Micro-scale Impact testing -
a new approach to studying fatigue resistance

Introduction

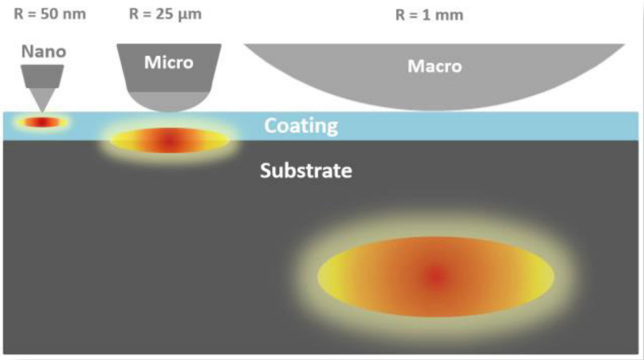

Figure 1. Schematic illustration of the influence of probe geometry on peak stresses. Changing load and/or probe radius makes the test more sensitive to coating mechanical properties or interfacial adhesion

Testing mechanical and tribological properties at the correct length scale and temperature provides more relevant data e.g. for optimising coating composition for improved performance in demanding applications such as cutting tools or in mechanical contact in aero-/auto engines. Although popular for their simplicity, many macro-scale mechanical contact tests are less sensitive to the properties of thin CVD and PVD coatings when the large probe radius and very high contact forces in the test result in peak stresses far into the substrate as illustrated schematically in figure 1.

Improved testing is possible by an integrated multi-scale – (nano- and micro-scale) characterisation. In integrated nanomechanical testing the probe radius and applied load can be tuned to increase the sensitivity to the region of interest. For coating-only mechanical properties from nanoindentation, sharp (Berkovich) probes and small loads are needed. For micro-scratch and impact tests, larger loads and blunter probe geometries enable the peak stresses to be positioned closer to the interface so that the full coating-substrate system behaviour can be evaluated.

Testing at the correct temperature is particularly important where coating and/or substrate properties are strongly temperature-dependent as this can result in a change in deformation mechanism from room temperature. In addition to nanoindentation, the NanoTest Vantage can easily be configured to perform many other high temperature tests which are described in this technical note: (1) micro-scratch (2) repetitive micro-scratch [see fig. 2] (3) micro-impact (4) nano- impact and how they can be used to streamline coating research. The nano- and micro scale scratch and impact tests correlate with coating performance in cutting tests [1-4].

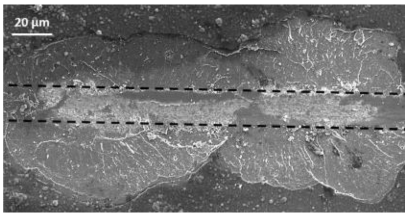

Figure 2. SEM of repetitive scratch at 500 °C on AlCrN

Experimental high temperature data can be supported by modelling [fig. 3]. Simulated stress distributions of the temperature-dependent changes in scratch tests provide mechanistic information explaining why and where coating systems fail in scratch tests, enabling data to be interpreted more effectively.

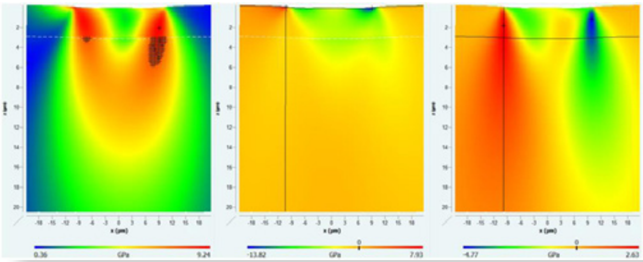

Figure 3. Micro-scratch of TiAlN at 500 °C. Stress distributions at L_{c2}
Left = von Mises stress. Middle = normal stress. Right = shear stress.

Case studies

1.High temperature micro-scratch testing of AlTiN, AlCrN and TiAlN coatings

High temperature micro-tribological tests simulate high contact pressure sliding/abrasive contacts at elevated temperature. Ramped load micro-scratch tests to a peak load of 10 N were performed on 3 µm monolayer PVD AlTiN, AlCrN and TiAlN coatings deposited on H10A cemented carbide (6 wt.% Co) cutting tool insert with a 25 µm spherical diamond. Scan speed = 5 µm/s. Loading rate = 0.075 N/s. Track length = 1 mm. Tests were performed as a three-scan procedure (1 = pre-scratch surface topography scan; 2 = ramped scratch; 3 = post-scratch surface topography scan). Repetitive micro-scratch tests were performed as 12 scan experiments (1 = pre-wear surface topography; 2-11 = constant load scratches; 12 = post-wear surface topography). The ramped load and repetitive scratch tests were performed at 25 and 500 °C. Nanoindentation tests (25 mN, diamond Berkovich) were also performed at these temperatures.

