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Augmented multi-scale instrumented indentation test characterization of complex multi-layered coatings for tribological application

Giacomo Maculotti[1,a *], Gianfranco Genta[1,b] and Maurizio Galetto[1,c]

[1]Department of Management and Production Engineering, Politecnico di Torino, Corso Duca degli Abruzzi 24, 10129 Turin, Italy

[a]giacomo.maculotti@polito.it, [b]gianfranco.genta@polito.it, [c]maurizio.galetto@polito.it

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Abstract. Multi-layer coatings for steel bushings consisting of an innermost layer of sintered bronze and an outermost composite layer of lead-reinforced polytetrafluoroethylene (PTFE+Pb) have been used in several power transmission applications. The PTFE+Pb layer provides lubrication by material transfer on the counter-body reducing friction and smoothing motion. The mechanical characterization of such a complex system is challenging and essential to provide input data necessary to design and predict the service life of the components. This work innovatively mechanically characterizes the coating by augmented multi-scale Instrumented Indentation Test (IIT). Nano-IIT will evaluate the uniformity of the Pb particles' dispersion. Dynamic nano-IIT will investigate the damping properties of the material as a function of load frequency. Micro-IIT will tackle the layer thickness evaluation and the gradient of mechanical properties through the layers, by continuous multi-cycle and by data augmentation provided by electric contact resistance.

Introduction

Metal-polymer coatings exploiting polytetrafluoroethylene (PTFE)-based materials have high lubricity and are an efficient alternative to conventional low-friction metallic materials, e.g., bronze and lead, for sliding bearing elements. In fact, these coatings rely on the superior tribological performance of PTFE, which has a friction coefficient lower than traditional materials. Several power transmission and automation applications that need gentle driving of sliding and rotating mechanical components under moderate load with very low frictional losses have adopted metal-polymer PTFE-based coatings. Due to its self-lubricating properties, PTFE can support the interfacial sliding motion and bear contact loads even without adding any lubricating material. This is crucial for applications in controlled environments where a spill of fat contaminants must be prevented at all costs, such as surgery machines and the food industry [1].

Using the symbiotic tribological mechanism between PTFE and a low-friction tin-bronze porous structure, metal-polymer composite coatings with reinforcing low-friction metal structure have proven to be the most successful solution for the specific application to plain bearings and bushings. Such a combination takes advantage of the polytetrafluorethylene's self-lubricating qualities and the tin-bronze's mechanical properties, which bears some of the contact load preventing the PTFE from wearing out too soon. Metal-polymer coatings are usually manufactured as strips of steel sheet reinforcement on which the porous bronze matrix is sintered with a thickness of (0.2∼0.4) mm. The manufacturing of guide bushings for linear pneumatic actuators is among the uses for these composite coatings that are of interest. Their incorporation with servo systems for highly automated Industry 4.0 production lines is a vital feature that has lately allowed them to broaden their area of use.

The literature reports some research works where component testing was carried out to investigate the wear process of the guide bushings of pneumatic actuators [2]. However, despite the wide application of this class of coatings, a thorough characterization of the mechanical behaviour of PTFE-based metal-polymer composite coatings with reinforcing low-friction metal structures is still missing. Some research papers have been published on this topic, but only application-oriented tribological results were presented [3,4].

The objective of this work is to thoroughly characterize the surface mechanical characteristics of the PTFE/Sintered Bronze composite coatings for simple bearings and bushings. Instrumented Indentation Tests (IIT) will be exploited to characterize the mechanical response of the material, which, to the best of the author's knowledge, is unreported and will complement tribological characterization already available in the literature. Innovatively, in-situ electric contact resistance measurement (ECR) will augment the mechanical characterization to highlight the relationship between the electromechanical response and the material structure, which can be henceforth non-destructively investigated. With a multi-scale methodology, the characterization will also address the material's elastic and viscoelastic properties. The paper introduces a methodology to characterize multi-layer composite coating. The methodology is tested on a commercial grade PTFE+Pb/Bronze coating.

