CSM Instruments
Advanced Mechanical Surface Testing

# APPLICATIONS BULLETIN

# Special Issue: Biomedical Applications

## Micro-mechanical properties of calcium phosphate coatings for titanium prostheses

Mineralised tissue, such as bone and teeth, are composed of inorganic and organic components which are in intimate association with each other. The latter, and particularly proteins, serve not only to strengthen these tissues but also to stabilise their mineral contents. This is one of the main driving forces for coating metal implants with calcium phosphate layers which improve their biocompatibility and osteoconductive properties.

In a previous investigation [1] it has been demonstrated that when Bovine Serum Albumin (BSA) is biomimetically coprecipitated with $Ca^{2+}$ and $PO_4^{3-}$ ions upon titanium alloy implants, it becomes incorporated into the crystal lattice and is not merely deposited on the surface. The BSA elicits a concentration-dependent change in the crystal structure of the coating (from an octacalcium phosphate type to a carbonated apatite one) and a decrease in its crystallinity. BSA-containing coatings thus bear a closer resemblance to natural bone mineral than do non-BSA-containing ones.

Although calcium phosphate coatings can easily be characterised by conventional means (FTIR spectroscopy, X-ray diffraction, imunohistochemical evaluation, etc.), the mechanical properties are not so easy to obtain. The objective of this study was to investigate the correlation between BSA content and mechanical strength in a standard calcium phosphate coating. The dual-purpose Micro Combi Tester was used to evaluate the scratch resistance and microhardness of seven different coatings having BSA concentrations over the range 0.01 µg/ml to 1 mg/ml. Fig. 1 shows the almost linear correlation between BSA content

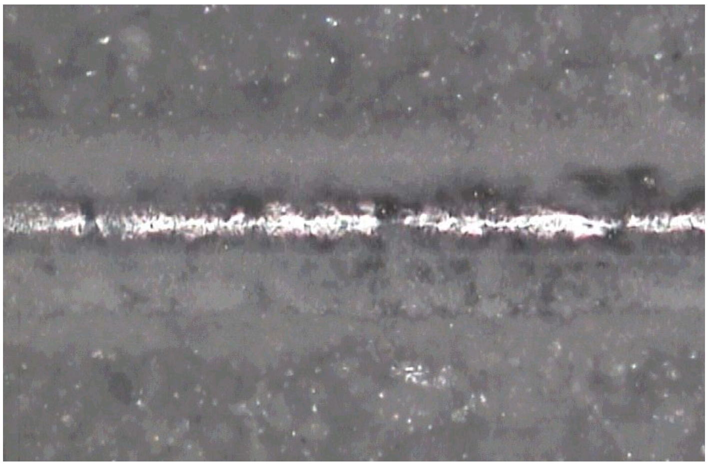


**Figure 2:** Typical scratch track on a BSA-containing calcium phosphate coating after a progressive load scratch up to 5 N with a 200 µm diamond indenter.

Some deviation between successive scratches was observed due to the high porosity and surface roughness of the coatings and because of the mismatch between the very soft coating and the much harder Ti substrate. An example of a typical scratch is shown in Fig. 2 where the substrate has been reached. Microhardness results (Fig. 3), however, showed increased hardness up to a BSA concentration of 0.1 µg/ml followed by a sharp decrease at 1 µg/ml.

Maria Yuelian Liu of the Biomaterials Research Group in Leiden University is acknowledged for providing the presented results.

[1] Y. Liu, P. Layrolle, J. de Bruijn, C. van Blitterswijk and K. de Groot, Biomimetic co-precipitation of calcium phosphate and bovine serum albumin on titanium alloy, J. Biomed. Mater. Res., 57 (2001) 327 - 335

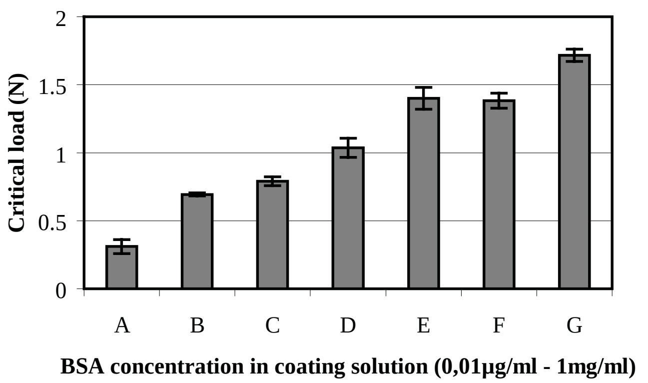



**Figure 1:** Micro-scratch test critical loads as a function of different BSA concentrations in the calcium phosphate coating. The general trend shows increased scratch resistance with BSA content.

