Received: 23 January 2023 | Revised: 22 February 2023 | Accepted: 16 April 2023
DOI: 10.1002/sia.7214

SHORT COMMUNICATION

SURFACE and INTERFACE ANALYSIS WILEY

A sample holder for the X-ray photoelectron spectroscopy analysis of multiple mini-traction machine ball samples

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Funding information
Engineering and Physical Sciences Research Council, Grant/Award Number: PR16195

A simple sample holder has been developed for the K-alpha and Nexsa range of X-ray photoelectron spectroscopy instruments that allows for the rapid throughput analysis of the ball sections of mini-traction machine, the analysis of which can be useful in understanding of, for example, surface scuffing and other phenomena in mini-traction machine instruments.

KEYWORDS
MTM, sample holder, tribology, XPS

1 | INTRODUCTION

Tribology studies wear, friction, and lubrication between surfaces, hence understanding the interaction of the two surfaces and the introduction of materials that can affect that interaction is of importance. The use of a mini-traction machine (MTM), schematically shown in Figure 1, in tribological studies is widespread, allowing the measurement of the frictional properties of both lubricated and unlubricated contacts under a range of sliding and rolling conditions and different lubricants (see, e.g., previous works[1–8]).

MTM ball and discs are generally fabricated using AISI 52100 steel, although chrome free versions (AISI 1015) and austenitic and martensitic steels are also available, and typically come in ¾″ ball on 46 mm discs (the most common, and used herein), ½″ ball on 32 mm discs (designed for higher contact pressures than the 1.25 GPa limit for the ¾″ ball) and a barrel on a 46 mm disc.

Given the inherent surface sensitivity of X-ray photoelectron spectroscopy (XPS), it is ideal to study a surface after a tribochemical reaction, resulting from the thermal and mechanical stresses involved in the wear process. An understanding of the chemical changes in the surfaces of these materials is paramount in the elucidation of whether a tribochemical reaction has, for example, a beneficial[9] or deleterious effect[10] or an idea of the elemental distribution from depth profiling experiments.[8]

It is common for XPS analysis to be performed solely on the MTM disc themselves, and despite their size can fit in to most modern XPS spectrometers if mounted correctly (Figure 2). However, there is an interest also in the corresponding wear tracks of the ball and how the surface chemistry of that component may be different from that of the disc or to investigate 'scuffing' of the lubricant-metal system.[11]

With the advent of coincident techniques, such as Raman (allowing phase identification) and ion-scattering spectroscopy (ISS)[12,13] and depth profiling using monoatomic ions or argon clusters,[12,14] significantly more chemical information can be extracted from a single sample.

2 | DESIGN OF THE NEW HOLDER

Wear tracks on both disc and ball are typically narrow and consequently small-area analysis is required within the central area of the wear track to avoid potential artefacts from uneven edges. As seen in Figure 2, the mounting of the discs is straight forward, and hence alignment for analysis can be relatively facile depending on camera geometry or using XPS imaging to determine the area for analysis.[15]

Analysis of the balls is more difficult. No longer do we have a flat surface for mounting, but instead a sphere, typically coated in a layer of lubricant, which can make adhesion difficult. To circumvent this, we developed a holder that would not only remove the need for the

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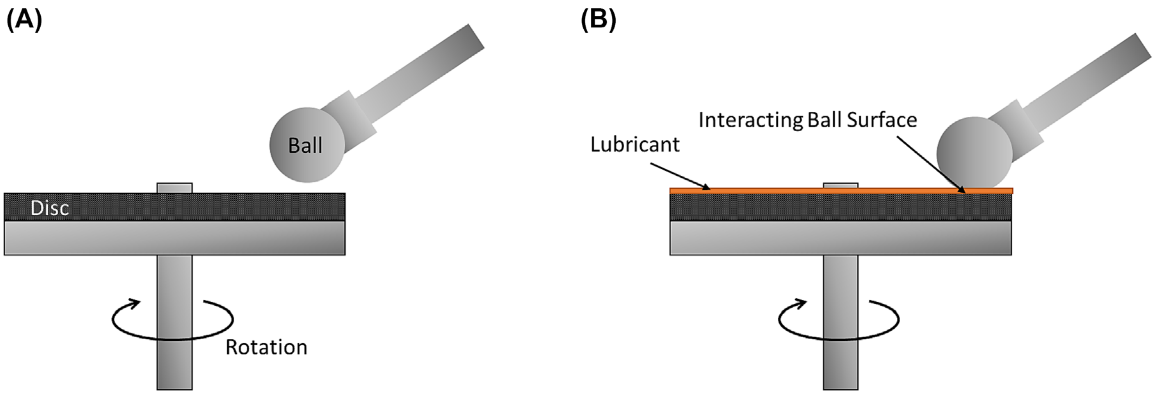

FIGURE 1 Schematic of the (A) ball and disc setup and (B) interaction of ball and disc with a lubricating layer within a mini-traction machine (MTM).