Materials and Methods

This work considers a commercial grade of PTFE+Pb/Bronze composite coating manufactured by GGB Bearing Technology Inc (Thorofare, New Jersey, USA). The nominal composition of the coating presents a steel lamina as backing, coated with a low-friction lining consisting of two superimposed layers. The innermost layer (nominally of 280 μm) is a sintered porous bronze structure impregnated with a polymer compound of PTFE filled with Pb. The outermost layer is a 20 μm thick lining out of the same PTFE+Pb compound (see Figure 1). While the PTFE-based polymer composite offers self-lubrication, the porous bronze structure reinforces the metal-polymer coating, ultimately extending the service life. In fact, during early operation, running-in occurs, and part of the PTFE-based material is transferred to the opposite surfaces, forming a protective third layer that insulates the metallic counterpart from direct contact with the coating.

The thickness of the layers has been measured with a commercial optical metallographic microscope, taking 15 images and, per each of them, measuring the layers' thickness by manual marking, as in Figure 1. Results in terms of average and measurement uncertainty are reported showing a PTFE+Pb thickness of (50±20) μm and for the Sintered porous Bronze of (278±10) μm. Measurement uncertainty has been evaluated at a 95% confidence level, combining the resolution and the reproducibility according to the law of uncertainty propagation [5]. The resolution of the objective pixel size (0.5 μm) was propagated with a uniform resolution as a type B contribution. The reproducibility was estimated from the empirical standard deviation as a type A contribution. Surface roughness was measured on ten different locations with a white light interferometer, showing Sa of (0.95±0.224) μm, Sq (1.47±0.32) μm, and Sz of (37.38±23.88) μm, propagating the sole contribution of reproducibility at 95% confidence level.

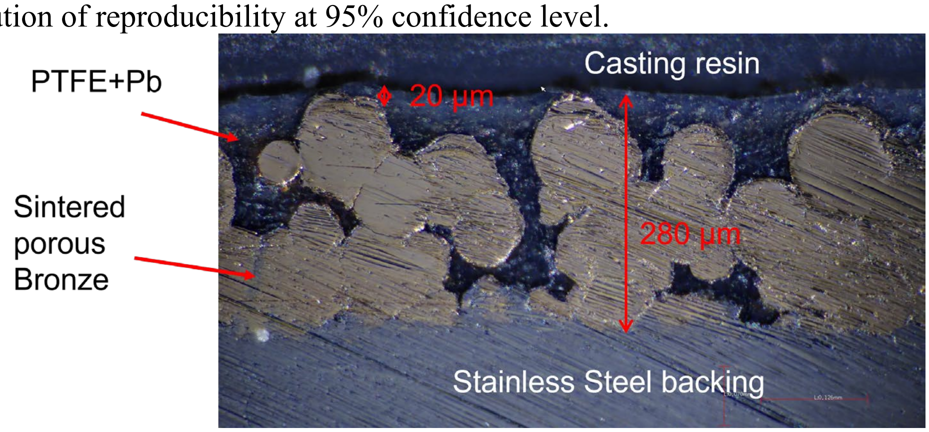

Figure 1 Section view of the DU® coating from GGB Inc.

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Instrumented Indentation Test for mechanical characterization. Instrumented Indentation Test (IIT) is a depth-sensing nonconventional hardness measurement technique that can characterize materials in terms of indentation hardness H_{IT}, Young modulus estimate, i.e. the indentation modulus E_{IT}, creep and relaxation from macro- to nano-scale. IIT applies a loading-holding-unloading force-controlled cycle with an indenter of known and calibrated shape to a test specimen [6]. During the indentation cycle, the applied force F and indenter penetration depth h in the material are measured, and the analysis of the measured quantities allows characterization relying on fundamental equations which are standardized [7]. IIT allows characterizing composite materials [8], coating [9] and surface treatments [10], while distinguishing and quantitatively characterizing different phases and structure variations of materials [6].

