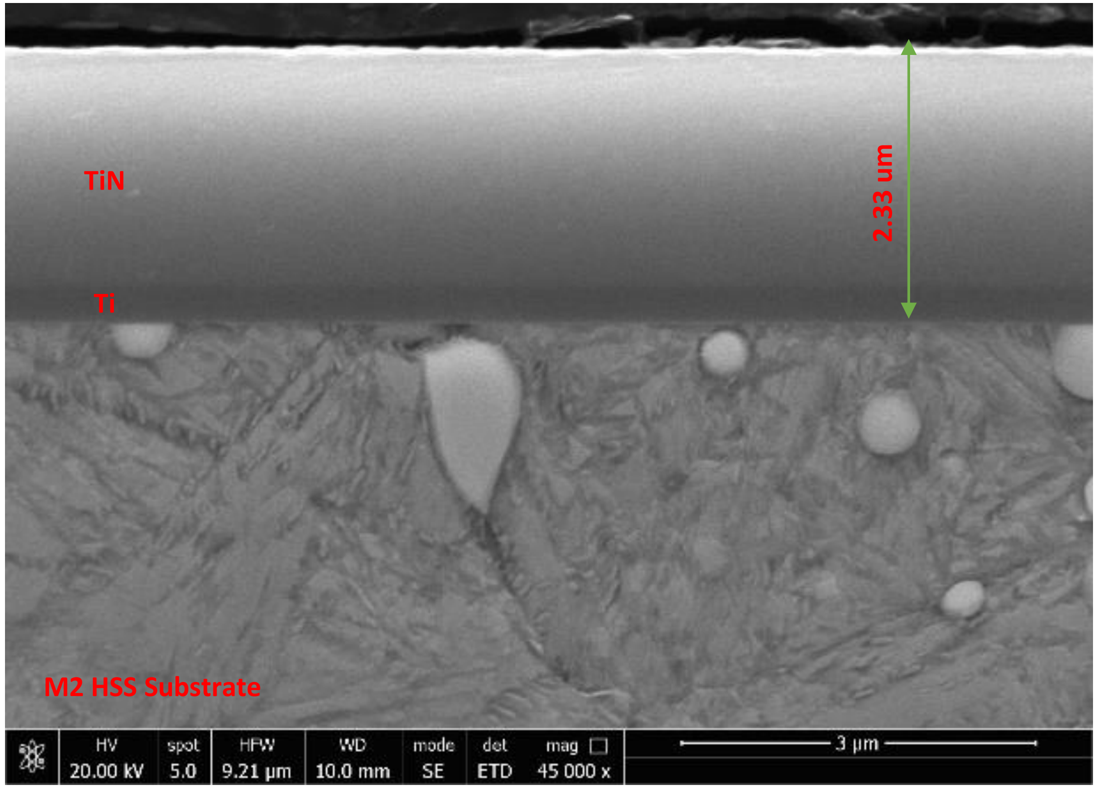


SEM Micrograph : Q+T+Coat+DCT – Coating surface

Coating cross section



SEM micrograph: Q+T+Coat – Cross section



SEM micrograph: Q+T+Coat+DCT – Cross section

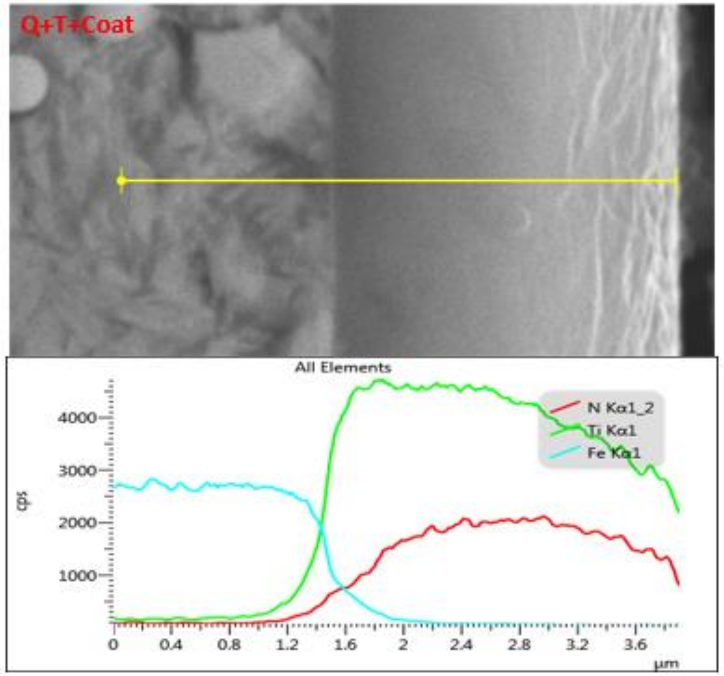
- Thickness - mean 2.33 (±0.01) µm, underlayer appro. 298 (±0.03) nm

EPSRC Engineering and Physical Sciences Research Council
UNIVERSITY OF LEICESTER
UNIVERSITY OF BIRMINGHAM
The University of Nottingham
UNITED KINGDOM · CHINA · MALAYSIA