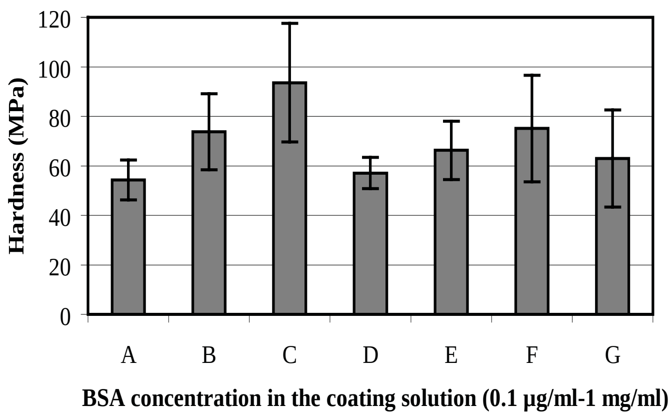
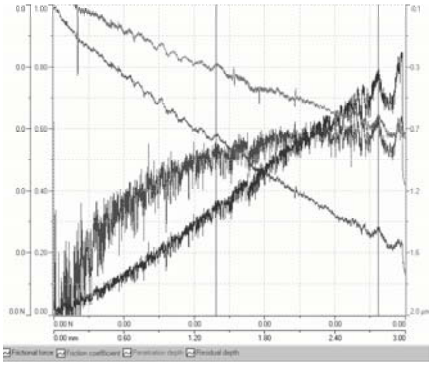
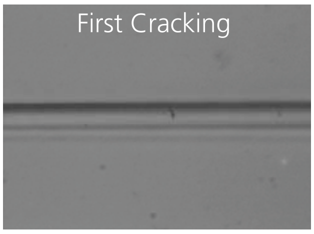


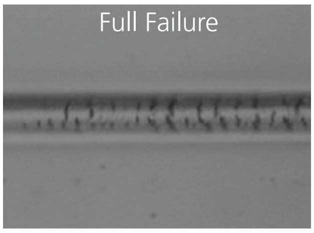

**Figure 3:** Microhardness results as a function of different BSA concentrations in the calcium phosphate coating. Measurements were made with a Vickers indenter and a maximum applied load of 1 N.

