

Supplementary Information for

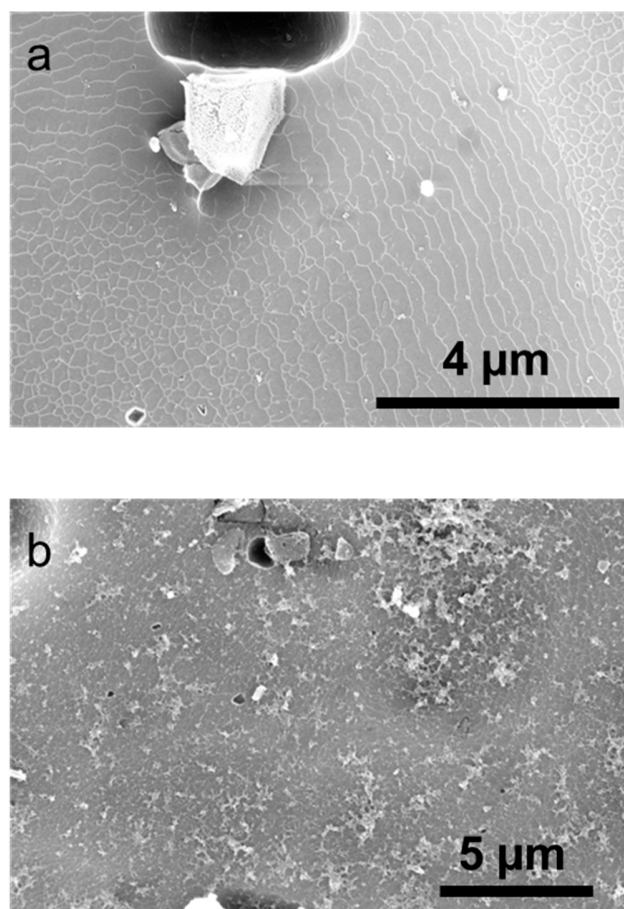
**Correlation of fabrication methods and enhanced wear performance in nanoporous anodic aluminum oxide with incorporated molybdenum disulfide (MoS<sub>2</sub>) nanomaterials**

Hatfield, Kendrick O'Donaghue<sup>1</sup>; Brown, Nathan<sup>1</sup>; Dervishi, Enkeleda<sup>1</sup>; Carpenter, Bradley<sup>1</sup>; Janusz, Jordyn<sup>1</sup>; Hooks, Daniel E. <sup>1,2\*</sup>

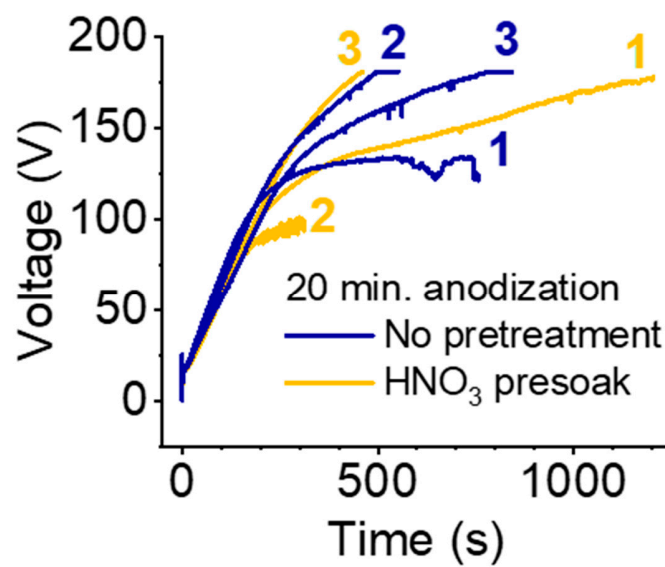
1: SIGMA-2: Finishing Manufacturing Science, Los Alamos National Laboratory, SM-30 Bikini Atoll Road, Los Alamos, NM 87545, USA; khatfield@lanl.gov (K.O.H.)

2: MPA-CINT: Center for Integrated Nanotechnologies, Los Alamos National Laboratory, Los Alamos, NM 87545, USA

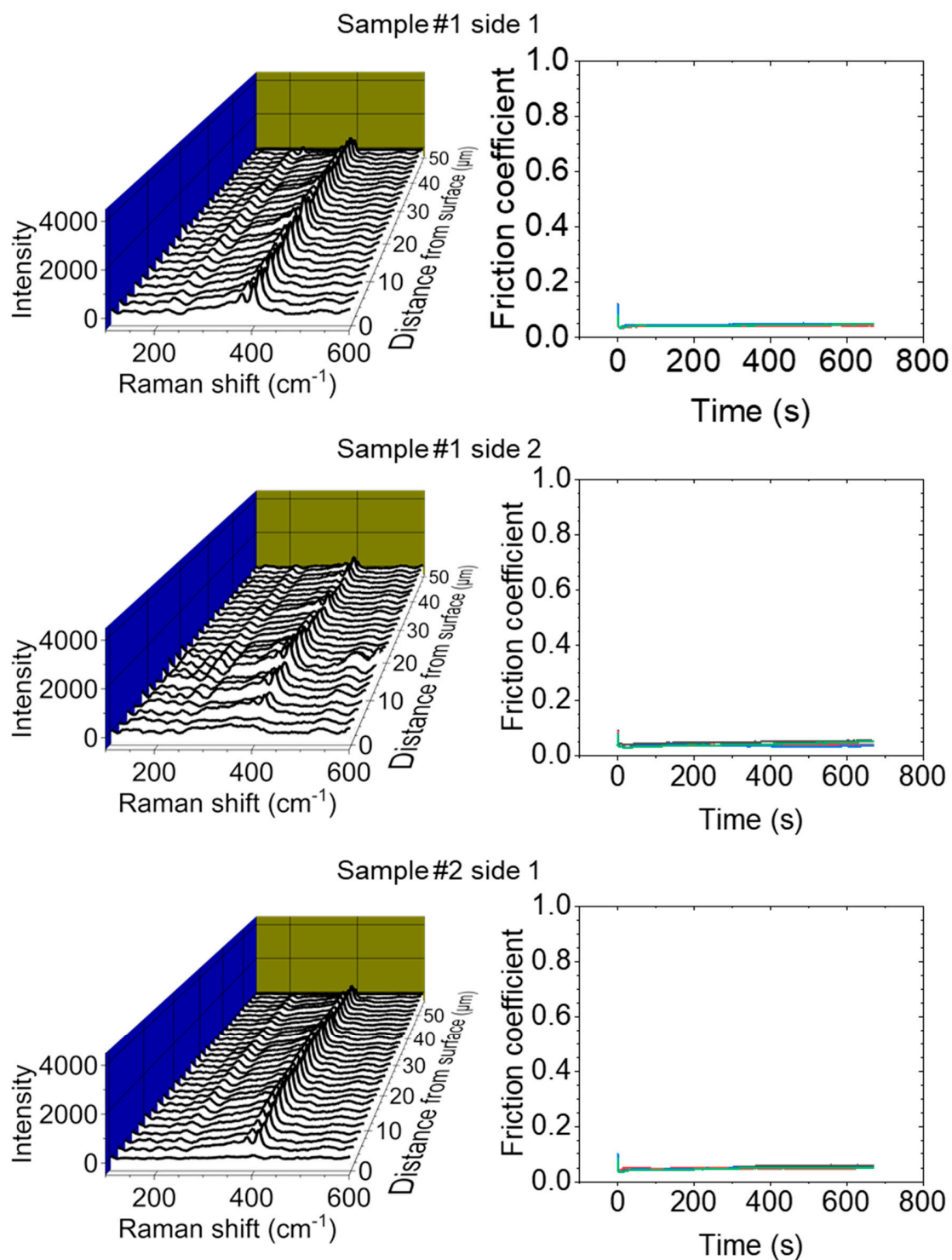
\*Corresponding author, email: dhooks@lanl.gov, phone: 505-667-6407