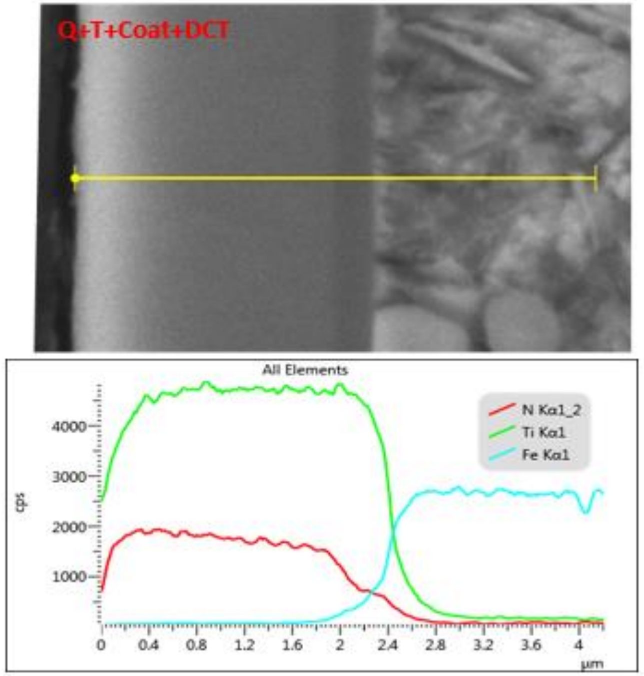
Coating cross section

Surface roughness of the system and standard deviation

Material	Surface Roughness (Ra)
Q+T	0.04 ± 0.02
Q+T+DCT	0.04 ± 0.02
Q+T+Coat	0.05 ± 0.002
Q+T+Coat+DCT	0.05 ± 0.001



a) Line scan on cross section: Q+T+Coat



b) Line scan on cross section: Q+T+Coat+DCT

Scratch tracks

Q+T+Coat

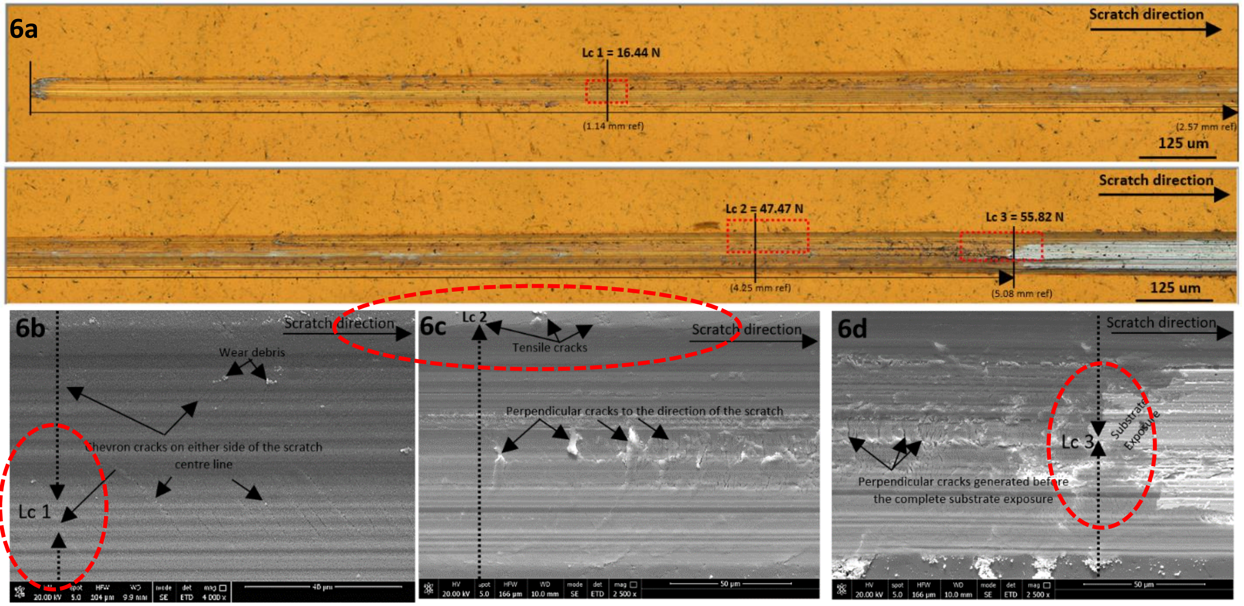


Fig. 6a Split up image from optical profiling microscope showing the scratch tracks to complete substrate exposure (for Q+T+Coat sample). SEM micrograph showing the Lc positions and cracks observed: (6b) Lc1 – 16.44 N (6c) Lc2 – 47.47 N (6d) Lc3 – 55.82 N (substrate exposure)