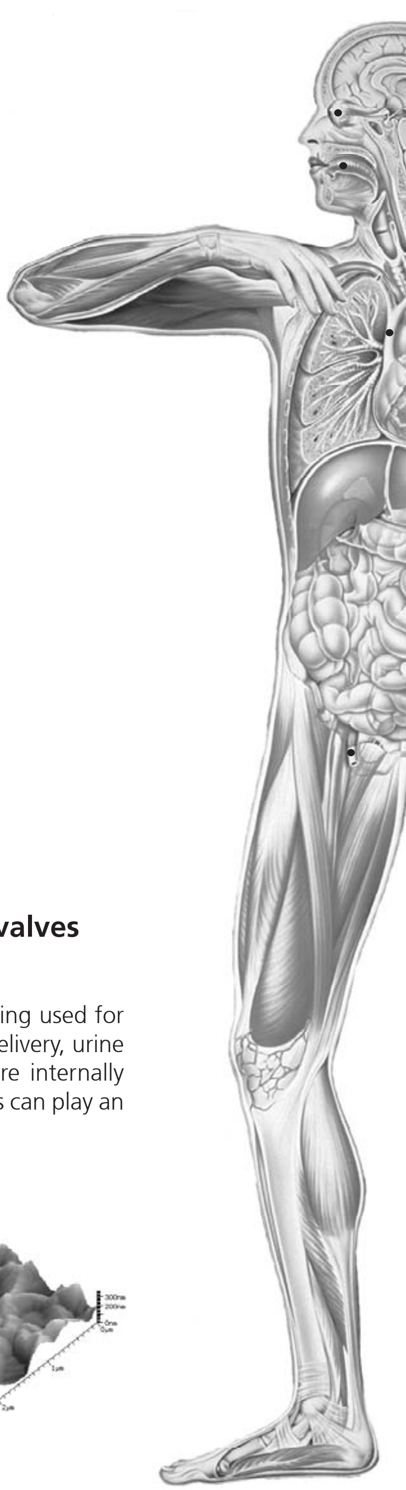


## Adhesion of contact lens coatings

Contact lens technology has seen many advances in recent years. One of these is intra-ocular implantation of the lens between the cornea and the iris. The soft lens is injected into a small incision and then spread out under the iris. Coatings are used to ensure sufficient biocompatibility with the contacting tissues and can be easily characterised with the Nano Scratch Tester using very low loads (< 50 mN). The soft lens is flattened on a plate with a drop of water which keeps it flat during the test (by capillary forces) and ensures that it remains sufficiently humidified. The force feedback of the instrument follows any curvature of the surface.

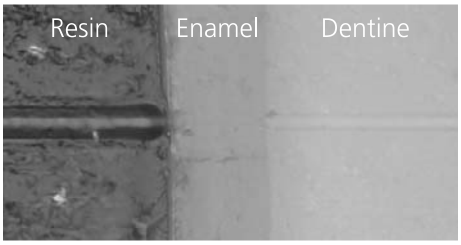


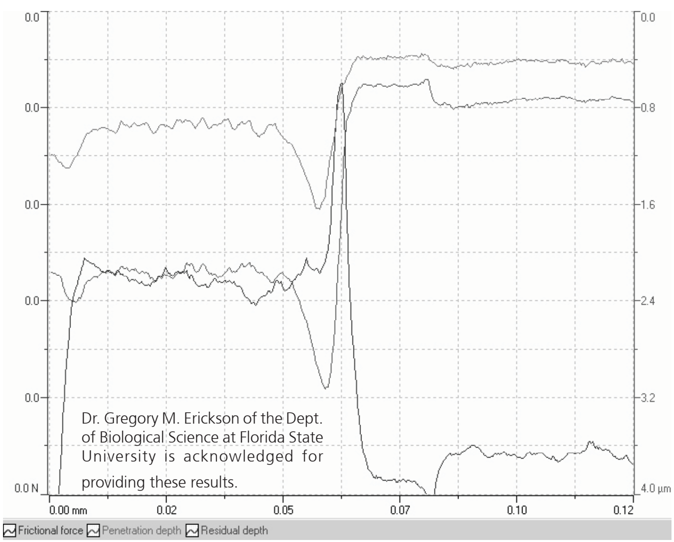



## Mechanical properties of teeth materials

The tooth is a craniofacial structure that is the focus of intensive tissue engineering studies. The outer enamel layer is almost 95% mineral, the hardest structure in the human body. During formation, enamel consists of a protein matrix that forms the framework for mineral deposition. If the matrix proteins can be identified and cloned then such knowledge can be used to replicate the natural enamel-forming process. The nano scratch test has been used to determine mechanical properties of polished tooth sections as shown below. A constant load scratch (5 mN) is shown across the interface between the dentine, enamel and mounting resin. The differences in hardness are evident from the depth plots and it is interesting to note that variations in friction coefficient between the enamel and dentine are also well characterised.

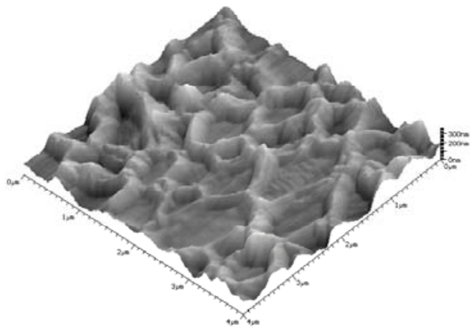


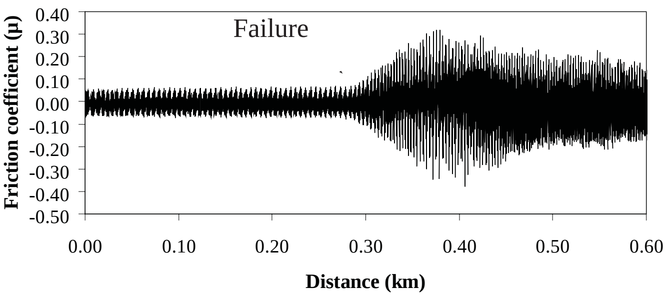

Dr. Gregory M. Erickson of the Dept. of Biological Science at Florida State University is acknowledged for providing these results.