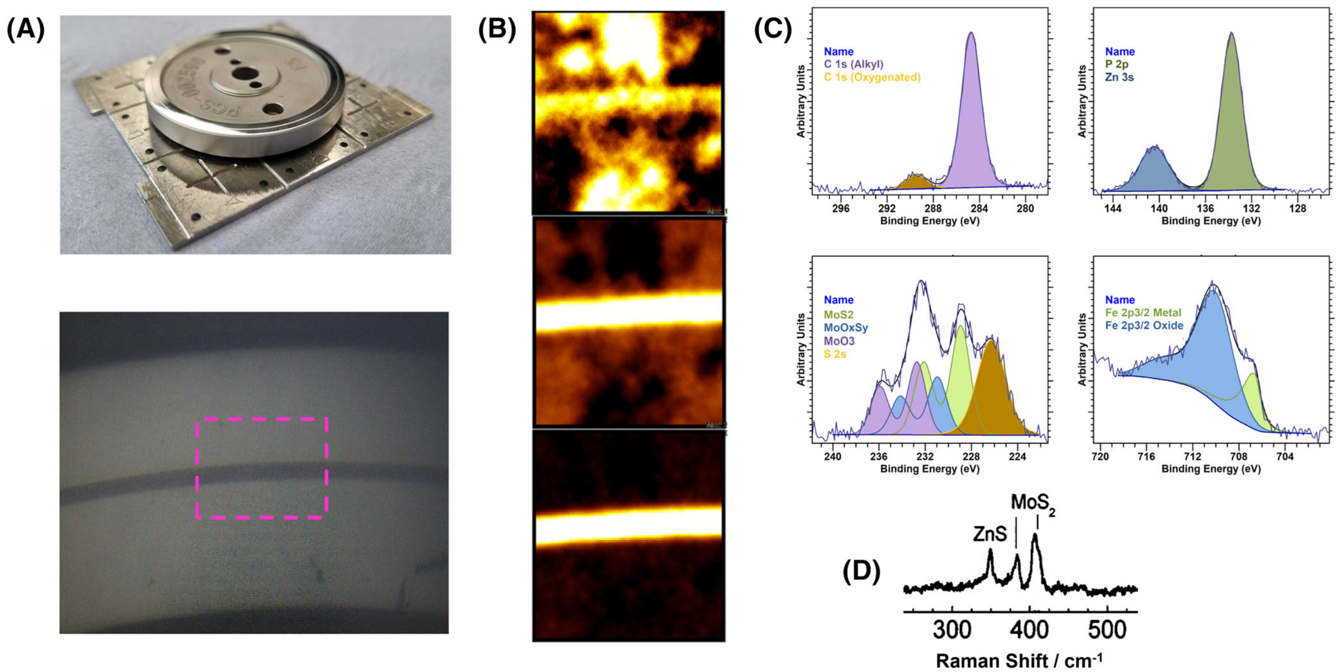

FIGURE 2 (A) Typical mounting of an MTM disc for analysis of a wear track and internal image of the wear track with examples of (B) XPS imaging, (C) snapshot XPS data from a depth profile, and (D) in-situ Raman analysis of such a wear track.

balls to be (precariously) held with tape but give a method of mounting, which would also allow a fixed alignment of the wear track on the ball with the geometry of the X-ray spot, or other coincident techniques.[12]

The rationale for the current design of the holder is twofold; the first is to enable a higher throughput of samples for analysis and secondly for the ease of positioning and lack of adhesive tape as already stated. The holder has been designed for ThermoFisher Scientific K-alpha and Nexsa systems, which utilise the same sample holder design, although simple modification can be made for inclusion into similarly designed sample platters.

The evolution of the design can be seen in Figure 3. A proof-of-concept and successful prototype was formed using strips of nickel foil, together with polythene stoppers taken from common laboratory 25 mm diameter, flat bottomed glass sample tubes, the tops of which were cut off. The polythene rings were secured using smaller strips of nickel spot welded to the larger strip, which also had the advantage of acting like springs to allow for a tighter fit of the balls in the polythene rings. This prototype holder could be held down with double-sided carbon tape or clamped using clips, which can be inserted into the base of the ThermoFisher Scientific holder.