In-situ electrical contact resistance (ECR) augments IIT to evaluate the electromechanical response, highlighting differences in the microstructure due to a different electrical response [11], and estimating critical stress states for material phase transformation [12]. ECR relies on a doped-diamond conductive indenter and applies a controlled current at the contact between the indenter and the test sample surface. While the indentation cycle is performed, the voltage is measured, and changes in the contact resistance are appreciated, allowing electromechanical characterization [13,14].

An IIT variation, namely, continuous multi cycle (CMC) applies in the same location several indentations with increasing maximum test force [15]. CMC allows characterizing material properties in depth: it determines the mechanical properties as a function of the (increasing) maximum penetration depth $h_{c,max}$, for each successive unloading, resulting in a mechanical characterization, which can be related to the maximum penetration depth.

More recently, dynamic indentations have been implemented, by superimposing a sinusoidal trend to a conventional indentation cycle. Dynamic indentation allows evaluating viscoelastic properties of the material in terms of the loss modulus $E^{''}$ and the storage modulus $E^{'}$, whose evaluation is essential to provide a thorough evaluation of polymeric materials also apt for damping [16,17]. Instrumented indentation test will be performed with an Anton Paar STeP6 platform equipped with the measuring heads NHT^3 and MCT^3 hosted in the metrological room at the MInd4Lab laboratory of the Politecnico di Torino.

PTFE+Pb characterization. The layer of PTFE+Pb was mechanically characterized by Anton Paar NHT^3. Quasistatic indentation cycles (loading and unloading of 30 s, holding of 10 s) at 10 mN mapped an equally spaced grid of 7×7 indentions with a step of 90 µm. Characterization is reported in terms of H_{IT} and E_{IT} and investigates the homogeneity of the PTFE+Pb layer.

The viscoelastic properties of the PTFE+Pb layer are obtained by dynamic indentation through a set of 15 indentions (distance of 90 µm) at a maximum load of 10 mN. A typical oscillation frequency for linear pneumatic actuators applications of 2 Hz is considered to superimpose the sinusoidal trend on the holding part of the indentation cycle with the amplitude of oscillation set at 5% of the maximum load [16,17]. The replication of tests on bulk PTFE aims at comparing the response with respect to the base material.

Additionally, a full factorial design with 15 replications and factors the material (bulk base material, i.e. PTFE, and reinforced PTFE+Pb) and loading frequency, i.e. (1, 2, 3, 5) Hz, (for 120 tests overall) was deployed to test the factors' effect on the mechanical response.

Micro-scale characterization. Micro-IIT is considered to evaluate the material's mechanical response as a function of penetration depth by CMC, which avoids cross-sectioning the sample. Data augmentation by ECR of micro-IIT is performed to obtain insights on the composition of the multi-layer coating through the electromechanical characterization. In fact, the PTFE+Pb has a resistivity greater than the sintered bronze layer. A set of 36 CMC indentations were performed with Anton Paar MCT^3, each featuring 15 cycles with a quadratic increase of the maximum load from 0.5 N to 30 N and with the loading, holding and unloading each lasting 30 s. A further set of

18 CMC indentations ranging from 0.01 N to 0.5 N (other parameters were left the same) was performed to get data at shallow depths. ECR was set up in controlled current at 10 mA with a maximum voltage of 6 V to avoid sparks.

Results and Discussion

PTFE+Pb characterization. Nanoindentation resulted in mapping shown in Figure 2, in terms of E_{IT}. Both the indentation modulus and indentation hardness did not show significant deviations from a normal distribution, according to the Anderson-Darling test, thus supporting the hypothesis of homogeneous PTFE+Pb composition. The higher precision of the E_{IT} identifies outliers (see Figure 2), which can be ascribed to surfacing bronze particles (see Figure 1).

Viscoelastic response of the material was performed, and Table 1 summarizes the characterization results, reporting expanded uncertainty with a confidence level of 95%. Hypothesis tests on the average mechanical characterization, at a confidence level of 95%, show that the Pb fine powder stiffens the base material, e.g. induces an increase of the Young modulus.