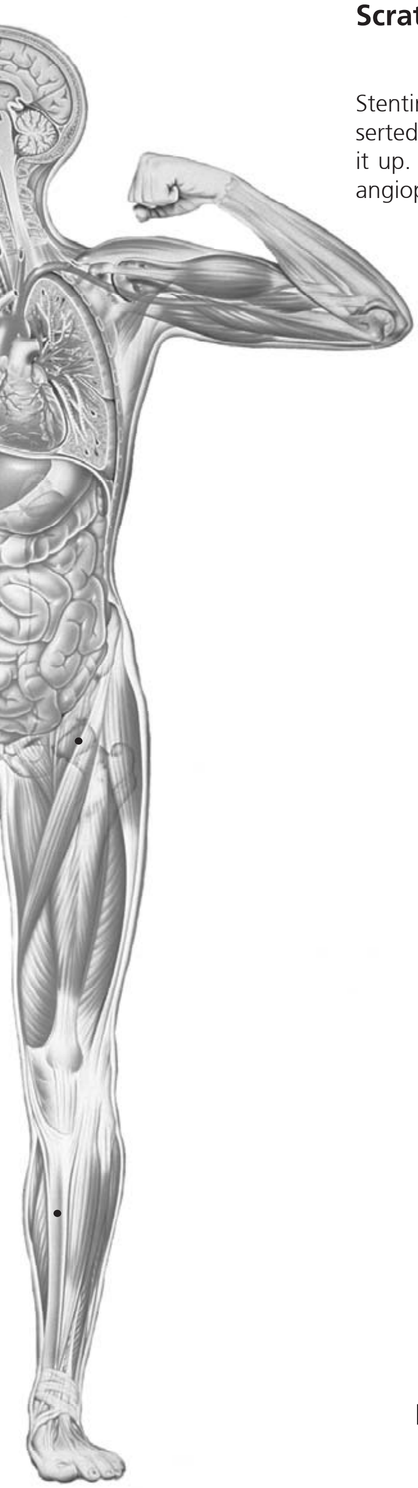
## Controlled drug release valves

Biocompatible valves are now being used for many applications, e.g., insulin delivery, urine control, etc. Such tiny devices are internally coated and the surface roughness can play an important role in the fluid flow.

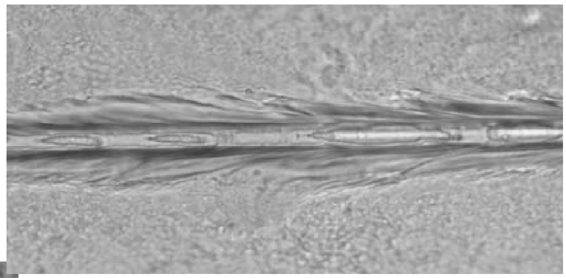


Scanning Force Microscopy (SFM) is used to characterise the surface roughness (3D image shown above) of the coating and the Linear Tribometer can be made to simulate opening and closing of the valve. Coating failure can be detected by plotting frictional variations as shown in the example below.

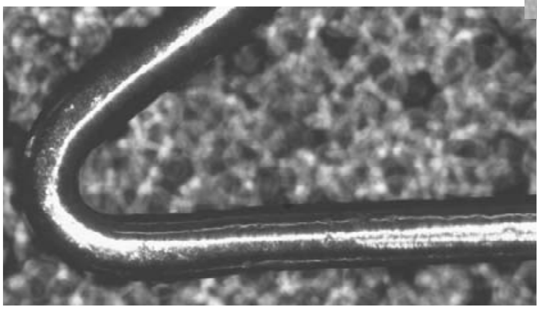




## Scratch resistance of arterial implants



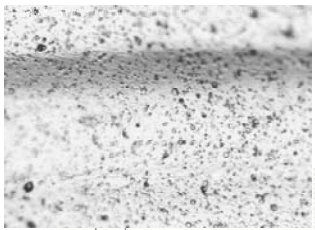
Stenting is a procedure in which a wire mesh tube is inserted through a catheter and placed in an artery to hold it up. Stenting is usually performed right after a balloon angioplasty, while the catheter is still in place.