**Figure S1.** SEM images of 20-minute high-acid anodized a) unmodified AAO and b) MoS<sub>2</sub>/AAO.

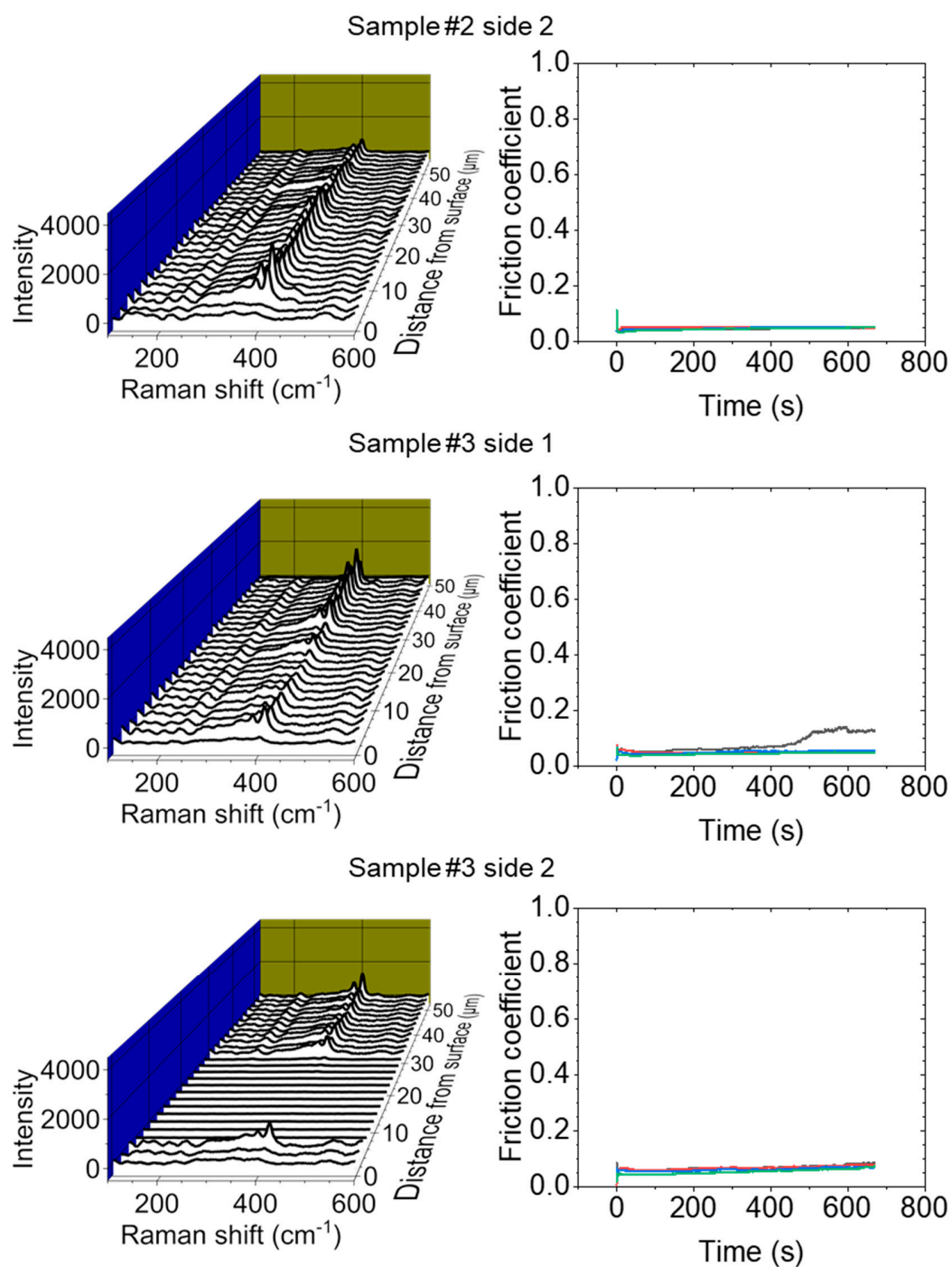


**Figure S2.** V-t deposition curves for 20-minute anodized Al 5052.

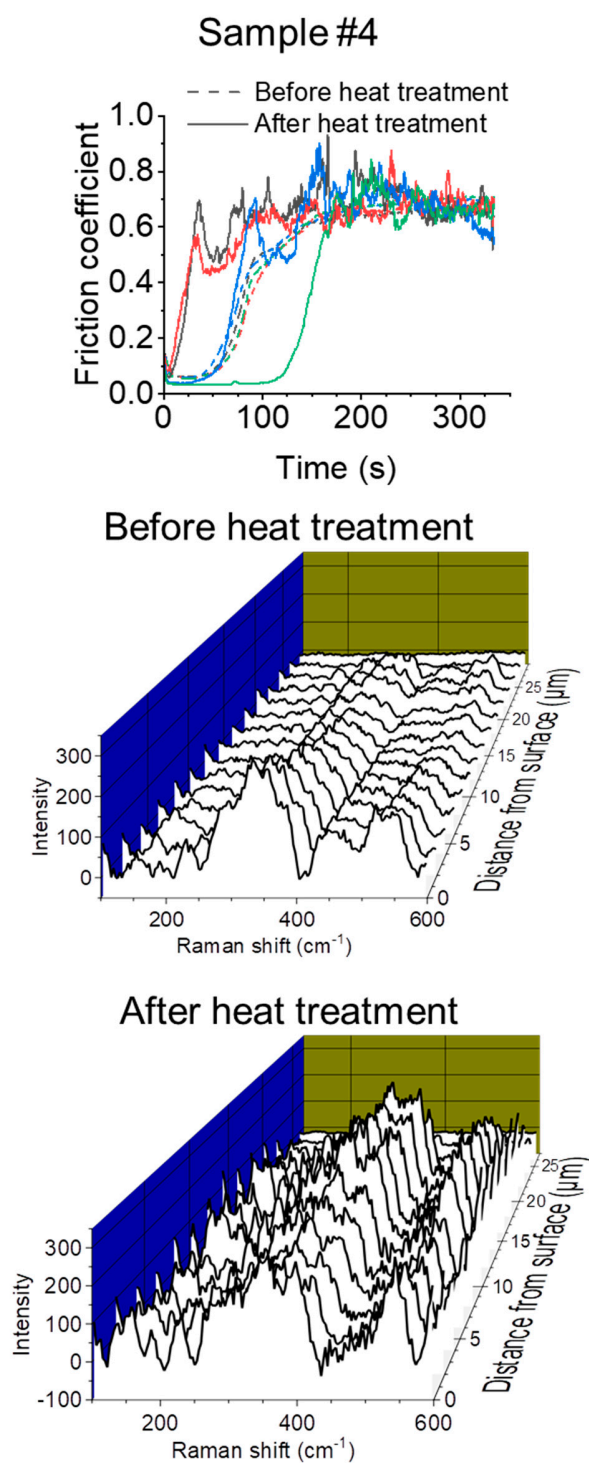


**Figure S3.** Cross-sectional Raman spectra and tribological scratch tests of 120-minute high-acid anodized Al 5052 samples with no acid pretreatment. Four scratches were performed per sample side.

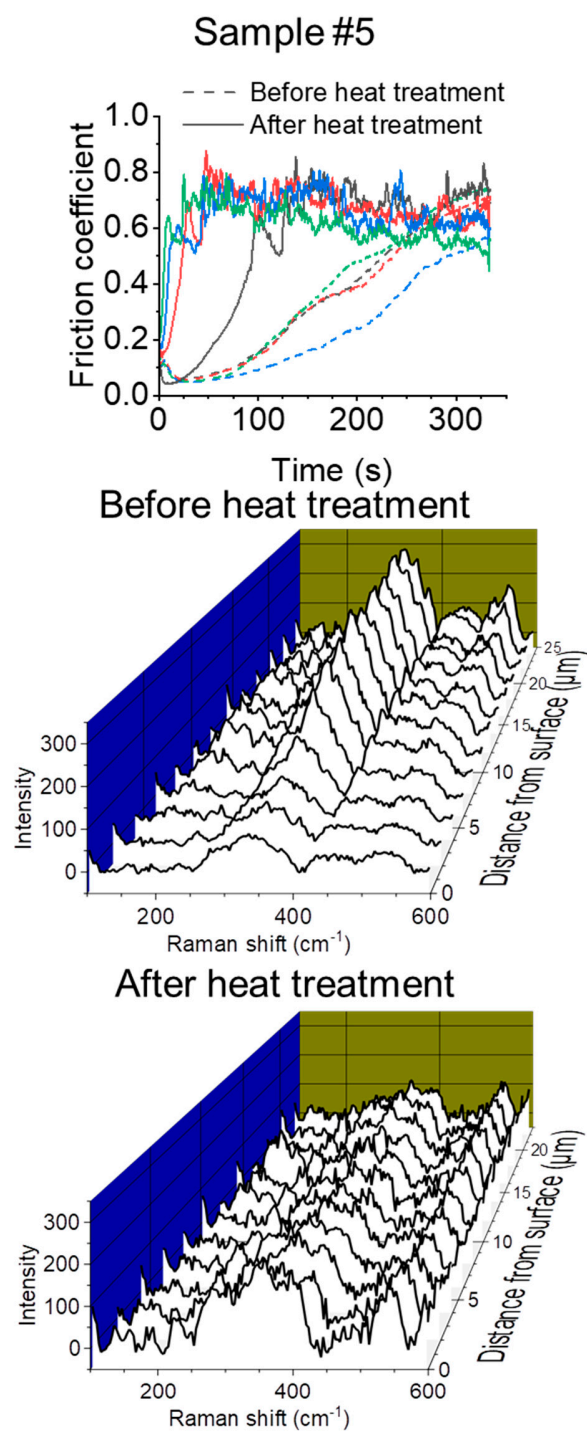




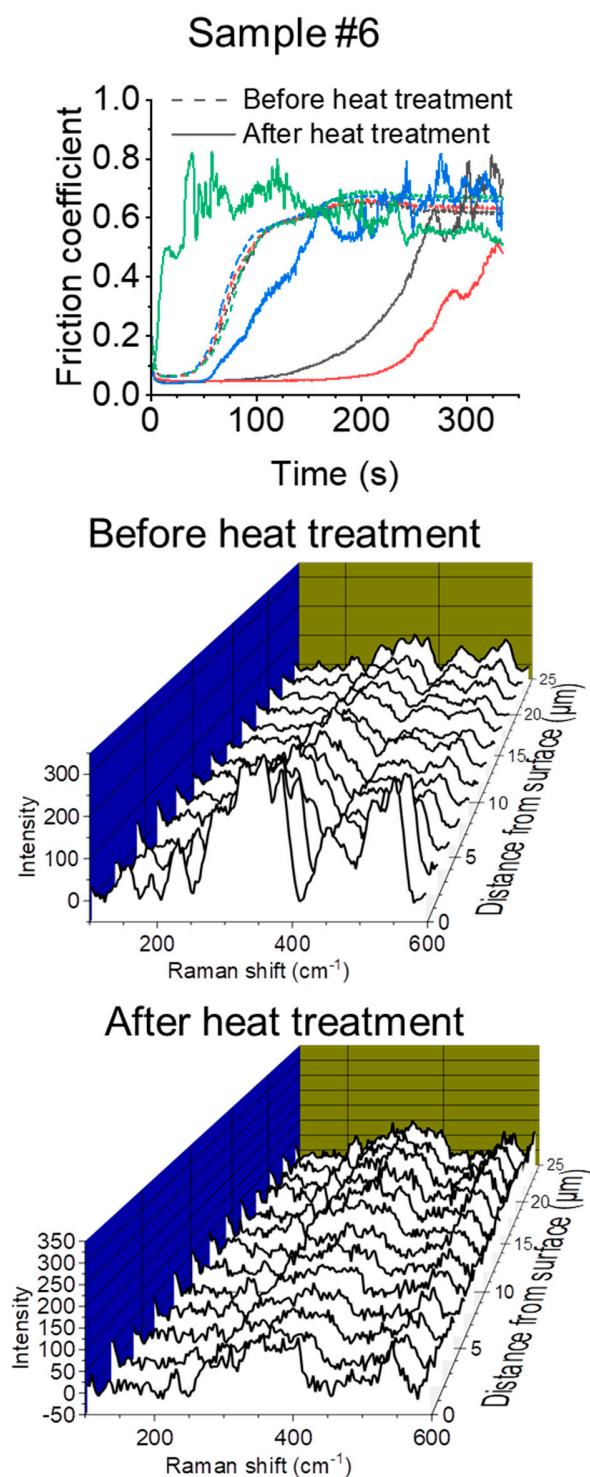
**Figure S3 (continued).** Cross-sectional Raman spectra and tribological scratch tests of 120-minute high-acid anodized Al 5052 samples with no acid pretreatment. Four scratches were performed per sample side.