Temp	AlCrN		TiAlN		AlTiN	
	L_{c1} (N)	L_{c2} (N)	L_{c1} (N)	L_{c2} (N)	L_{c1} (N)	L_{c2} (N)
25 °C	2.8 ±0.2	5.7 ±0.2	2.1 ±0.4	4.2 ±0.5	3.5 ±0.8	4.5 ±0.1
500 °C	2.3 ±0.4	6.0 ±0.4	1.5 ±0.2	3.7 ±0.2	4.0 ±0.6	5.8 ±0.7

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In micro-scratch tests the critical load decreased with temperature for TiAlN but increased for AlTiN and AlCrN. The post-scratch topography profile revealed unloading failures at lower critical load, especially on AlCrN (fig.4). SEM showed that these were within the coating.

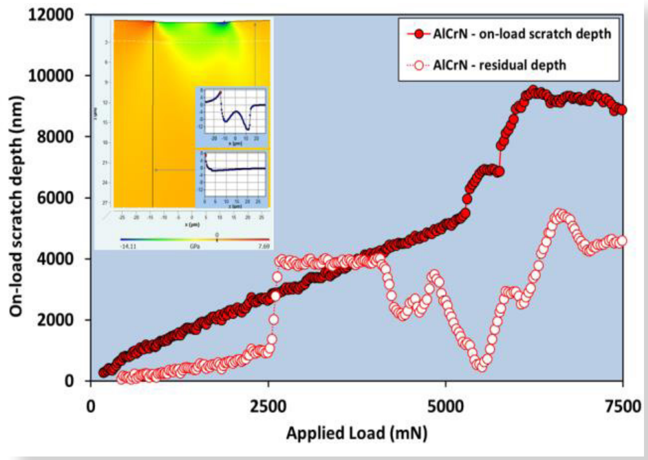

Figure 4. On-load and residual scratch depth data on AlCrN at 500 °C. Inset = simulated normal stresses.

Analytical modelling (SIO software) with the NanoTest high temperature scratch and mechanical property data from the high temperature nanoindentation used as inputs shows that maximum normal stress is high (e.g. 7.7 GPa at L_{c2} failure) at the surface immediately behind the sliding contact (sliding direction is left to right in inset).

Many factors can influence the critical load in the scratch test (e.g. substrate hardness, coating thickness, interfacial adhesion…). The increase in L_{c2} critical load for AlCrN, and especially AlTiN, as the temperature is increased to 500 °C was not directly related to an improvement in adhesion strength. The SIO modelling shows that the interfacial shear stress actually decreased with increasing temperature and the higher L_{c2} is a result of the changing stress distribution in high temperature sliding [1].

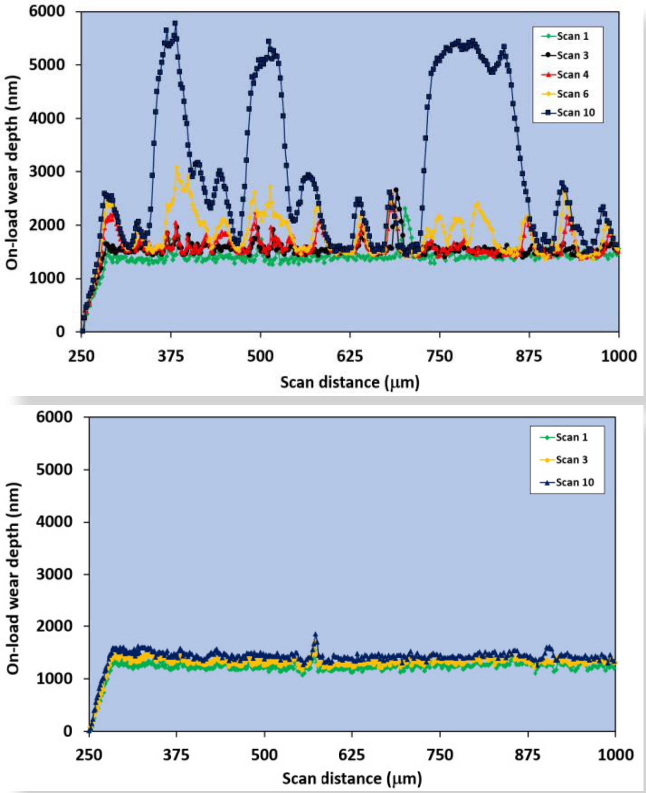

Figure 5. Evolution of on-load wear depth in repetitive scratch tests at 1 N AlTiN at 25 °C (top) and 500 °C (bottom).