ANOVA on the full factorial design showed, with a risk of error of 5%, that both the material and the loading frequency introduce a systematic effect on the response. In particular, an increase is appreciated due to the PTFE+Pb, with respect to base material, and at increasing frequency, this being an apparent effect due to the reduced time for elastic recovery of the material.

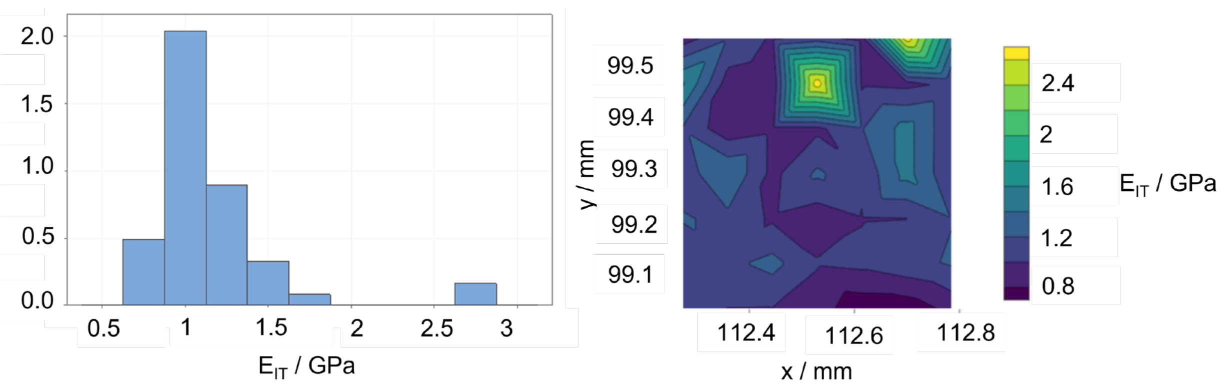

Figure 2 Histogram and spatial mapping of PTFE+Pb E_{IT}.

Table 1 Nano-IIT mechanical characterization in terms of Indentation Modulus E_{IT}, storage modulus E', loss modulus E'' and indentation hardness H_{IT}. Notice the increase of mechanical properties induced by the Pb reinforcement.

Material	E_{IT} / GPa	E' / GPa	E'' / GPa	H_{IT} / MPa
PTFE+Pb	1.1±0.07	2.1±1	0.15±0.01	46.2±2.4
PTFE	0.7±0.05	0.8±0.2	0.10±0.02	40.4±1.9

Micro-scale characterization. The experimental plan based on CMC indentations aimed at characterizing the gradient of mechanical properties as a function of depth was implemented and Figure 3(a-b) shows the trend of the mechanical characterization in terms of E_{IT} and H_{IT}, respectively. Consistently with the material structure appreciated with optical microscopy (see Figure 1), the mechanical response presents a trend depending on the indenter penetration. Specifically, an increase of the mechanical response is measured when moving from the PTFE+Pb to the sintered porous bronze, with a gradient in between.

The data augmentation through ECR provides insights. In fact, the transition can be seen in Figure 3(a-b) onsetting at 10 μm and terminating at 50 μm from the surface. In particular, ECR highlights a highly insulating material up to 20 μm, i.e. the PTFE+Pb. Electrical resistance decreases, as shown in Figure 3(c), at deeper penetrations up to 50 μm, where both electrical resistance and mechanical response tend to constant values, indicating the end of the transition to the sintered porous bronze. Furthermore, Figure 3(c) shows a highly insulating material, i.e. PTFE+Pb, even farther from the surface, e.g. values of 600 Ω at depths greater than 40 µm. This is in agreement with the optical analysis and suggests the presence of sacks of PTFE+Pb in the bronze matrix, consistently with the manufacturing procedure of the material. Such insights are further highlighted in the CMC indentation curve shown in Figure 4. In fact, the resistance starts to drop at about 45 µm, indicating the end of pure contact with PTFE+Pb, and then further decreases at about 50 µm indicating that the contact takes place majorly with bronze.