The final version of the sample holder was printed on an Ender 3 Pro 3D printer, using a polylactic acid (PLA+) filament source. As shown in Figure 3B, the holder is printed with recesses for grub screws, which have their thread tapped later, and for the insertion of copper pads to allow conduction to the base of the holder. Whilst this is not critical, and charge compensation can easily aid analysis of electrically isolated samples, care must be taken with both micro-focussed

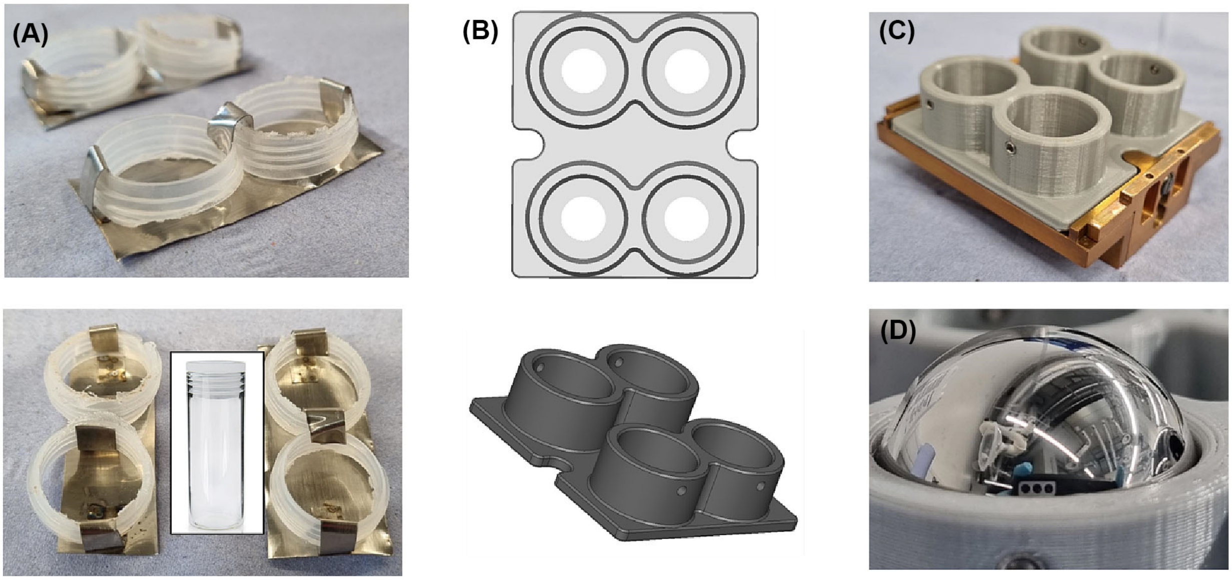

FIGURE 3 (A) Prototype of the holder (bottom insert: example of glass vial used to source the polymer top), (B) CAD-style projections of the holder for printing, (C) final 3D-printed version of the sample holder and (D) close-up of a ¾″ ball in the holder where the wear track is visible and aligned to the most vertical point of the holder.

