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Analysis of failure modes under nano-impact fatigue of coatings via high-speed sampling

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ABSTRACT

Multiple nano-impact testing of coatings has recently been significantly exploited for fatigue testing of hard coatings. Using high-speed sampling, dynamic impact fatigue testing can provide significantly more information about fatigue deformation of coatings during cycling. Here, the additional analysis parameters provided by dynamic impact fatigue have been utilised to uniquely identify delamination (without fracture) and failure during cycling of a sol–gel coating on stainless steel. This has been used to map the evolution of fatigue damage as a function of impact velocity and cycles.

1. Introduction

The fatigue performance of thin films is highly relevant to a large number of technological applications. As such, a large number of techniques [1] have been developed for investigating fatigue deformation of thin films: thermal cycling of films on substrates, tensile testing of free-standing films and films on substrates, multi-cycle indentation, bulge testing, and microbeam bending. With the exception of dynamic microbeam bending [2–4], most of these test methods are limited to lower strain rates ($<10^{-2}\ s^{-1}$) and cycling rates.

Nano-impact indentation testing is a relatively novel technique which allows testing with extremely high stresses and high strain rates and cycling rates. Beake et al. [5–7] pioneered this technique for simulating repetitive impact loading, such as interrupted cutting for hard tool coatings. Testing of this type has been applied to evaluate the impact and fatigue resistance of a wide variety of coatings such as DLC and amorphous carbon [6,8,9], polymers [10], plasma electrolytic oxidation surface treatments [11,12], and numerous hard tool bit coatings [13–21]. Nano-impact indentation can also be applied at elevated temperatures [14,15,18] to evaluate coating fatigue performance at representative service temperatures.

Adhesive failure of the coating is typically observed in the data as a single rapid increase in penetration depth, corresponding to fracture and removal of the coating and penetration of the substrate. When failure is characterised by a series of small jumps in penetration depth this has been previously designated [6] as cohesive failure with delamination fractures. By testing over a wide range of impact accelerating loads and impact numbers a fatigue failure probability plot can be constructed using Weibull statistics [13].

Most investigations using this nano-impact indentation technique have lacked the temporal resolution to observe the displacement–time relationship during the initial stages of impact penetration. Instead, only the depth after the indenter had come to rest on the sample could be measured. High-speed sampling allows the entire mechanical response of the coating under impact to be observed. This has been used to investigate the energy dissipation and damping of polymers [22] and biological tissues [23] and to investigate the temperature-dependent superelastic response of NiTi at high strain rates [11]. By measuring the energy consumed during deformation, a dynamic hardness value can also be determined for high strain rate deformation [24–26]. The energy consumed during impact indentation has also been linked to extraction of fracture toughness [27].

Sol–gel methods are a particularly low-temperature route to preparing ceramics by chemical synthesis. The output product can be made in many forms: nanoparticles, monoliths, fibres and coatings [28] and in a wide range of ceramic materials. However, the most widely studied material is silicon dioxide (silica) due to its convenient chemistry. The production of coatings is of particular interest, since the thin film facilitates relatively simple curing and the low processing temperatures of sol–gel routes allow ceramic properties (e.g. hardness, corrosion resistance and good barriers to oxygen and moisture ingress)

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to be conferred on relatively low melting point metallic or polymeric substrates. Whilst producing a pure silica coating is a relatively straightforward process and, as a pure ceramic coating, offers the greatest hardness and the best diffusion barrier properties, in practice, its brittle ceramic nature restricts its uses. To increase flexibility, an additional silane component can be included during formulation of the coating solution to modify the network.

The silane would typically have one or more of its siloxane groups substituted by a direct silicon–carbon bond (with organic functionality beyond the carbon atom). These substituted groups are unable to participate in network formation, and additionally induce steric hindrance to full network formation. Consequently, addition of an organic component results in increased toughness in the coating, at the cost of a slight loss in overall ceramic character (e.g. reduced hardness, scratch resistance and barrier properties).

The presence of carbon groups can be exploited by selecting organic groups with particular functionality. An area of particular industrial interest is durable coatings with low surface energy, for use in applications including anti-fouling coatings for heat exchangers, aircraft wings and wind turbine blades. In the present work, the coating assessed included a fluorinated component which provides this function.

In this work, the advantages of using high-speed sampling are demonstrated for fatigue nano-impact testing, using a sol–gel coating on 316 L stainless steel as a case study. The additional parameters made available by dynamic multiple impact testing allow a type of instrumented erosion test to be performed, analogous to the way in which instrumentation of scratch testing enables an instrumented abrasion test to be undertaken.

2. Materials and methods

2.1. Sample production and characterisation

The sol–gel coating was produced at TWI Ltd. using a sol (ceramic precursor) consisting of 80% silica source, in this case tetraethyl orthosilicate (TEOS), and 20% silane network modifiers, acquired from Sigma-Aldrich Company Ltd. The TEOS and each silane were hydrolysed separately, then mixed using magnetic stirring. The sol was then allowed to mature, during which time the silaceous species underwent a degree of co-reaction, forming polymeric structures. The 316 L stainless steel substrate was cleaned with acetone prior to coating, and after cleaning, the substrate was handled only with powder-free gloves.

