

In-situ Raman Studies of Adaptive Tribological Surfaces

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The study provides examples of in-situ Raman spectroscopy use to investigate adaptive tribological surfaces lubricated with solid and liquid materials. The in-situ Raman spectroscopy is shown to capture tribologically induced changes of adaptive surfaces inside the lubricated sliding contact in response to applied mechanical and thermal stresses. The studies show a clear correlation of observed evolution of chemical and phase compositions in the tribological contacts as a function of sliding cycles and temperature with recorded friction coefficients, corroborating the versatility of the in-situ Raman analysis method.

Keywords: sliding friction, adaptive surface, Raman analysis, in-situ monitoring

oxidations, which increased to 0.4-0.5 at elevated temperatures.

1. Introduction

In-situ Raman spectroscopy is a powerful analytical method to investigate the evolution of tribological surfaces [1]. It is especially beneficial for understanding changes of adaptive contacts, which undergo surface chemical and phase composition self-adjustments that depend on humidity or temperature. Examples of such are solid lubricating “chameleon” coatings designed to change tribofilm composition depending on the operating environment [2]. In-situ Raman method is also important for the analysis of liquid lubricated contacts, where lubricant starvation regimes can lead to a lubricant decomposition and surface chemical changes which affect the system tribological behaviour. In this study, several examples highlighting the use of in-situ Raman spectroscopy are presented with a focus on investigating adaptive sliding contacts.

2. Methods

A Renishaw Raman Microscope with 532 nm wavelength was paired with a high-temperature pin-on-disk tribometer (Fig. 1, a) [1]. For solid lubricated adaptive contacts, duplex plasma-electrolytic oxidation (PEO) and chameleon coating produced on Al and Ti alloys were investigated. These were composed of a 80-160 μm thick hard load-supporting Al-Si-O or Ti-Si-O layers and a 5–8 μm top layer of a chameleon coating made of graphite or BN, MoS₂ or WS₂, and Sb₂O₃ [3]. For liquid contacts, 52100 steel coupons were tested under lubricant starvation regimes using low viscosity hydrocarbons. The counterpart pins were made of 52100 steel and Si₃N₄. The tests were performed at temperatures ranging from room (liquid lubrication) to 600 °C (solid lubrication). Raman spectra were continuously collected during the tribology tests.

3. Discussion

In-situ Raman spectroscopy revealed the chemical stability of the dual-phase PEO-Chameleon coatings at temperatures up to 500 °C, above which oxidation of the chameleon coating components was detected. This was directly correlated to the measured friction coefficients that ranged from 0.02 to 0.1 before the on-set of

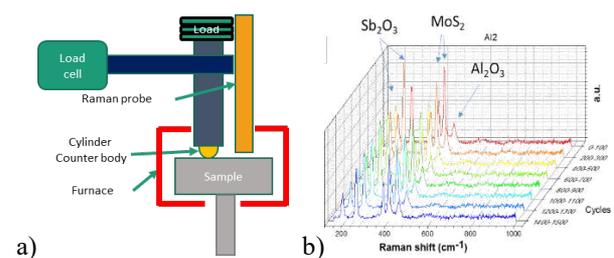


Fig. 1. In-situ Raman characterization: a) schematic of the test arrangements and b) example of spectra recorded during PEO-Chameleon coating sliding test at 300 °C [3].

In-situ Raman spectroscopy revealed that the lubricating phases, i.e. MoS₂, WS₂, and graphite, were protected from oxidation by the porous PEO structure. It also revealed a gradual evolution of chameleon composition with diminishing orthorhombic Sb₂O₃ phase presence at the surface in favor of hexagonal lubricating phases of chameleon components (Fig. 1, b). The low shear strength of MoS₂, WS₂, and graphite and the integration of the chameleon coating with the PEO sublayer were responsible for the ultra-low friction behavior. Similarly, in starved liquid lubricating contacts, the formation of hexagonal graphitic films was detected and linked to the hydrocarbon decomposition in the tribological contacts. In all examples, the in-situ Raman spectroscopy was shown to reliably detect contact chemical change correlated with the observed friction behavior.

4. References

- [1] C. Muratore et al., “In situ Raman spectroscopy for examination of high temperature tribological processes,” *Wear* 270 (2011) 140-145.
- [2] A. A. Voevodin and J. S. Zabinski, “Supertough Wear Resistant Coatings with “Chameleon” Surface Adaptation”, *Thin Solid Films*, 370 (2000) 223-231.
- [3] A. Shirani et al. “PEO-Chameleon as a potential protective coating on cast aluminum alloys for high-temperature applications”, *Surface and Coatings Technology* 397 (2020), 126016 1-12.