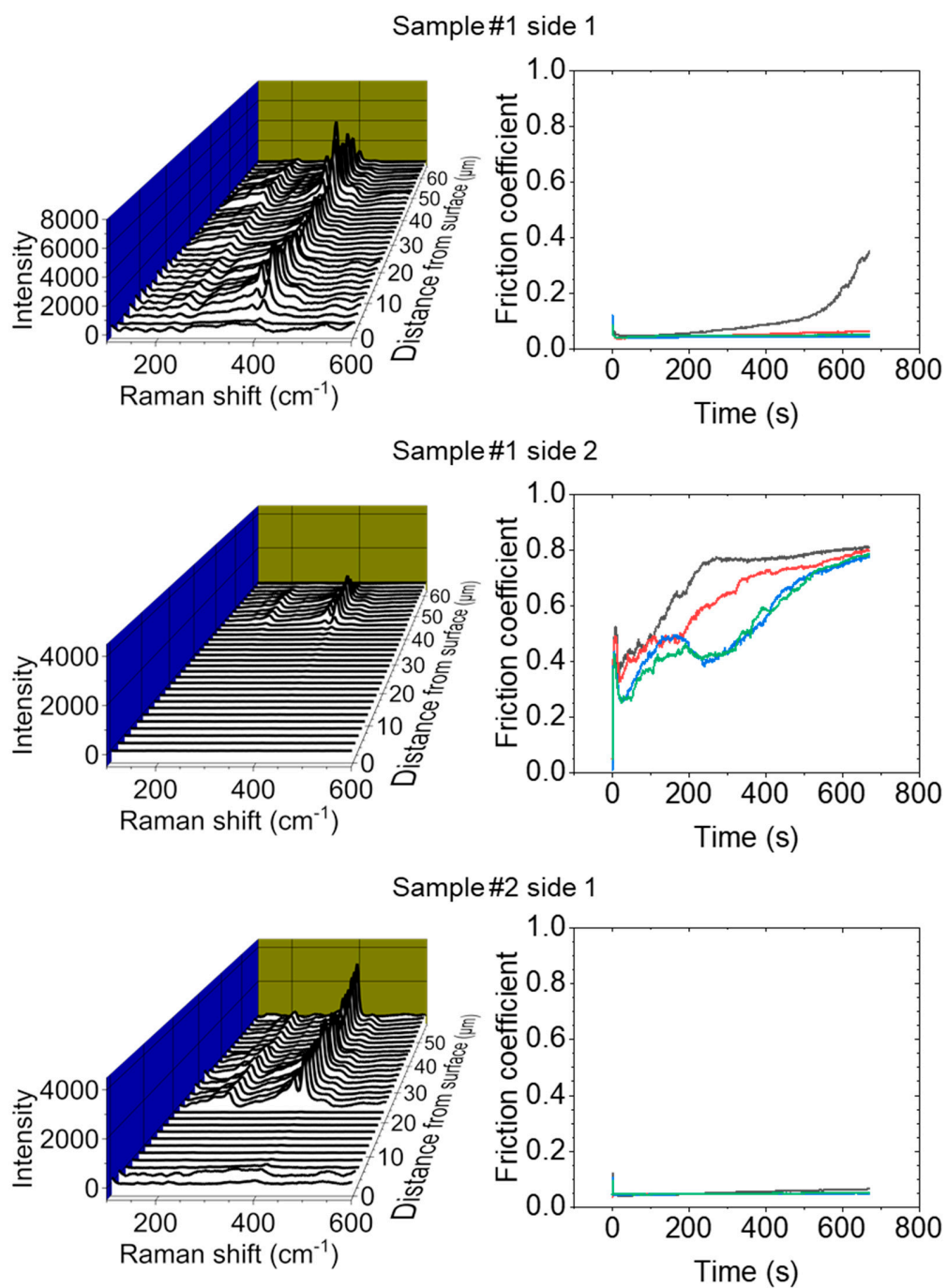
**Figure S4.** Cross-sectional Raman spectra and tribological scratch tests of 120-minute high-acid anodized Al 5052 samples with no acid pretreatment before and after heat treatment.



**Figure S4 (continued).** Cross-sectional Raman spectra and tribological scratch tests of 120-minute high-acid anodized Al 5052 samples with no acid pretreatment before and after heat treatment.

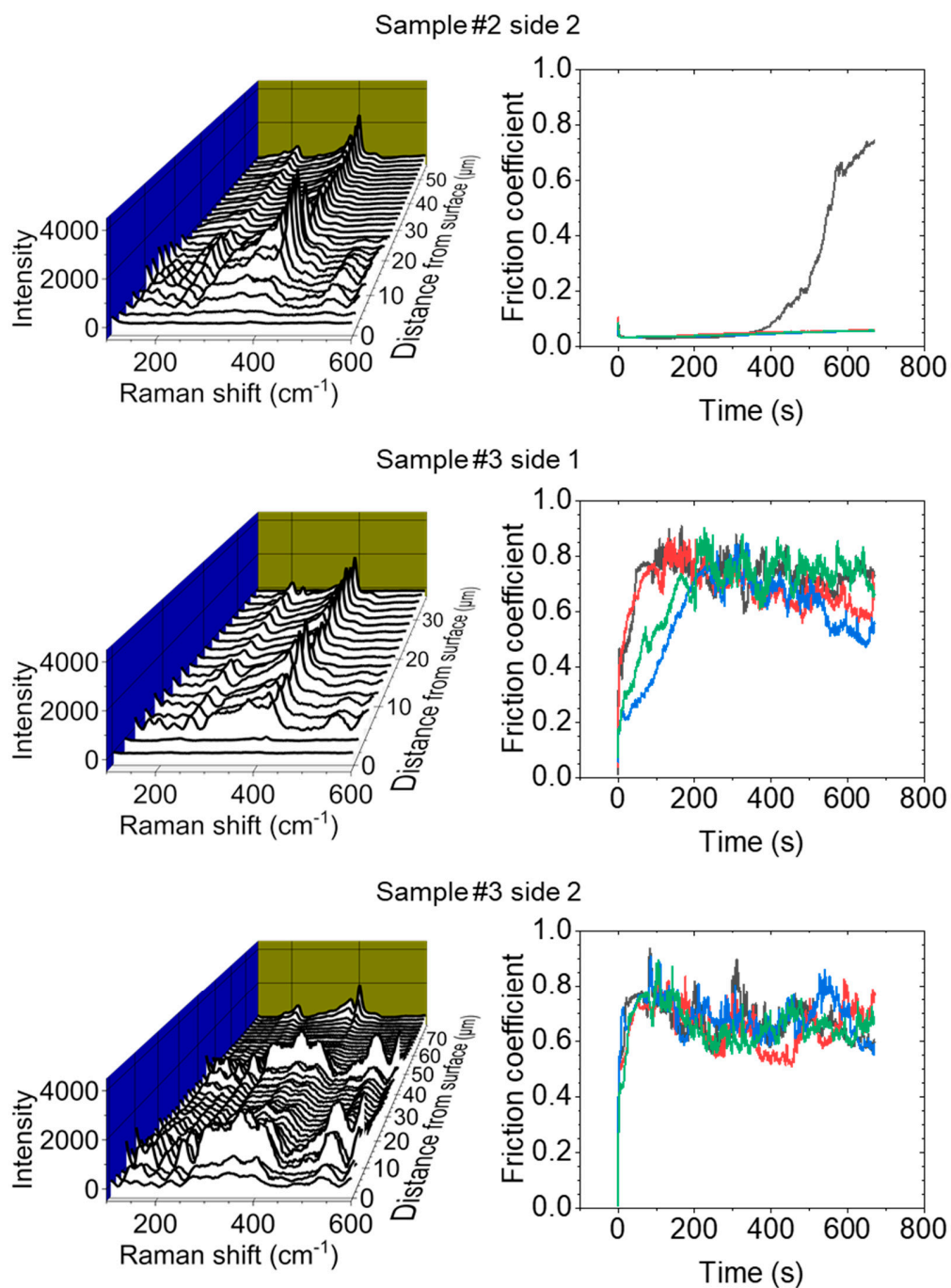


**Figure S4 (continued).** Cross-sectional Raman spectra and tribological scratch tests of 120-minute high-acid anodized Al 5052 samples with no acid pretreatment before and after heat treatment.

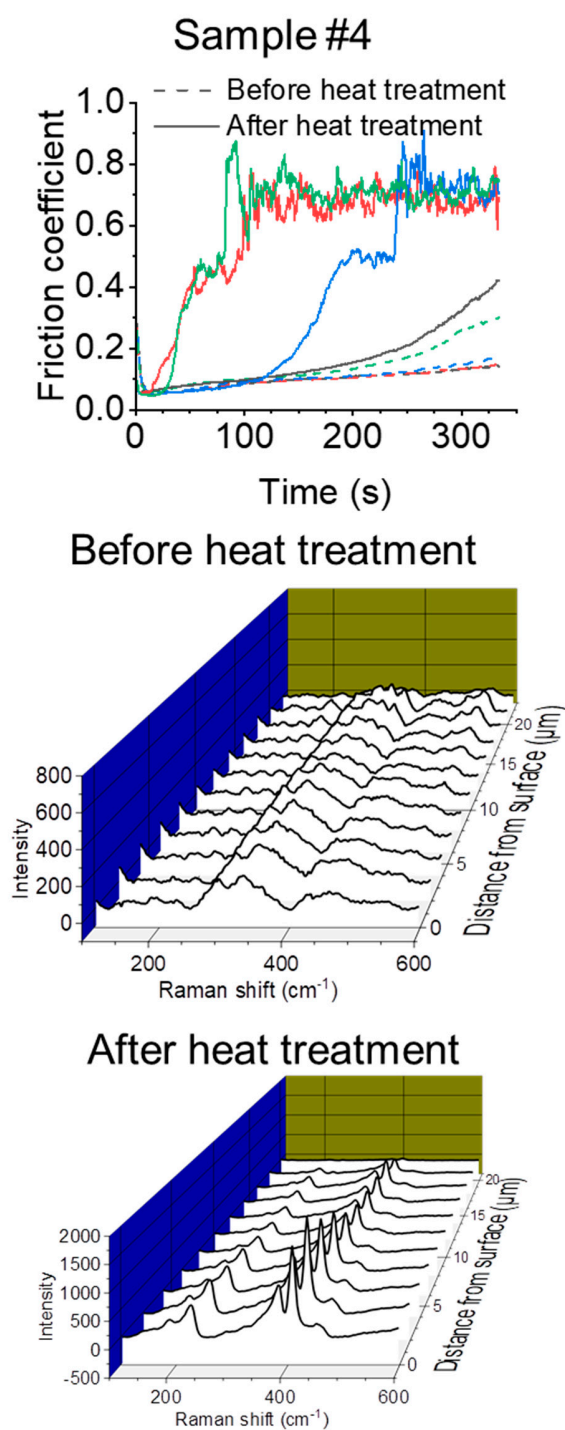


**Figure S5.** Cross-sectional Raman spectra and tribological scratch tests of 120-minute high-acid anodized Al 5052 samples with HNO<sub>3</sub> presoaking. Four scratches were performed per sample side.