Prior to coating, sols were diluted with methanol as required. Substrates were manually dip coated at a withdrawal rate of approximately 20 mm/s. Coated parts were allowed to dry at 50 °C for a minimum of 10 min before transfer to an oven at 200 °C for 10 min to form a stable coating with a thickness of 940 nm. These conditions have been determined as described previously [29]. Coating thickness measurements were made with a Surfcom 130A profilometer. Optical microscopy of specimens after multiple impact was conducted using a calibrated Zeiss Axiolab optical microscope in reflected light mode fitted with a Motic Moticam 1000 digital camera.

2.2. Multiple impact

The MicroMaterials Ltd. NanoTest NTX series controller features high-speed datalogging capability in the 1–5 MHz range, so that displacement can be measured dynamically using capacitor plates positioned directly behind the indenter tip during impact events. The system consists of a stiff, ceramic pendulum which is balanced on a frictionless leaf spring pivot (Fig. 1). Force is applied using a permanent magnet and electromagnetic coil. During impact loading, the base of the pendulum is restrained using the impact solenoid while the accelerating load is applied at the force coil. When the impact solenoid current is released, the pendulum swings forward under the applied accelerating load. A range of incident velocities spanning nearly two orders of magnitude can be achieved by varying the acceleration load and spacing between the solenoid and pendulum (Fig. 2). The system is used in a single damping plate configuration, with the

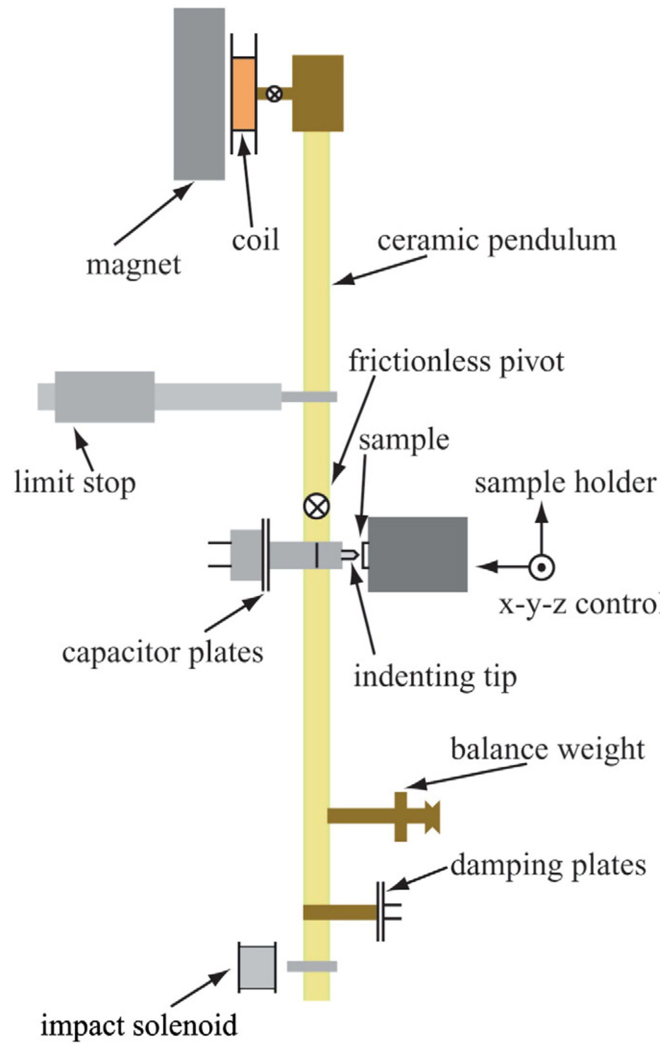

Fig. 1. Schematic of MicroMaterials Nanotest pendulum with impact solenoid after Muir Wood [30].

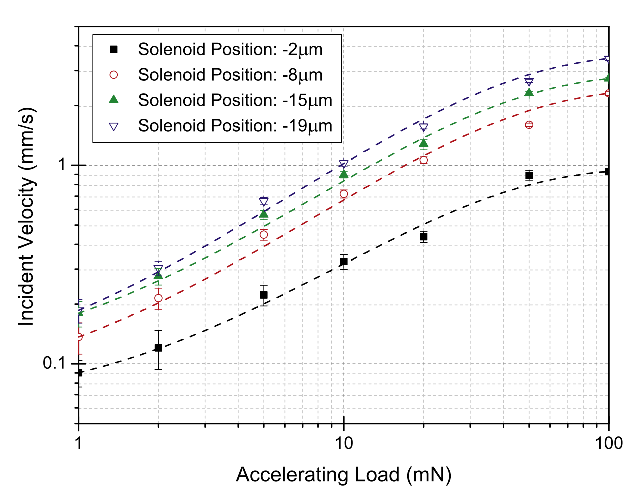

Fig. 2. Impact velocities prior to impact attainable using this system.