In the repetitive scratch tests at 1 N AlTiN was more wear resistant than AlCrN and the wear process proceeded by a different mechanism on TiAlN. At room temperature coating wear proceeds by a fracture-dominated process. At 500 °C the coatings and substrate soften reducing the maximum stresses developing in the coating-substrate system, consistent with lower driving force for brittle fracture. The improved performance of AlTiN in the 1 N repetitive scratch test at 500 °C is explained by the stress distribution in contact resulting in a change in yield location due to the different high temperature mechanical properties on this coating.

The improved high temperature performance for the high Al-fraction coatings AlTiN and AlCrN is strongly correlated with their tool life, where they out-perform TiAlN in many cutting applications as summarised in [1,3].

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2. High temperature micro-impact testing of monolayer and multilayer TiAlSiN coatings

Repetitive micro-impact tests were performed at 25-600 °C on 2 µm PVD monolayer TiAlSiN and multilayer TiAlN/TiSiN coatings deposited on ISO P30 cemented carbide (10 wt.% Co) at 1.5 N with a 17 µm spherical diamond [2]. Test duration was 300 s at 25 °C and 150 s at 300-600 °C (75 impacts). Ramped load micro-scratch tests to a peak load of 5 N were performed with a 25 µm spherical diamond. Test temperatures were 25, 300, 500 and 600 °C for the impact and 25, 300, 600 °C for the scratch tests. The experiment load history was modified to reduce time in contact at elevated temperature. Nanoindentation tests were performed at 30 mN with a Berkovich diamond indenter at room temperature and a cBN Berkovich indenter in a purged Ar atmosphere at 400 and 600 °C. Temperature-dependent changes in the elastic properties of the cBN indenter were accounted for in calculating the coating elastic modulus.

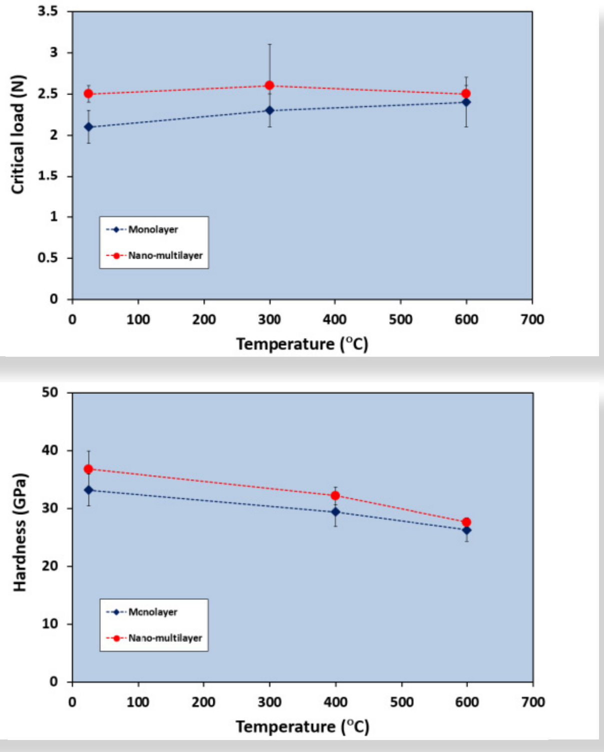

Figure 6. (top = nanoindentation, bottom = Lc2 in micro-scratch)

In comparison with the monolayered ternary nitride coatings discussed in Case study 1, the TiAlSiN coatings have excellent high temperature mechanical properties, with only a small decrease in hardness at 600 °C. The micro- scratch test critical load was relatively insensitive to temperature reflecting increased coating bending due to lower load support from substrate softness at high temperature being offset by reduced brittleness.

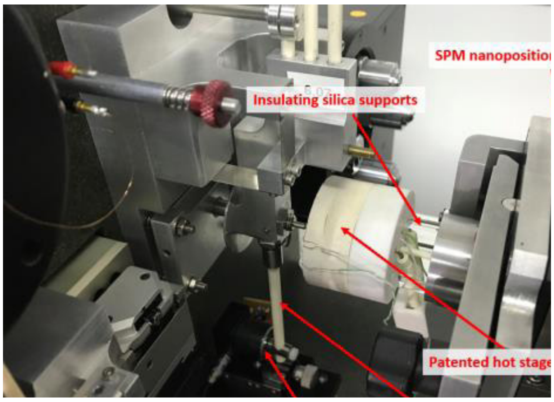

Figure 7. Configuration for high temperature impact

In the micro-impact tests at room temperature both coatings show a brittle response with a transition to more rapid wear after ~15-20 impacts (fig. 8).

