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High temperature impact testing of a thin hard coating using a novel high-frequency *in situ* micromechanical device

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A B S T R A C T

High-impact applications such as forging, punching or fine blanking often utilise hard-coating systems to improve wear and lifetime performance. For rational design of such systems, evaluation after repeated impacts at the application temperature is critical. However, such investigations are time-consuming and costly to perform in-line on an industrial scale. Small-scale impact testing allows for rapid and inexpensive investigation of attractive thin coating designs, which can be performed over a range of controlled temperatures. Here, a novel *in situ* nanoindentation device was developed, able to perform precise displacement cycles to 1 kHz at temperatures up to 500 °C within a scanning electron microscope. The system is based on a piezoelectric driving tip containing four discrete piezo devices, allowing for the collection of load-displacement data for each individual cycle. Operation of the device at 500 Hz was demonstrated on arc-PVD chromium nitride coated nitrided steel with a diamond flat-punch counter-body, and the effects of temperature and impact number on fatigue deformation studied using electron microscopy techniques. It was found that as the impact number increased as did the residual plastic damage from the impact force. Additionally, the deformation of the tooling was highly dependent on the temperature, which became more strongly apparent with an increased number of impacts. Such testing will be useful for evaluating the lifetime and fatigue properties of potential new thin films and coatings for high-impact operations, where understanding the influence of temperature is crucial.

1. Introduction

Many applications of precision tooling require operation at both high temperatures and with tens-of-thousands-of impact cycles. Such repetitive loading can fatigue the system, ultimately leading to tool failure through significant plastic deformation, fracture, or coating debonding. An understanding of the properties and processes of tool fatiguing and failure under service conditions is therefore crucial for the design and optimisation of tools with long service lifetimes. The effects of high impact energies have been traditionally investigated using Charpy tests [1], electro-dynamic or electro-mechanical testing actuators, and servo-hydraulic impact testing [2]. A split Hopkinson pressure bar, Taylor, or planar plate impact tests are additionally used for high strain rates [3]. Along with in-line industrial testing of parts, such testing and analyses may be expensive, inefficient, or do not allow for the influence of impact cycling for large tool pieces which are designed to survive many thousands of impact cycles. Moreover, for the study of thin-films and coatings on tool pieces, the thin coating is not isolated in the large stress fields generated under loading and is therefore not exclusively tested. In addition, large scale tests do not take into account asperity contact at the micron scale, and third-body contact which may occur during tool operation [4]. Such factors are highly important for a number of technical applications where wear and coating failure is to be avoided, such as for scratch resistant transparent coatings [5,6] and micro- and nano-electro-mechanical system (MEMS/NEMS) based devices [7–9]. For forging and cutting tool applications, physical vapour

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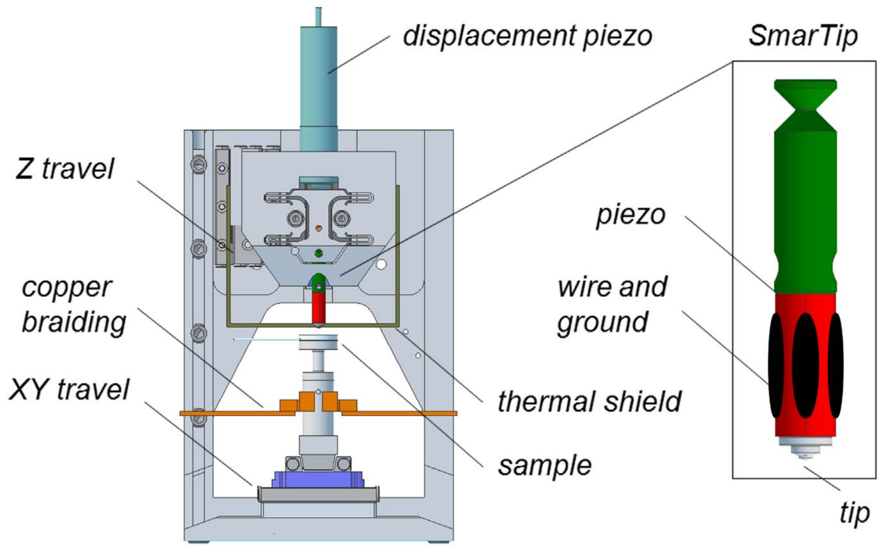

Fig. 1. Alemnis SEM indenter with SmarTip, and magnified schematic image of the SmarTip.

deposited (PVD) thin ceramic films have shown particular promise [10].