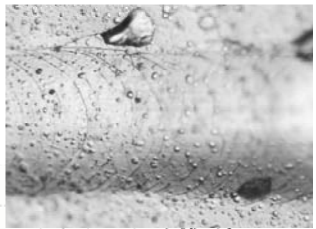


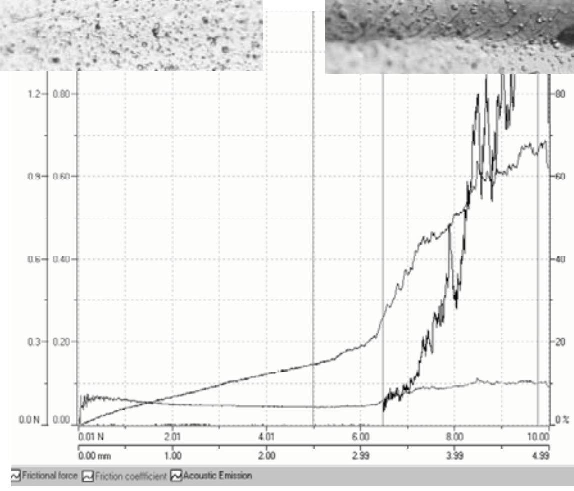
The surface properties of a typical wire mesh stent are complex as it is usually covered with a coating which has a protective function, but which can also be implanted with various drugs which will help the artery wall to heal properly. The Nano Scratch Tester has been used to successfully characterise the stent coating by making scratches along single strands of the mesh structure. This provides useful data concerning adhesion and friction.

## TiN coating for hip prostheses

Different hard coatings such as TiN have already been used for some time for wear resistance in prosthetic implants. Quality control of such materials can be performed with the Micro Scratch Tester, even on curved surfaces such as the ball of a ball-and-socket joint. The instrument force feedback ensures that the applied load remains as-programmed for the duration of the test. Frictional data is also of importance and can be related to the in-vivo application.



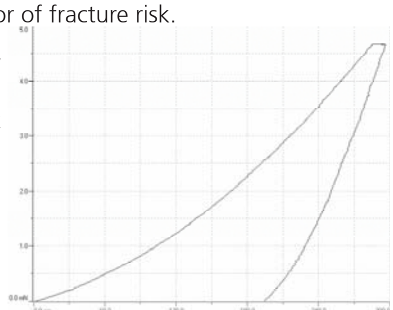





## Hardness of bone tissue

The inorganic component of bone makes the tissue hard and rigid whereas the organic component gives bone its flexibility and resiliance. The strength of a bone is directly related to the distribution of mineral within it and can be measured with the Micro Hardness Tester. The resulting hardness values at different points across a polished bone section give a measure of Bone Mineral Density (BMD) which is an accurate indicator of fracture risk.

Such results can subsequently be correlated with collagen content which suggests that the collagen reinforces the bone tissue. Other studies have also been performed on artificial bone materials to investigate whether these are suitable for in-vivo implantation.



## Characterisation of Tin Oxide coatings for biomedical applications

Tin oxide coatings are presently being developed for applications within the body where good adhesion and biocompatibility are the main considerations. Such coatings are spin-coated onto a standard stainless steel (316L) substrate in a way that the coating thickness can be accurately controlled. Mechanical properties are characterised by nanoindentation and pin-on-disk tribology, the latter utilising the liquid heating option on the standard Tribometer. This optional device means that the sample can be completely immersed in a solution which simulates actual body fluids and the temperature of the pin-sample contact can be maintained at 37°C for the duration of the test.

A typical result is shown in Fig. 1 (b) for a tin oxide coating in contact with a 100Cr6 ball. The applied load was 2 N, linear speed 10 cm/sec. and wear radius 3 mm.