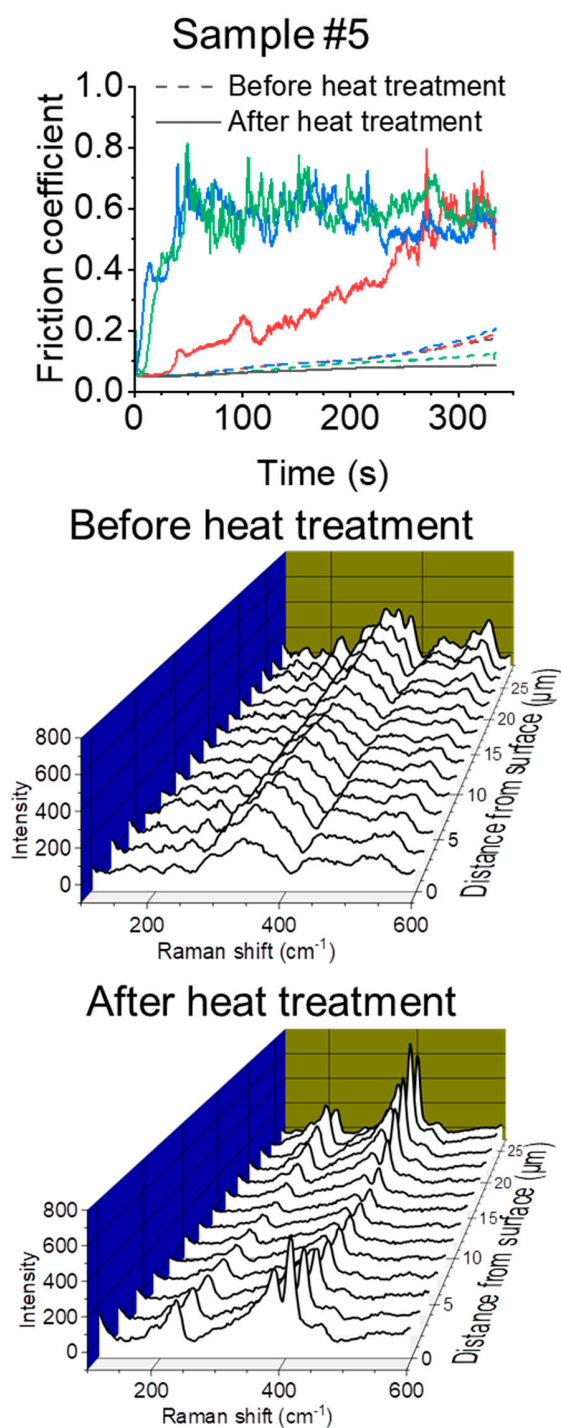




**Figure S5 (continued).** Cross-sectional Raman spectra and tribological scratch tests of 120-minute high-acid anodized Al 5052 samples with  $\text{HNO}_3$  presoaking. Four scratches were performed per sample side.

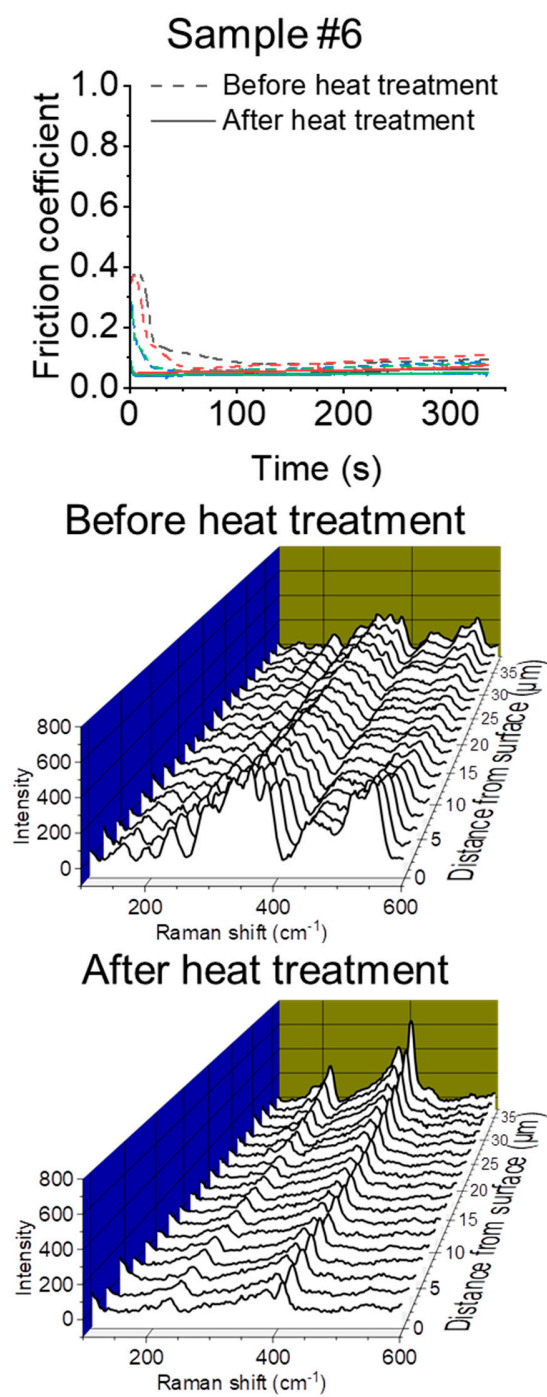


**Figure S6.** Cross-sectional Raman spectra and tribological scratch tests of 120-minute high-acid anodized Al 5052 samples with  $\text{HNO}_3$  presoaking before and after heat treatment.

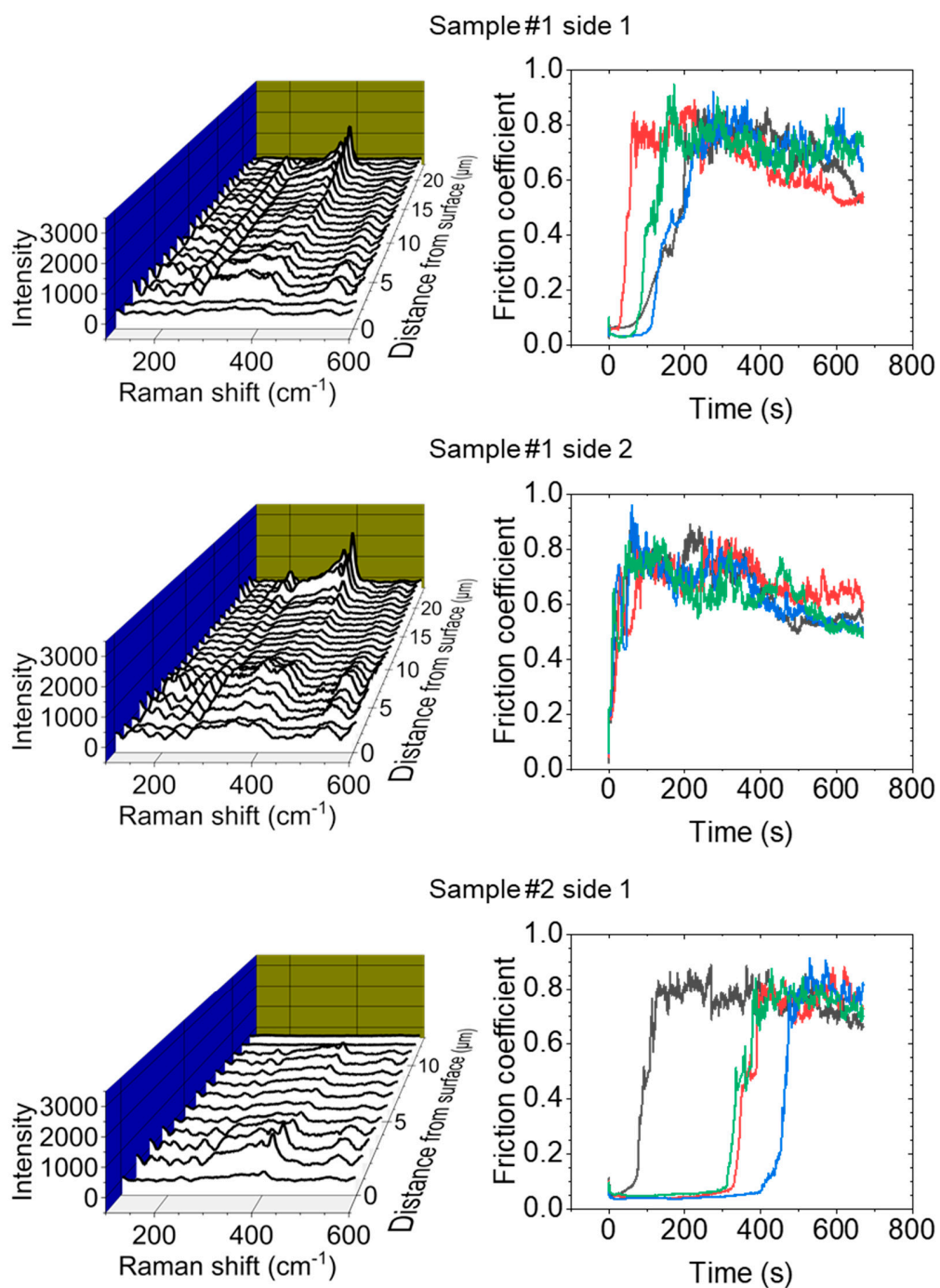


**Figure S6 (continued).** Cross-sectional Raman spectra and tribological scratch tests of 120-minute high-acid anodized Al 5052 samples with  $\text{HNO}_3$  presoaking before and after heat treatment.