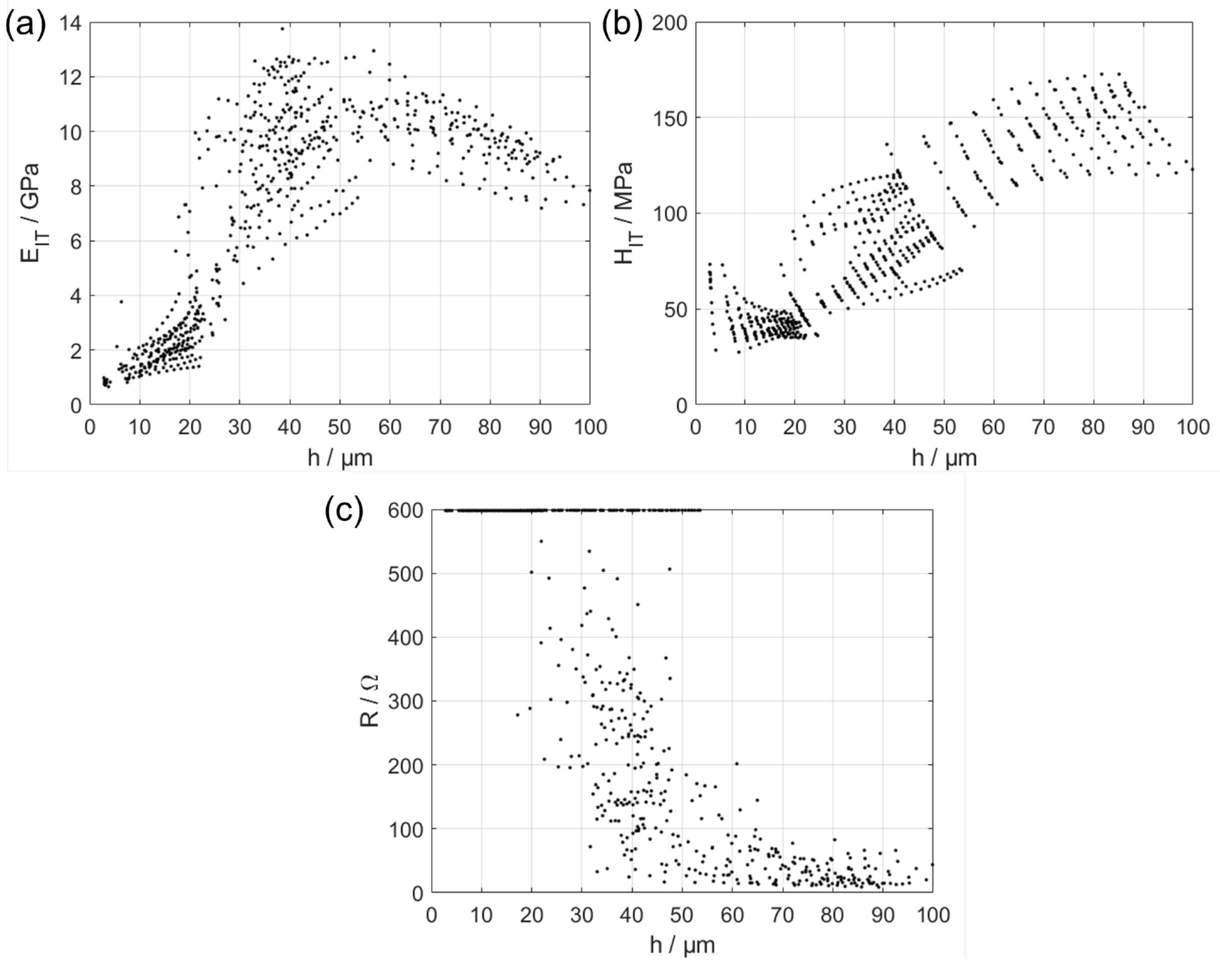

Figure 3 Results of the micro-scale characterization with augmented CMC indentation. Scatter plot of (a) E_{IT}, (b) H_{IT} and (c) R as a function of the penetration depth.