Q+T+Coat+DCT

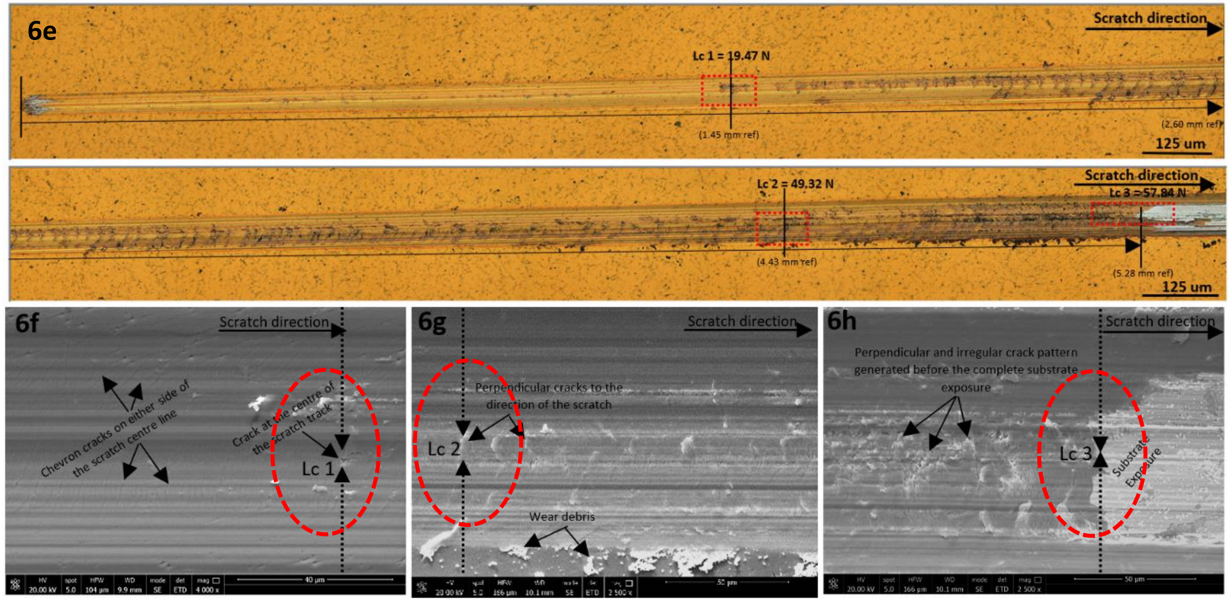
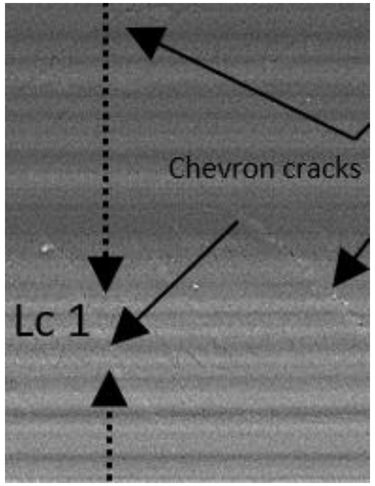


Fig. 6e Split up image from optical profiling microscope showing start of scratch tracks to the substrate exposure (for Q+T+Coat+DCT sample). SEM micrograph showing the Lc positions and cracks observed: (6f) Lc1 – 19.47 N (6g) Lc2 – 49.32 N (6h) Lc3 – 57.84 N (substrate exposure)

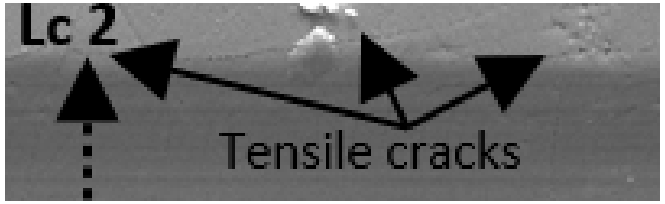
Lc positions

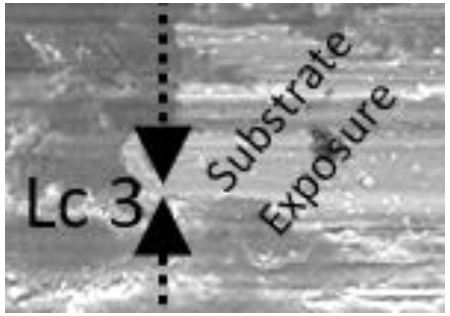
Table: 1 Results of scratch test and standard deviation

Samples	Lc1	Lc2	Lc3
Q+T+Coat	16.44 ± 2.08	47.47 ± 2.03	55.82 ± 2.92
Q+T+Coat+DCT	19.47 ± 1.70	49.32 ± 2.02	57.84 ± 3.11



Q+T+Coat

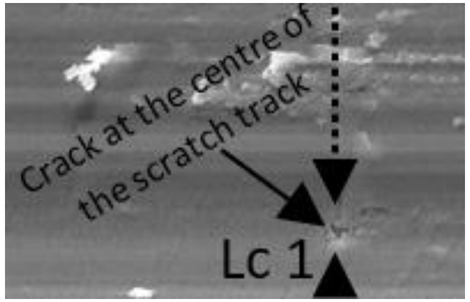


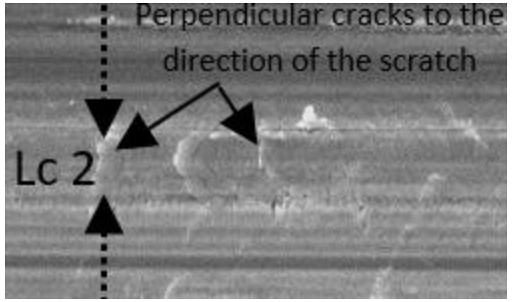


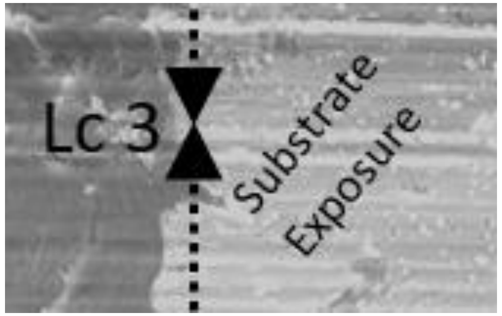
Chevron, transverse and tensile cracks

- Chevron cracks – either side of scratch centre line as well as cracks
- High Lc – irregular pattern cracks – fit into track and open away, perpendicular to the direction of the scratch.
- Denser and extensive
- Modulus and hardness

