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Data science techniques applied to *in-situ* XRD measurements of copper under tribological load

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Microstructural alterations in metals are often the precursor to catastrophic wear. To trace the early stages of plastic deformation in a model polycrystalline metal under tribological load, we conducted *in-situ* X-ray diffraction (XRD) analysis at the surface of a copper sample. As a result, more than 13,000 XRD diffractograms were collected, which complement the evolution of the friction force signal. Due to the sheer size of this dataset, machine learning was then involved to help unravel the possible links between friction and microstructure.

Keywords (from 3 to 5 max): copper, synchrotron, XRD, machine learning

1. Introduction

Friction is inherently a multi-scale phenomenon: the mechanics that govern macroscale sliding originate at the nanoscale, and often leave permanent microstructural alterations even if no apparent wear occurs at the surfaces of the contacting bodies. In metals, frictional energy is primarily dissipated through the material underneath the contact. XRD analysis is a well-established non-destructive technique, which provides information about a material's local microstrain accumulation, grain-to-grain interaction or grain size evolution. Thus, combining tribological experiments with the continuous tracking of diffraction patterns, acquired at the same location, is a promising avenue for gaining insights about the connection between surface stresses and resulting microstructural alterations.

2. Methods

We used the ANKA synchrotron source, at Karlsruhe Institute of Technology, to collect XRD patterns of copper, which was slid against a sapphire sphere between each X-ray exposure. The tribological experiments we conducted spanned normal loads of 1 to 4 Newtons, which correspond to maximum Hertzian contact pressure between 420 MPa and 670 MPa, at sliding speeds of 0.5 to 8 mm/s.

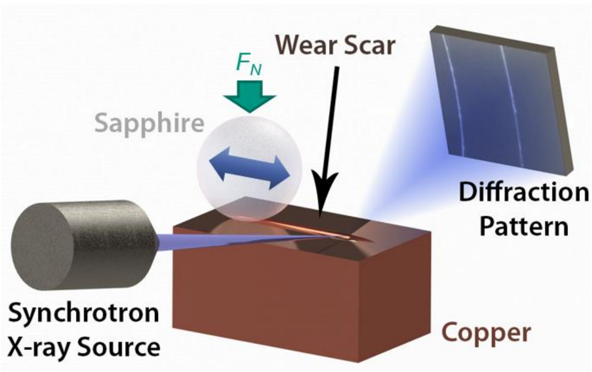

Figure 1: Experimental setup for collecting XRD diffractograms at the surface of copper. After a set number of sliding reciprocations of the sapphire ball, the test was paused to allow for the X-rays to reach the same position at the sample's surface.

Table 1: Experimental parameter used in the tests

Material	Annealed (2 hours at 500 °C), ~45 µm initial grain size, oxygen-free high conductivity Cu
Counterbody	φ10 mm sapphire sphere
Normal Load	1– 4 N (420–670 MPa)
$\boldsymbol{E}_{\mathbf{Xray}}$	7.99 keV
Effective Penetration Depth	14 µm

However, analyzing the many Debye-Scherrer rings in a traditional manner can often be a daunting task due to their sheer number: >13,000 in our case. Furthermore, finding meaningful correlations with the tribological data which was acquired in parallel, makes this effort extremely time consuming, if at all possible. To solve this issue, popular data science techniques were adapted and applied. Tribological patterns, previously hard to identify, were extracted and correlated with changes in the material's structure.

2.1. Results

After isolating the uniform Debye-Scherrer ring portions from individual large-grain diffraction spots, an asymmetrically-split Pearson VII function was fitted. [1] Comparing fitting parameters, such as θ_0 and full width at half maximum, to the corresponding friction force, revealed a striking positive correlation in the first 100 to 1000 cycles, which abruptly switches to a negative correlation thereafter. Additionally, it was discovered that the instance of correlation flip is strongly dependent on the normal force applied.

3. Discussion

This study demonstrates that the channels for frictional energy dissipation within a metal shift as plastic deformation is accumulated, especially at the onset of sliding. Thus, tuning the sliding conditions can be an effective way to mitigate gross wear events, which lead to catastrophic wear.

4. References

[1] Lohmiller, J., "Investigation of deformation mechanisms in nanocrystalline metals and alloys by in situ synchrotron X-ray diffraction," Dissertation, 2013, KIT, DOI: 10.5445/KSP/1000031997