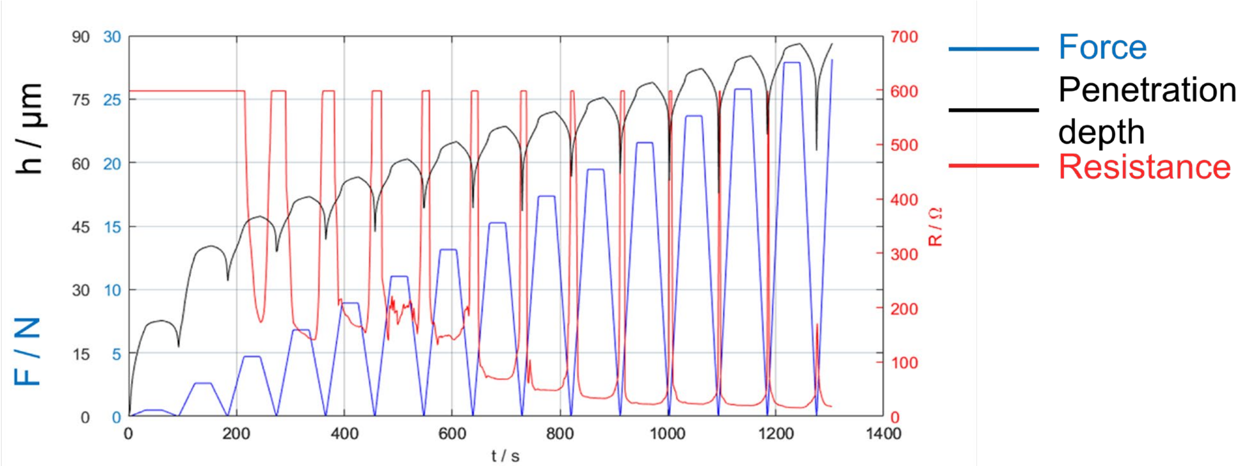

Figure 4 Augmented CMC indentation results: force (blue), penetration (black) and resistance (red) as a function of time.

Conclusions

This work presented a thorough mechanical and tribological characterization of a complex composite coating for plain bearings. Although this kind of coatings is widespread in many industrial applications, the detailed knowledge of their mechanical properties was lacking in the scientific literature.

This work has introduced an empirical methodology to achieve such a characterization. The characterization methodology was based on instrumented indentation test augmented by in-situ contact resistance measurements. The methodology has been applied, and demonstrated effective, on a commercially available grade of composite multi-layer coating (PTFE+Pb/Bronze). The approach proved effective in characterizing the material while obtaining information through the data augmentation otherwise achievable only through destructive cross-sectioning.

Quasistatic and dynamic nano-scale analysis performed on the PTFE+Pb layer evaluated its mechanical characteristic in terms of Young modulus (1.1±0.07) GPa, indentation hardness (46.2±2.4) MPa and dynamic loss modulus (0.15±0.01) GPa. Outliers in the surface mapping, otherwise homogeneous, were related to surfacing bronze particles inherent in the manufacturing of the coating, and their presence was confirmed by optical metallographic microscopy.

Microscale characterization with continuous multi cycle evaluated the evolution of mechanical properties as a function of depth. The mathematical description of the evolution of the material property as a function of depth will allow more accurate numerical modelling of the material behaviour, only roughly approximated in previous literature, even if essential in designing its application.

Data augmentation by ECR showed that transition and systematic differences in the material properties are consistent with the measured layer thickness and allowed insights into the data dispersion from the mechanical characterization.

The obtained results will allow future work to investigate traceable and metrologically trustworthy contact virtual experiments to support the design of components and applications.

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