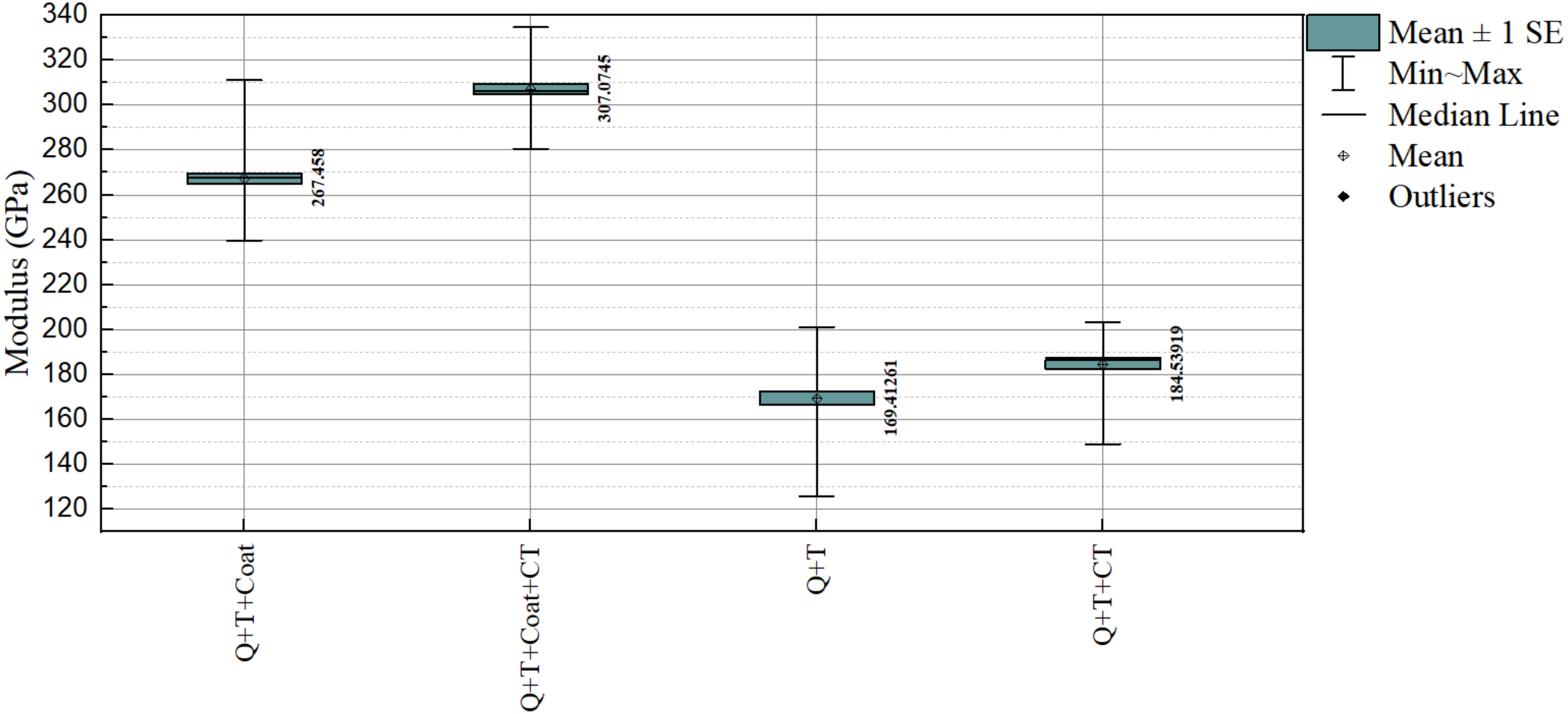
Q+T+Coat+DCT



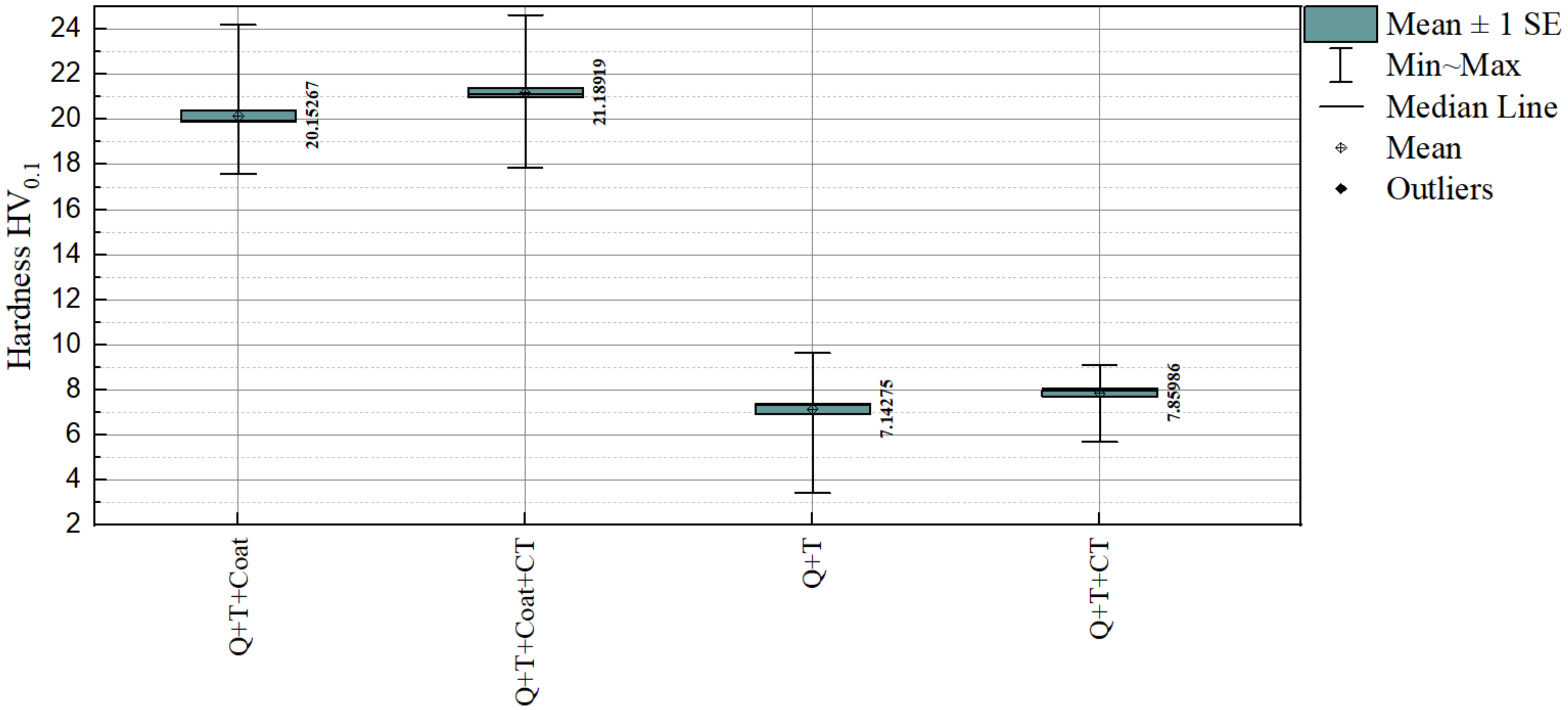




Modulus Measurement



Hardness Measurement



Summary table of mechanical properties

Table: 2 Summary table of the measured mechanical properties of the system and standard deviation

Material condition (Coating/substrate)	Elastic modulus (GPa)	Indentation Hardness (GPa)	Elastic modulus ratio (GPa) – E_{CD}/E_{SU}