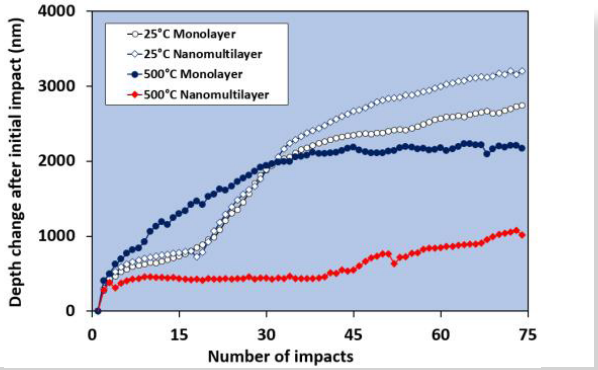

Figure 8. Impact depth increase vs. number of impacts

At higher temperature there was a transition to a milder wear regime with more plasticity and less brittle fracture. With its improved performance the nanomultilayered coating is promising candidate for high temperature applications involving repetitive contact (e.g. interrupted turning and milling applications).

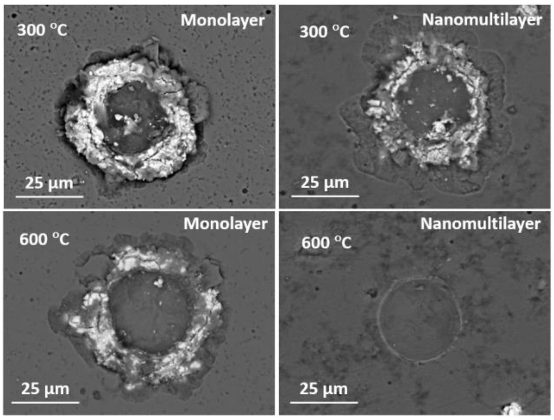

Figure 9. SEM images of impact craters at 300 and 600 °C

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3. High temperature nano-impact of TiAlN and AlTiN coatings

Elevated temperature nano-impact tests with a sharp cube corner on TiAlN and AlTiN coatings on H10A cemented carbide revealed decreased fracture probability in the nano-impact test at 500 °C than at 25 °C for both coatings [3,4]. This was consistent with enhanced plasticity at temperature shown by elevated temperature nanoindentation.

Coating	Temp	Plasticity index	Fracture probability
TiAlN	25 °C	0.48	0.8
TiAlN	500 °C	0.7	0.5
AlTiN	25 °C	0.52	0.4
AlTiN	500 °C	0.73	< 0.2

There was a correlation between the nano-impact results and the results of cutting tool tests with these coatings. The AlTiN coating exhibited better resistance to fracture in the elevated temperature nano-impact test and improved performance in (i) face milling AISI 1040 steel [+90% tool life] (ii) end milling AISI 4340 steel [+20% tool life] (iii) end milling Ti_6Al_4V alloy [+70% tool life].

Benefits of NanoTest high temperature nano- and micro-scale testing

- Streamline your research with an integrated multifunctional high temperature test capability in a single platform
- Fully instrumented nano- and micro-scale tests to simulate high temperature contacts e.g. as in metal cutting
- High temperature nano- and micro scale scratch and impact tests correlate with coating performance in cutting tests
- Continuous turning simulated by repetitive micro-scratch
- End milling simulated by high temperature nano- and micro- impact
- Integrated advanced analytical modelling option

Micro Materials at the forefront of nanomechanics since 1988:-

- The first commercial high-temperature nanoindentation stage
- The first commercial nano-impact stage
- The first commercial liquid cell
- The first commercial instrument for high-vacuum, high-temperature nanomechanics

NanoTest Vantage specifications

- NanoTest patented hot stage for elevated temperature testing (including indentation, scratch, impact etc.) at the nano- and micro- scale across the entire temperature range (to 850 °C in the Vantage).
- Both loading heads (0-500 mN and 0-30 N) are permanently mounted.
- The NanoTest patented hot stage is fully compatible with the SPM nanopositioner for high temperature operation allowing precise targeting of high temperature tests.
- NanoTest Vantage software includes data export routines for SIO formats so that the SIO modelling capability is fully integrated across the full temperature and load range.

References and acknowledgements

[1] Elevated temperature repetitive micro-scratch testing of AlCrN, TiAlN and AlTiN PVD coatings, BD Beake, JL Endrino, C Kimpton, GS Fox-Rabinovich, SC Veldhuis, IJRMHM 69 (2017) 215.

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[4] Investigating the correlation between nano-impact fracture resistance and hardness/modulus ratio from nanoindentation at 25-500 °C and the fracture resistance and lifetime of cutting tools with Ti1-xAlxN (x=0.5 and 0.67) PVD coatings in milling operations, BD Beake, JF Smith, A Gray, GS Fox-Rabinovich, SC Veldhuis, JL Endrino, Surf Coat Technol 201 (2007) 4585.

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