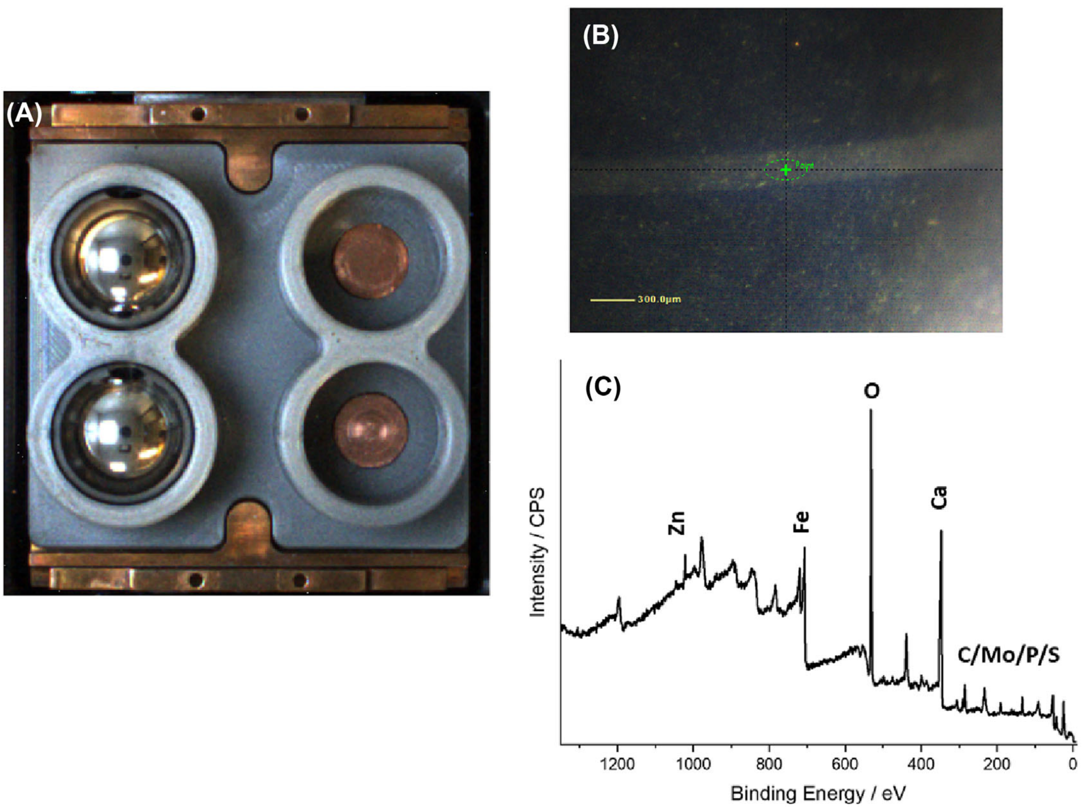

FIGURE 4 (A) Holder pictured using the system load-lock camera, (B) in-analysis chamber camera view of the sample at the correct analysis height, and (C) survey spectrum taken from the wear track.

sources and charge compensation systems as they could induce changes in chemistry.[16–18]

As PLA for 3D-printing can contain additives, especially if coloured, the holder was thoroughly rinsed in deionised water and placed in a dedicated vacuum chamber to outgas for 24 hours. After rough vacuum and turbomolecular pumping for 30 minutes, the outgassing vacuum chamber was ca. 5×10^{-5} mbar, whilst after 24 hours, the chamber pressure was in the low 10^{-8} mbar regime.

3 | OPERATION OF THE NEW HOLDER

The cleaned and out-gassed holder has been tested for transfer and analysis using a differing number of steel balls. Figure 4A shows one of these arrangements, with two balls loaded for analysis. In the same figure, panel (B) shows the in-system camera for the analysis position, together with the X-ray spot define analysis area, with (C) a wide scan spectrum recorded from the highlighted analysis area using the 200-μm spot mode at a pass energy of 150 eV and a step size of 1 eV. The total acquisition time for the survey spectra was ca. 3 min.

Close inspection of Figure 4A shows line of the wear track, which allows for easy alignment in the holder. This allows for ease of alignment of the wear tack for analysis using the internal optical system of the spectrometer and hence minimising any artefacts from an off centre or angled analysis point.

4 | SUMMARY

We have presented a 3D-printed sample holder for the analysis of the balls from a MTM set-up. Capable of accommodating a maximum of four balls in a single pump-down cycle, the holder has proven to be a simple, low cost and effective holder to maximise throughput of sample, especially for depth profiling experiments. We include as supplementary information, computer-aided design (CAD) format files for those wishing to recreate or adapt this holder.

ACKNOWLEDGEMENTS

The Engineering and Physical Sciences Research Council are acknowledged for their support of the National Research Facility for Photoelectron spectroscopy (HarwellXPS), which is operated by Cardiff University and UCL, under contract no. PR16195. Cardiff University are signatories of the Technician Commitment, and the input, advice, and expertise of technicians from the Cardiff School of Chemistry (Mark Reece and Steven Morris) is greatly acknowledged in this paper.

DATA AVAILABILITY STATEMENT

Computer Aided Design (CAD) data is available as supplemental material published with this paper. Inclusion of some XPS data was granted by third-parties, however due to the commercial nature of some datasets from which exemplar spectra were taken the data are not shared.

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How to cite this article: Morgan DJ. A sample holder for the X-ray photoelectron spectroscopy analysis of multiple mini-traction machine ball samples. *Surf Interface Anal*. 2023;1-4. doi:10.1002/sia.7214