Q+T+Coat	267.46 ± 13.40	20.15 ± 1.46	--
Q+T+Coat+DCT	307.07 ± 14.05	21.19 ± 1.34	--
Q+T	169.41 ± 17.56	7.14 ± 1.21	--
Q+T+DCT	184.54 ± 13.06	7.86 ± 0.80	--
Q+T+Coat – E_{CD}/E_{SU}	--	--	1.58 ± 0.04
Q+T+Coat+DCT – E_{CD}/E_{SU}	--	--	1.66 ± 0.04

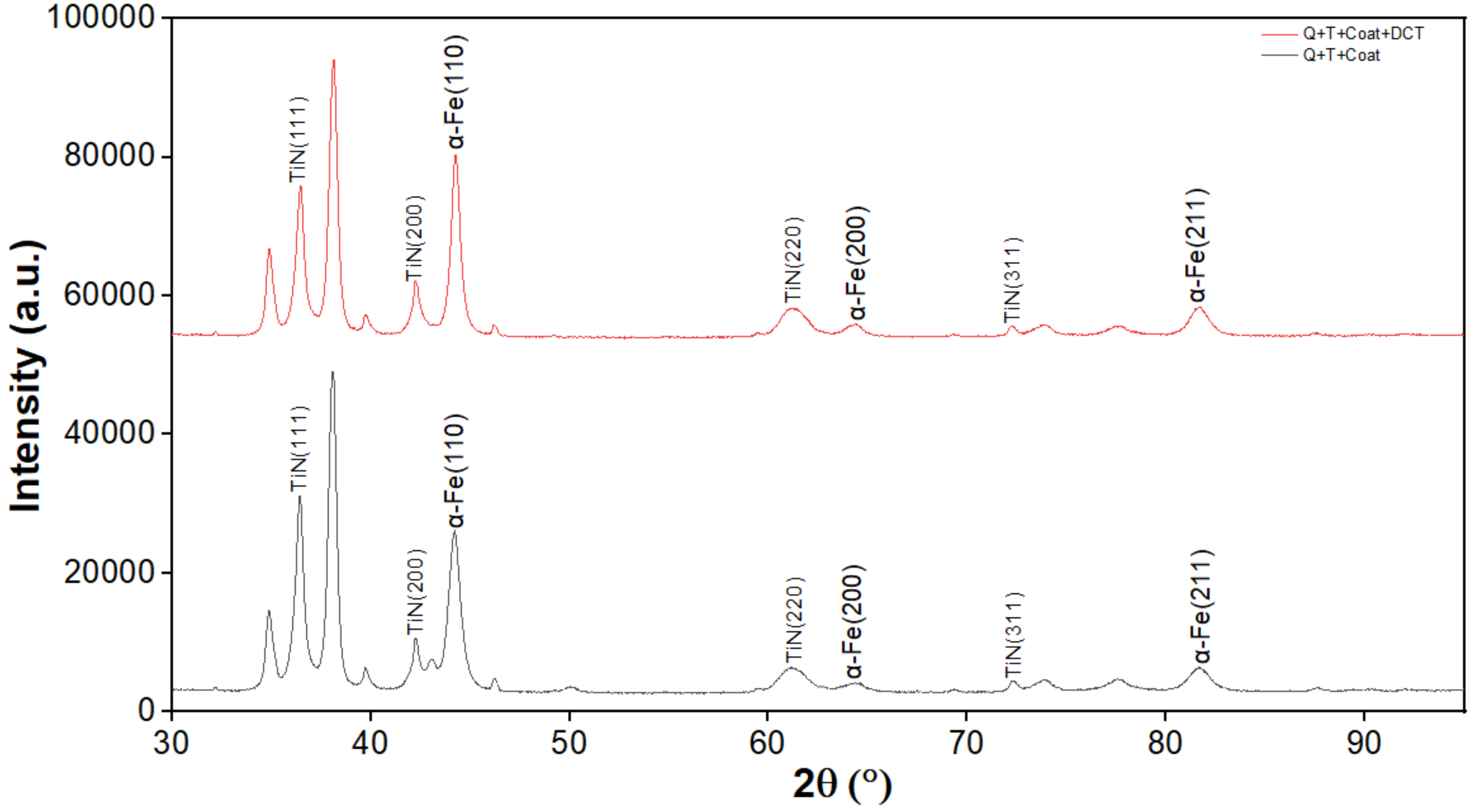
Table: 3 T-statistics for the measured values