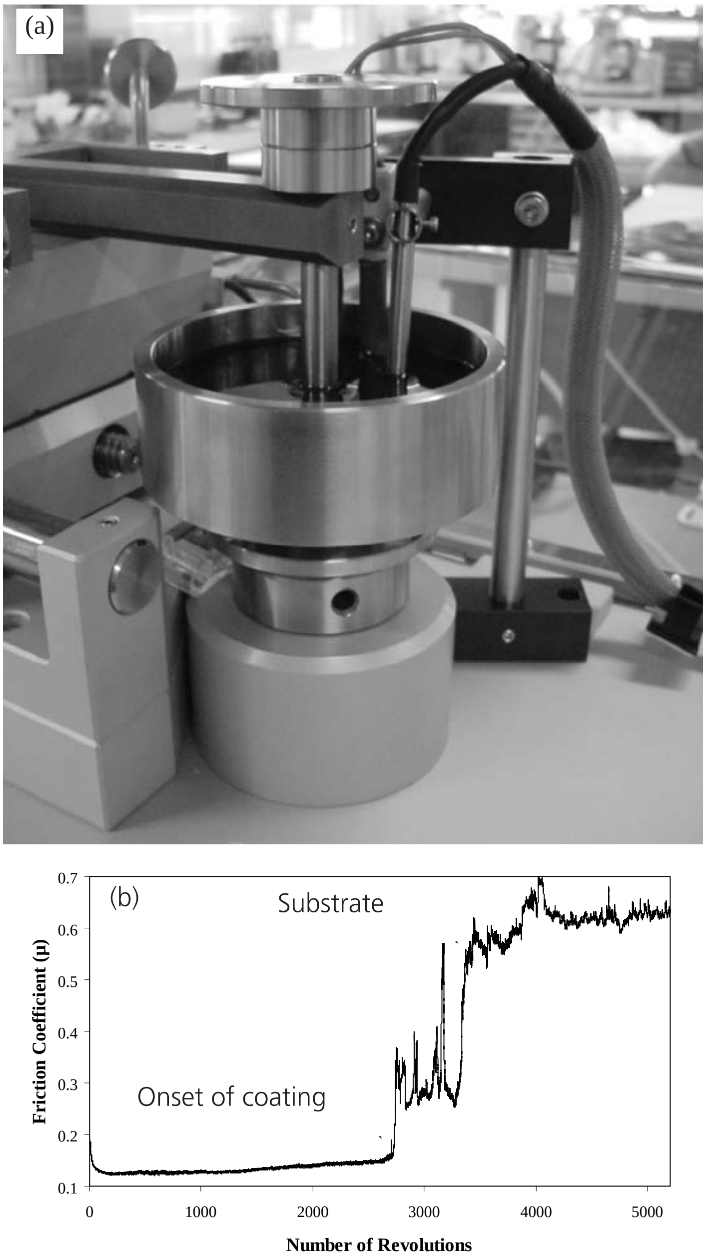
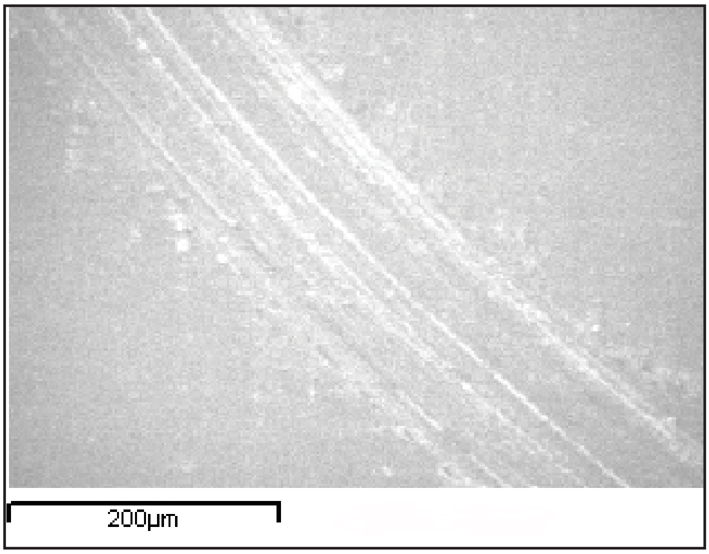



**Figure 1:** Liquid heating option mounted on the standard Tribometer (a) and typical result (b) of friction coefficient plotted as a function of number of revolutions for a tin oxide coating spin-coated onto a stainless steel substrate.




This result shows the initial running-in period, after which the friction coefficient stabilises at a value of approximately 0.13 before increasing gradually until first failure of the coating occurs. The coating then breaks down and when the substrate is reached, the friction coefficient stabilises at a value of about 0.64. The test was paused at significant points to allow SEM imaging of the wear track (see Fig. 2) which confirmed the state of failure of the coating

Niamh Rushe, Sinead Healy, Prof. Des Cunningham, Dr. Liam Carroll and coworkers from the National Centre for Biomedical Engineering Science and the Dept. of Chemistry of the National University of Ireland, Galway, are acknowledged for providing the results shown.

CSM Instruments

This Applications Bulletin is published quarterly and features interesting studies, new developments and other applications for our full range of mechanical surface testing instruments.

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