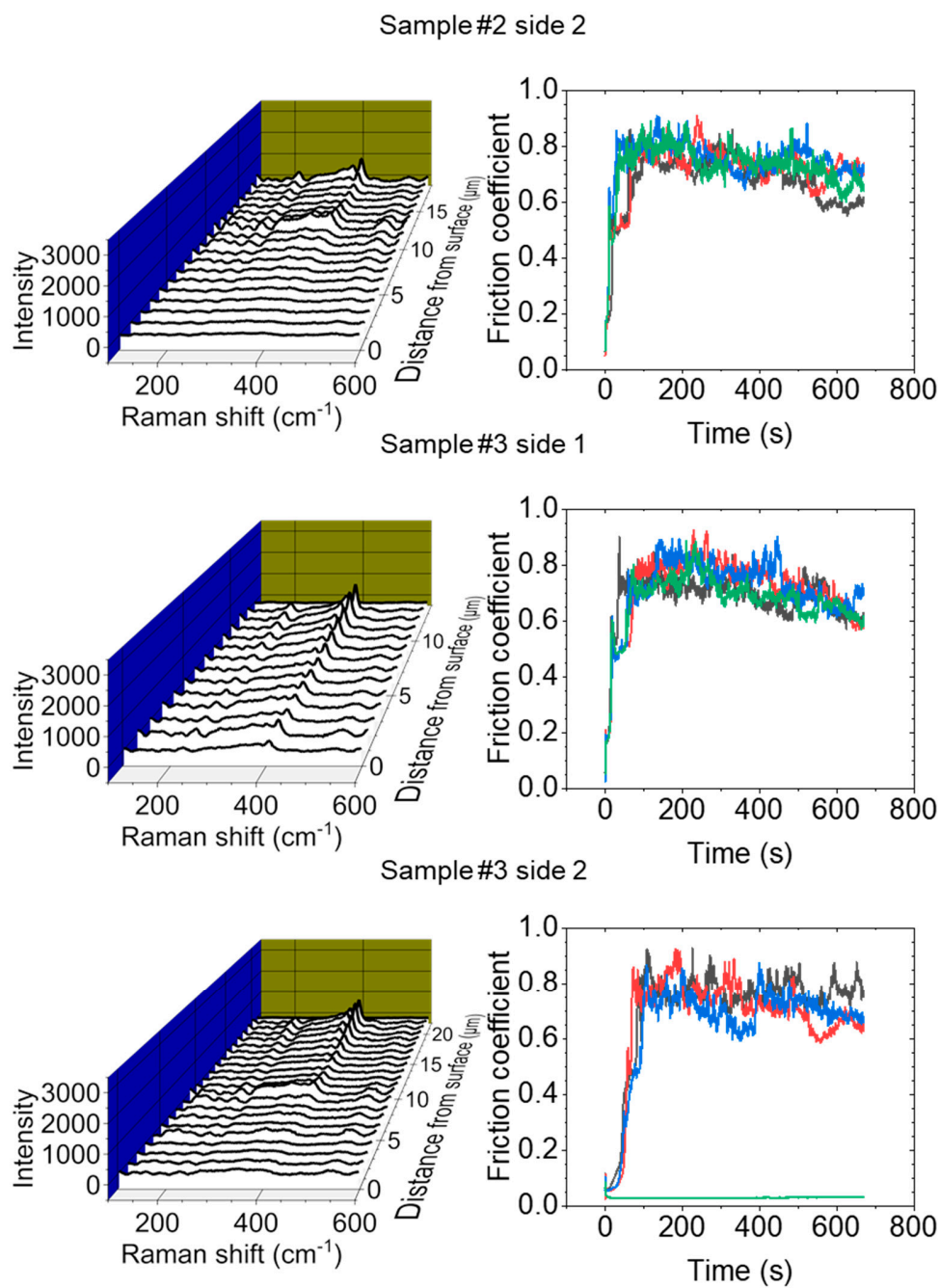




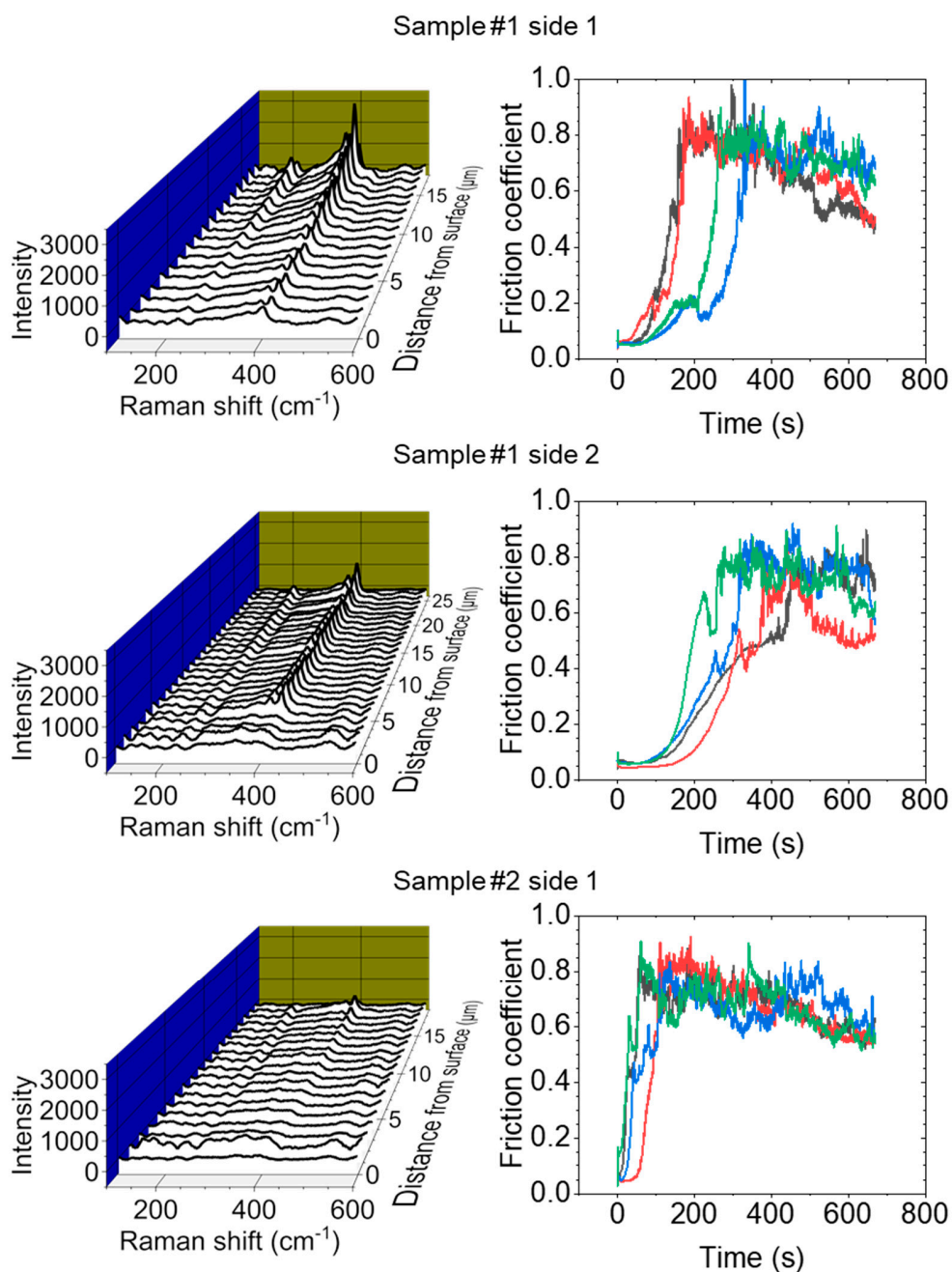
**Figure S6 (continued).** Cross-sectional Raman spectra and tribological scratch tests of 120-minute high-acid anodized Al 5052 samples with  $\text{HNO}_3$  presoaking before and after heat treatment.