T statistic	Test statistics (Q+T+Coat & Q+T+Coat+DCT) - Modulus	Test statistics (Q+T & Q+T+ DCT) - Modulus	Test statistics (Q+T+Coat & Q+T+ +Coat+DCT) - Hardness	Test statistics (Q+T & Q+T+ DCT) - Hardness
P - value	2.2572E-19	4.67168E-05	0.001248378	0.002026
Remark	Significant	Significant	Significant	Significant

- Elastic modulus - Lc 1 and Lc 2
- Hardness - Lc 3
- 3.62%

Diffraction pattern

- Q+T+Coat & Q+T+Coat+DCT exhibit similar pattern - Preferred orientation (111)
- TiN films: reflections corresponding to (111), (200), (220) and (311) was found at 36.51°, 42.57°, 61.37° and 72.26° respectively
- Substrate: reflections corresponding to (110), (200) and (211) assigned to 44.25°, 64.37° and 81.62° respectively



W-H: Crystallite size and strain

$$\boldsymbol{\beta_T \cos\ \theta = \ \varepsilon(4\sin\theta) + \frac{K\lambda}{L}}$$

$$\boldsymbol{Y = mx + c}$$

Y = $\beta_T\ cos\ \ \theta$; m = ε; $\boldsymbol{x}$ = 4 sin θ; c = $\frac{K\lambda}{L}$

Where: $\beta_T = \beta_D + \beta_\varepsilon$; and $\beta_D = \frac{k\lambda}{L\ cos\ \theta}$; $\beta_\varepsilon = 4\varepsilon\ tan\ \theta$; $L = \frac{k\lambda}{\beta_D\ cos\ \theta}$

β_T: Total broadening of the peak (combine effect of broadening due to the crystallite size (β_D) and broadening due to strain (β_ε));

L: Average crystallite size;

K: shape factor (constant 0.9)

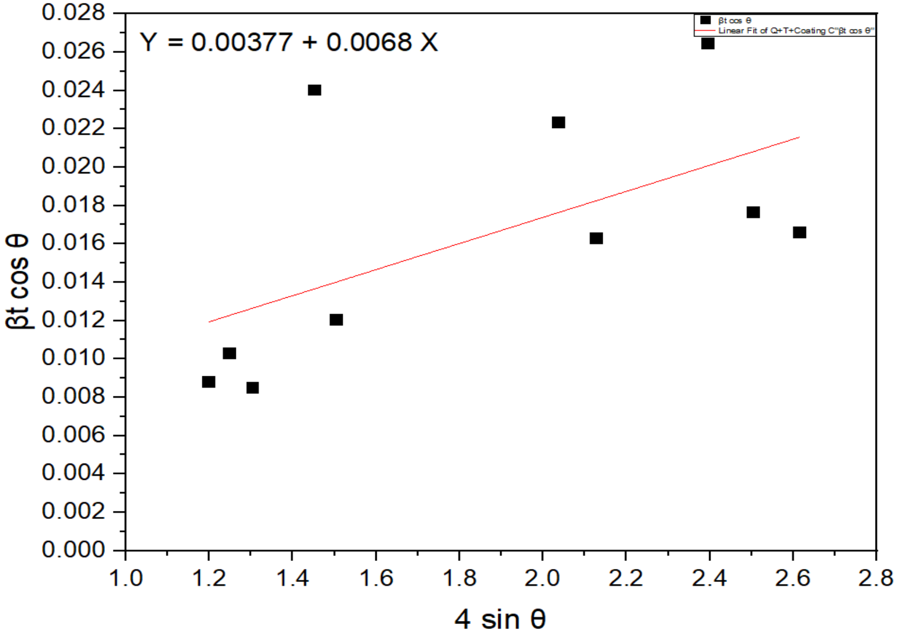
β_D: full width at half maximum (FWHM) broadening of peak (radians);

θ: peak positions (radians).