Precise nano-mechanical investigations are becoming more important as coating technology is engineered on smaller scales. Small-scale testing of materials allows for a number of inherent advantages, including that a series of measurements can be performed on a single specimen negating the influence of sample variation, and that damage can be restricted to the depth of the thin coating. Small-scale mechanical measurements have allowed for a significant reduction in the cost of materials testing, and the coupling of piezo-electric actuators can allow for the application of high-strain rates and high frequency responses. For small-scale repetitive impact testing of samples, significant advances have been made over the last decade. Pendulum impulse impact based systems have been demonstrated by Micro Materials with their NanoTest range of instruments, with reported rates of approximately 0.25–1 Hz [11,12]. There have been a number of reports utilising this system for room temperature impact testing of hard coating materials using a nanoindentation tip [11,13]. Such a system allows for a simple measure of indentation depth [13,14]. However recent advances in data acquisition, whereby the sampling rate is significantly increased, have allowed for the resolution of the mechanical response during each impact using dynamic capacitance measurement [15,16]. Data sampling at 20 kHz has been found to allow for investigation of kinetic and potential energies during impact, along with the elastic and plastic impact displacement, rebound heights and peak force [16]. Other important advances have also been made for increasing the scope and versatility of impact-based testing devices. Makrimallakis and colleagues introduced a system with a piezoelectric device allowing for modulation of the applied impact forces [17], and linked experimental observations to finite element analyses as was also reported for impact testing of hard ceramic coatings [18]. Regarding higher frequency testing, room temperature impact testing to 50 Hz and 5 kN has been developed at Fraunhofer IST allowing for up to a million impacts per test, while the NanoTest systems have demonstrated impact testing at 500 °C for TiAlN and AlTiN coatings at a rate of 0.25 Hz [19,20] where parameters such as the fracture probability could be established after testing at high temperatures.

Current testing approaches, and especially at high temperatures, remain in a fledgling state, and have not yet demonstrated high frequency impact testing to allow for high-cycle fatigue analysis. Of particular importance is the performing of such testing: at elevated temperatures; with *in situ* electron microscope operation; rapid sampling to allow for the generation of real-time load-displacement data. The collection of accurate load-displacement data is especially important for the on-line and time dependent analysis of material response in relation to high-cycle loading. There exists, therefore, a strong impetus for the design of a stable micro-mechanical impact tester able to produce load-displacement information at high-temperatures.

In the presented work, an *in situ* SEM device was designed and constructed using a quad-piezo driver, able to apply controlled displacements at up to 1 kHz of a nanoindentation tip and record load variation. Thermal calculations were performed, and a thermal shield designed, so that stable operation of the driving piezo could be maintained for sample temperatures up to 500 °C. The performance and limitations of the device were established through testing of a thin chromium nitride (CrN) coated nitrided steel at a range of temperature conditions with a diamond flat-punch micro-compression tip. The residual imprints from the impact tests were then characterised using electron microscopy techniques to highlight the usefulness of precision high temperature nano-impact testing, and the influence of temperature and impact number established for the model tooling system.

2. Design and operation of the SmarTip

2.1. High dynamic indenter

The device used in this work is an SEM indenter developed by Alemnis AG [21] (Fig. 1). It is composed of a load cell (500 mN maximum load) which measures the load at the milli-Newton scale (RMS noise 4 μN at 20 Hz bandwidth) and a piezo actuator which permits the application of displacements in the range of a few nanometers to 35 μm (RMS noise 2 nm at 20 Hz bandwidth). A detailed description of the device has been presented elsewhere [22]. The device can be used to perform indentation tests and micro-pillar compression, and can additionally be used *in situ* with an SEM in order to account for sample-tip alignment issues. Additionally, placing the indenter inside the SEM allows for direct observation of the material deformation during indentation or micro-compression. An x/y stage allows for nanometer-scale positioning of the indentation tip above any specific features or geometries of the material.

To perform high dynamic testing, the classical load sensor was removed and replaced with a dynamic load sensor, which is termed 'SmarTip' (patent pending). The new load sensor is composed of a piezo-tube which is fixed directly behind the diamond tip (Fig. 1b), allowing for high strain rate experiments to $1 \times 10^3\ s^{-1}$. Such high strain rate experiments are possible due to the small mass (~0.3 g) and low compliance ($\sim 3.9 \times 10^{-8}\ m{\cdot}N^{-1}$) of the SmarTip. Consequently, the SmarTip resonant frequency is around 60 kHz, which is two orders