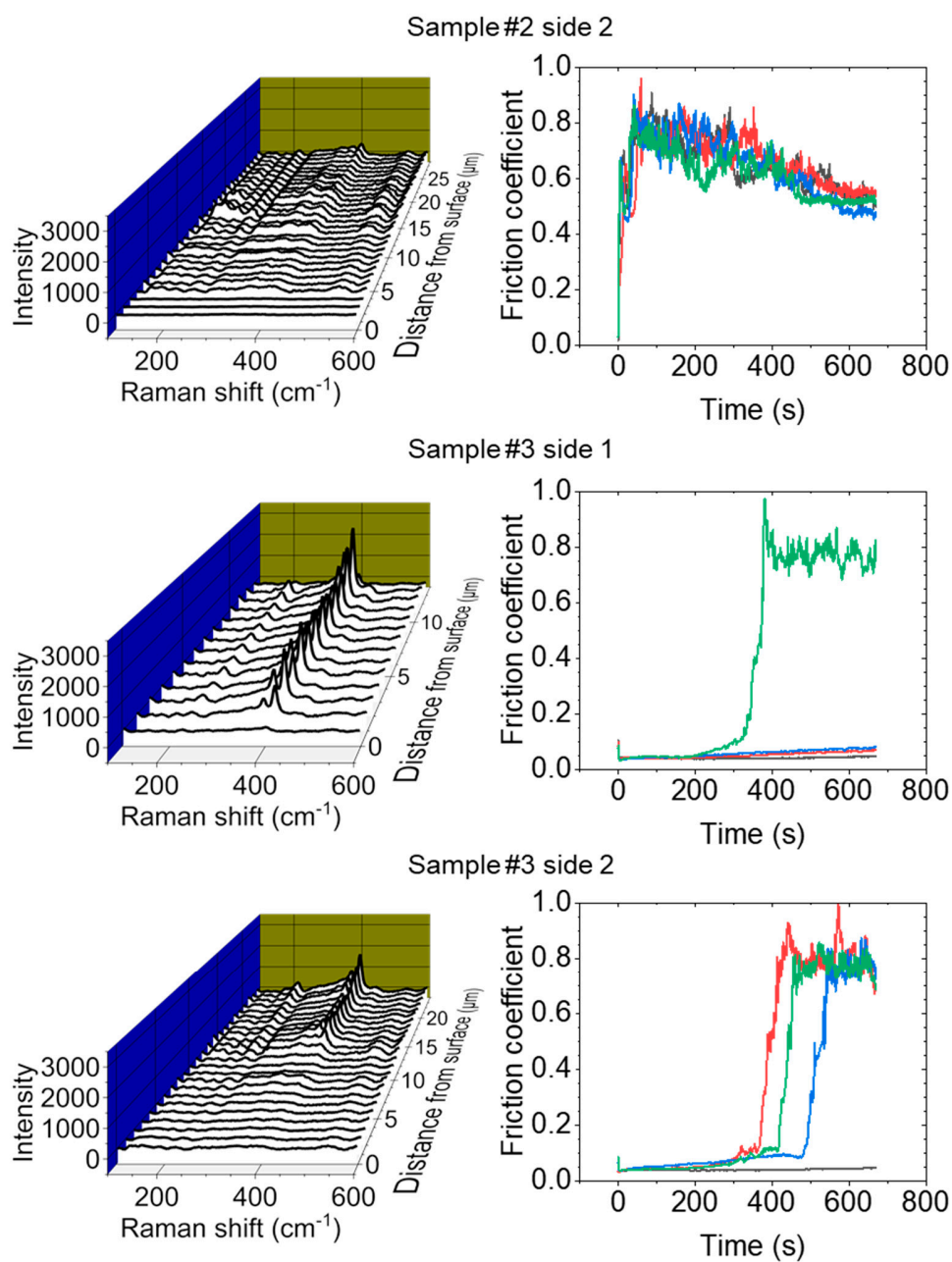
**Figure S7.** Cross-sectional Raman spectra and tribological scratch tests of 20-minute high-acid anodized Al 5052 samples with no acid pretreatment. Four scratches were performed per sample side.



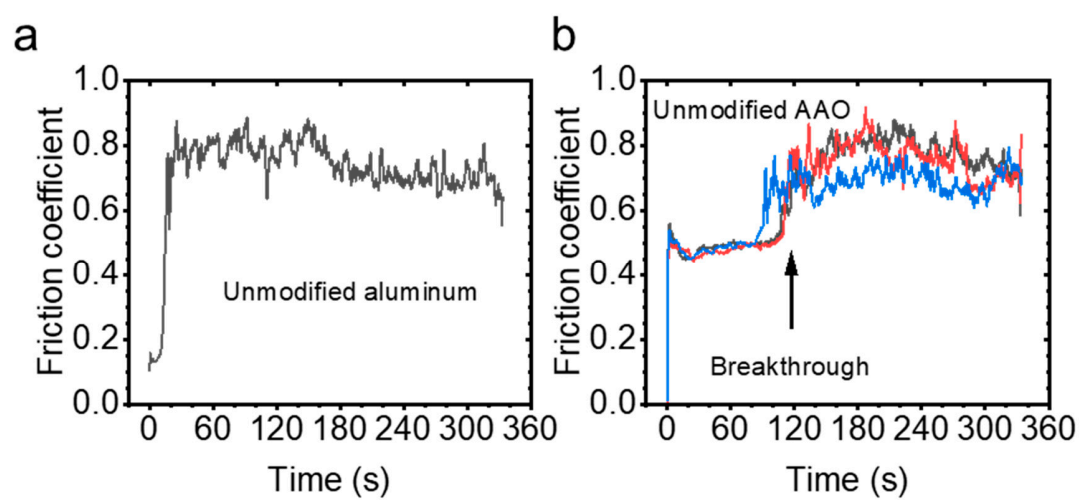
**Figure S7.** (continued): Cross-sectional Raman spectra and tribological scratch tests of 20-minute high-acid anodized Al 5052 samples with no acid pretreatment. Four scratches were performed per sample side.



**Figure S8.** Cross-sectional Raman spectra and tribological scratch tests of 20-minute high-acid anodized Al 5052 samples with  $\text{HNO}_3$  presoaking. Four scratches were performed per sample side.

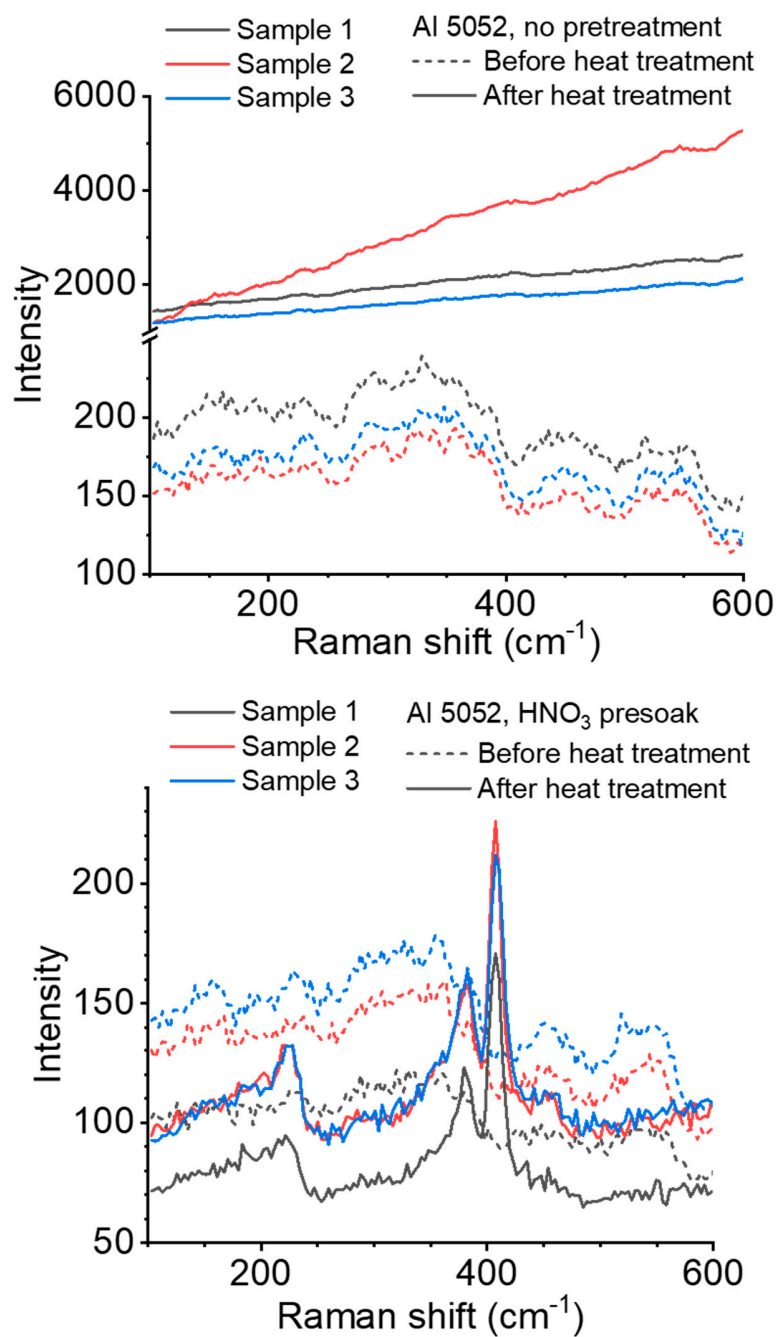


**Figure S8.** (continued): Cross-sectional Raman spectra and tribological scratch tests of 20-minute high-acid anodized Al 5052 samples with HNO<sub>3</sub> presoaking. Four scratches were performed per sample side.