λ: 0.15406 nm (x-ray source); ε: strain

W-H: Crystallite size and strain

Q+T+Coat



Plot of βt cos θ against 4 sin θ for Q+T+Coat

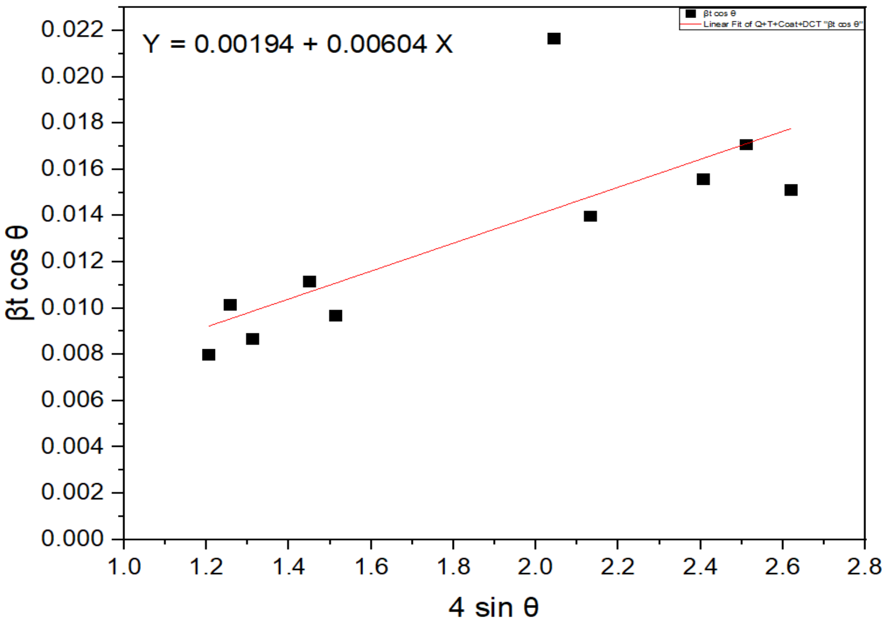
36.78 (±8.76) nm and strain of 0.00377 (±0.00634) %

- Constant, more scatter away from the fit

Williamson hall equation

$$\beta_T \cos\ \theta = \ \varepsilon(4 \sin \theta) + \frac{K\lambda}{L}$$

Q+T+Coat+DCT



Plot of βt cos θ against 4 sin θ for Q+T+Coat+DCT

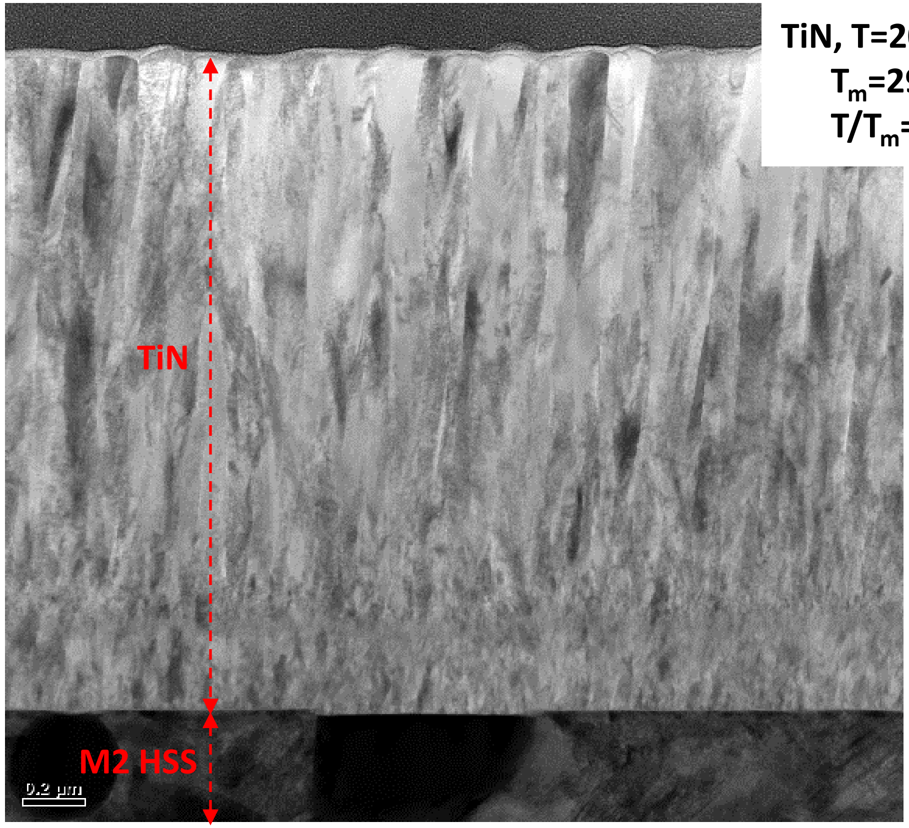
71.74 (±1.76) nm and strain of 0.00194 (±0.00908) %

- Constant, less scatter away from fit
- 71.47 (±1.76) nm, while strain decreased to 0.00194 (±0.00908) (0.49% reduction)

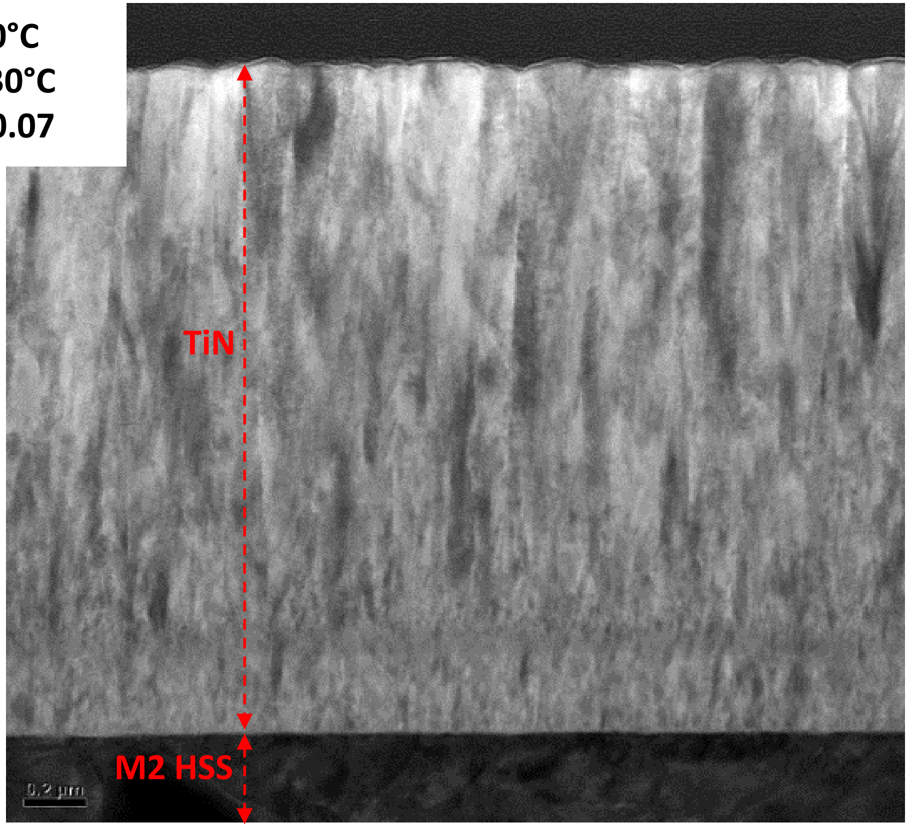
EPSRC Engineering and Physical Sciences Research Council · University of Leicester · University of Birmingham · The University of Nottingham UNITED KINGDOM · CHINA · MALAYSIA

Coating Morphology

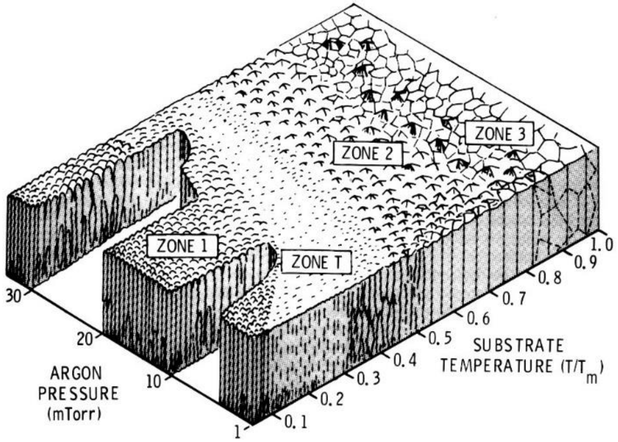
TiN, T=200°C
T_m=2930°C
T/T_m= 0.07



Cross section of Q+T+Coat via TEM - 80 000 mag



Cross section of Q+T+Coat+DCT via TEM - 80 000 mag



Structure zone Model 8]

1- Open fibrous like arrangement
T-fine grained, densely packed fibrous grains
2-fairly dense columnar grains
3-large recrystallised equiaxed grains

EPSRC Engineering and Physical Sciences Research Council
UNIVERSITY OF LEICESTER
UNIVERSITY OF BIRMINGHAM
The University of Nottingham UNITED KINGDOM · CHINA · MALAYSIA

Conclusions

- Improvement - 3.62%
- Combination of Elastic modulus and hardness
- Elastic modulus - Lc 1 and Lc 2
- Hardness - Lc 3

References

1. Collins, D. N. and Dormer, J., *Deep Cryogenic Treatment of a D2 Cold-work Tool Steel. Heat Treatment of Metals*. 1997. **3**: pp. 71-74.

2. Das, D., et al., *Effects of Deep Cryogenic Treatment on the Carbide Precipitation and Tribological Behaviour of D2 Steel*. Materials and Manufacturing Processes, 2007. **22**(4): pp. 474-480.

3. Gill, S.S., et al., *Effects of Deep Cryogenic Treatment on AISI M2 High Speed Steel: Metallurgical and Mechanical Characterization.* Journal of Materials Engineering and Performance, 2012. **21**(7): pp. 1320-1326.

4. Da Silva, F. J., et al., *Performance of Cryogenically treated HSS tools*. Wear, 2006. **261**(5): pp. 674-685.

5. Kelkar, R., et al., *Understanding the Effects of Cryogenic Treatment on M2 Tool Steel Properties*. Heating Treating Progress, 2007. **7**(5): pp. 57-60.

6. Mohan lal, D., et al., *Cryogenic treatment on argument wear resistance of tool and die steels*. Cryogenics, 2001. **41**(1): pp. 149-155.

7. Zhang, Z. and Dong, H., *Surface Engineering Technologies*. IMPaCT CDT lecture slide - Bham, 2018.

8. Slatter, T., and Thornton, R., *Cryogenic Treatment of Engineering Materials in Comprehensive Materials Finishing,* M.S.J. Hashmi, Editor. 2017, Elsevier: Oxford. Pp. 421-454.

Acknowledgements

- IMPaCT CDT is supported by EPSRC grant EP/R512308/1 & EP/L016206/1

- Jim Benson – Cryogenics Ltd (Industrial Partner)
- Advanced Microscopy Facility – University of Leicester
- Loughborough Materials Characterisation Centre – Loughborough University

Thank you for your attention