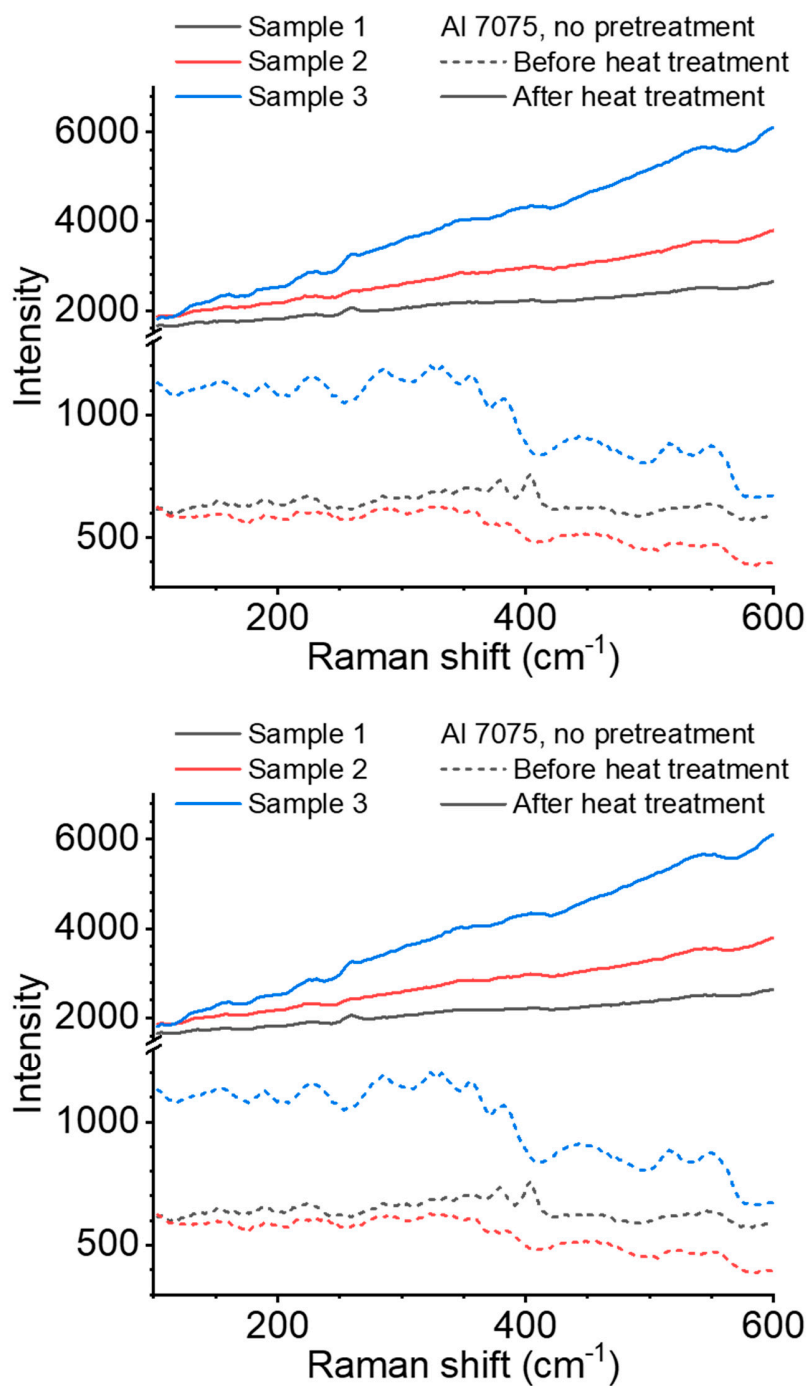


**Figure S9.** Reciprocating scratch tests of a) non-anodized Al 5052 and b) plain AAO from Al 5052.



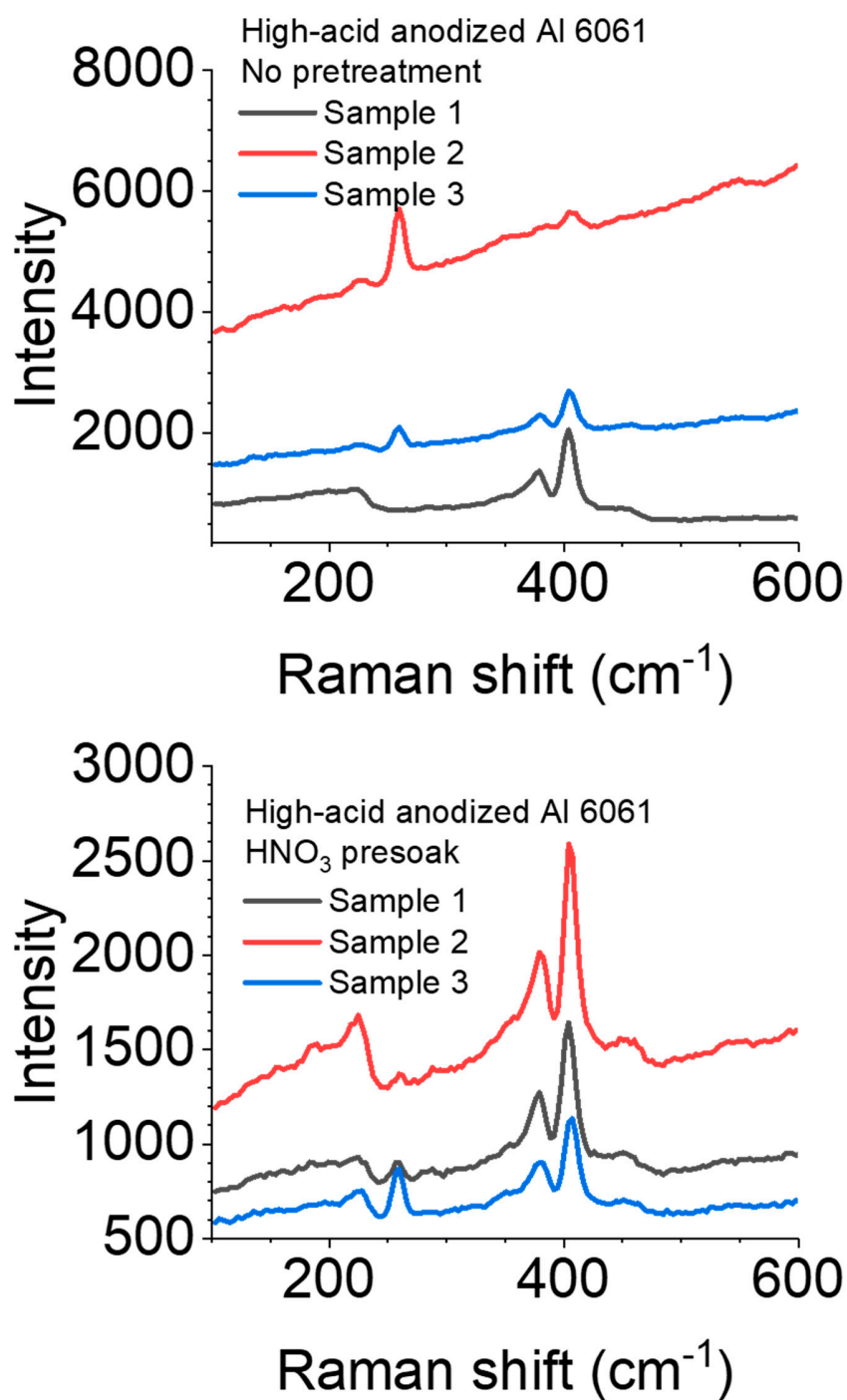


**Figure S10.** Raman spectra of Al 5052  $\text{MoS}_2/\text{AAO}$  surfaces. No background subtraction was performed on these spectra.

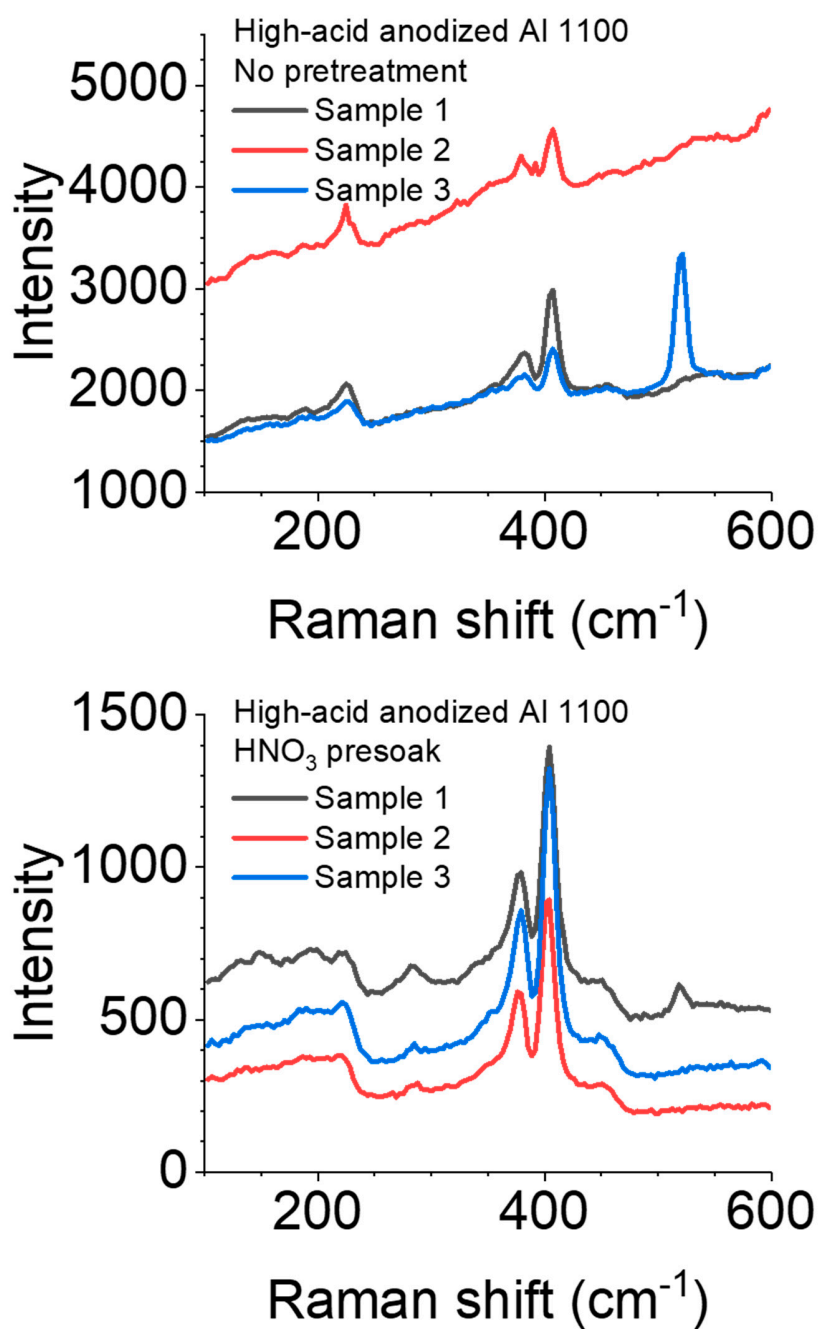


**Figure S11.** Raman spectra of Al 7075 MoS<sub>2</sub>/AAO surfaces. No background subtraction was performed on these spectra.

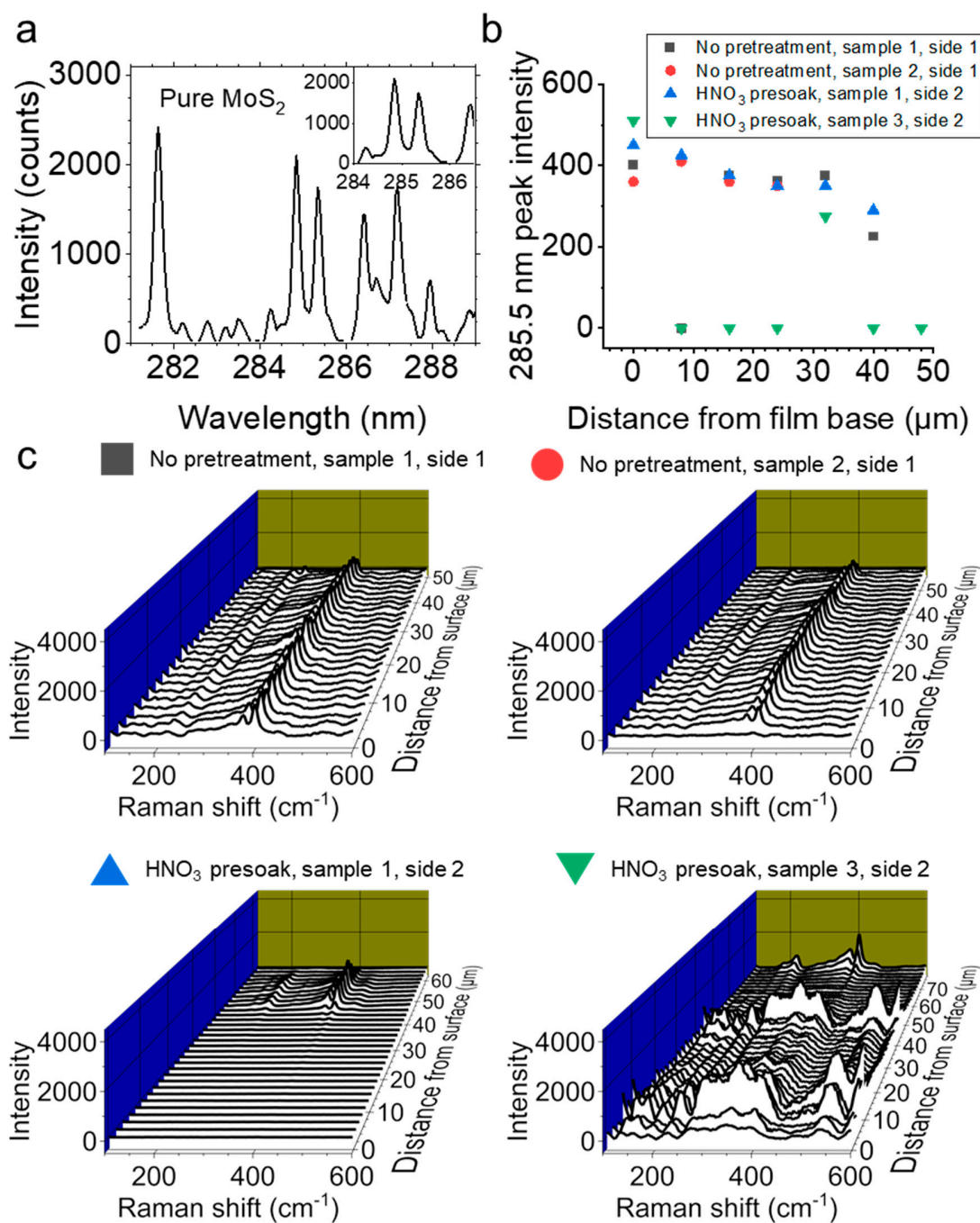




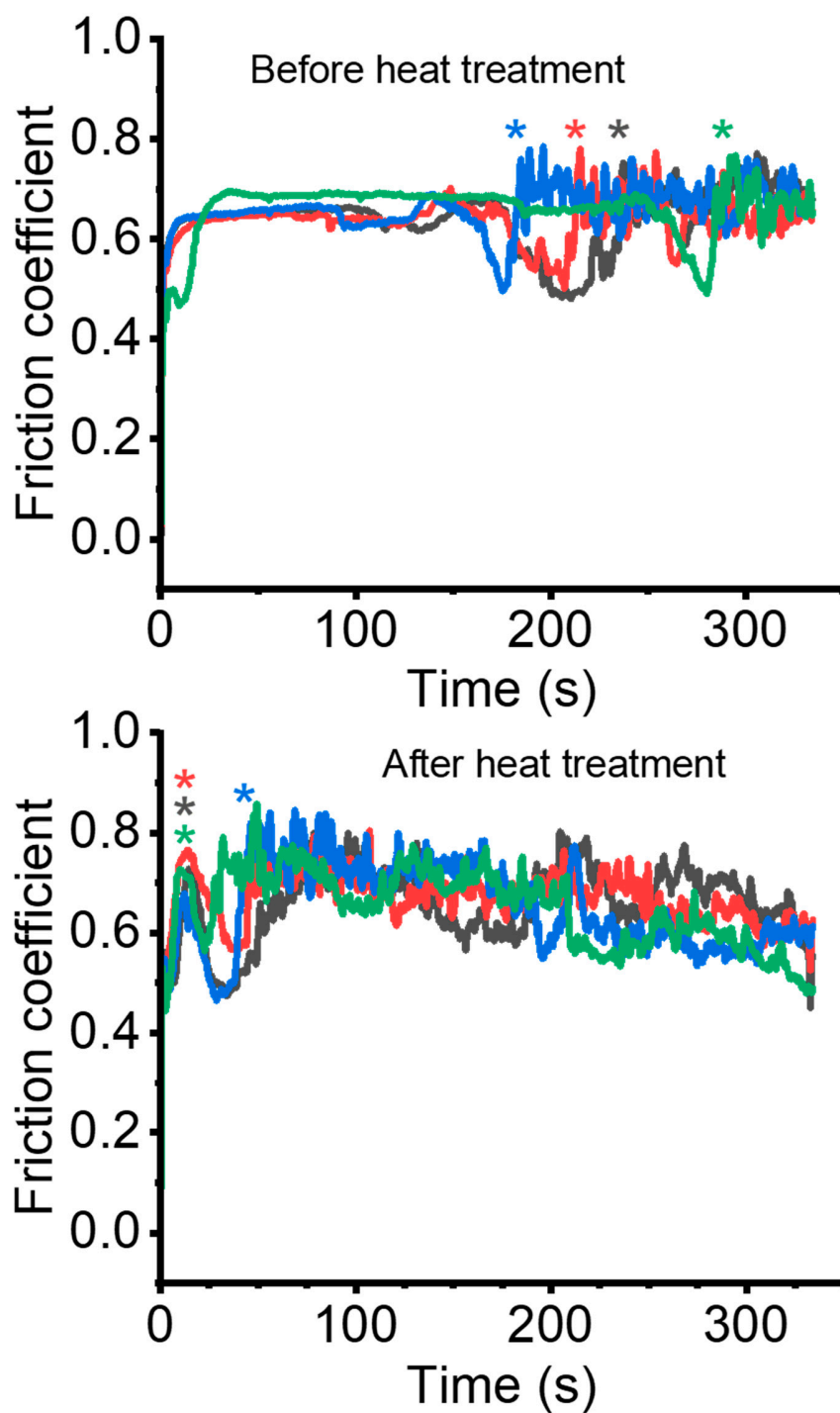
**Figure S12.** Raman spectra of Al 6061 MoS<sub>2</sub>/AAO surfaces. No background subtraction was performed on these spectra.



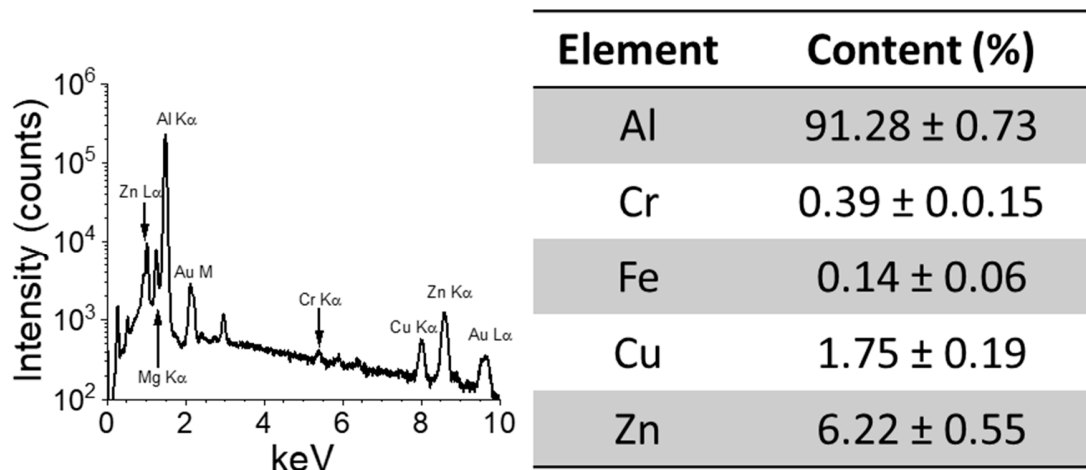
**Figure S13.** Raman spectra of Al 1100  $\text{MoS}_2/\text{AAO}$  surfaces. No background subtraction was performed on these spectra.



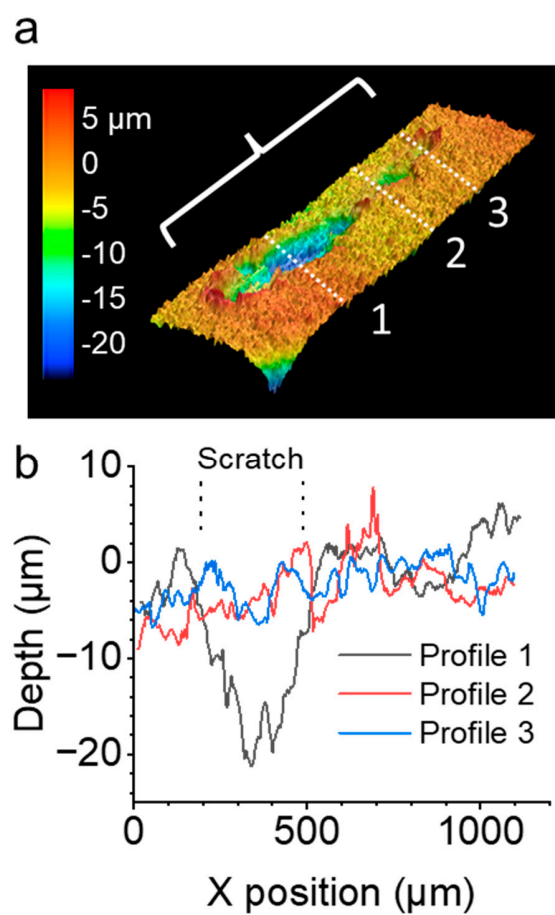
**Figure S14.** Laser-induced breakdown spectroscopy (LIBS) of MoS<sub>2</sub>/AAO. a) LIBS spectrum of pure MoS<sub>2</sub> with inset showing a zoomed plot of the peaks near 285 nm. b) 285.5 nm peak intensity for various 120-minute high-acid anodized Al 5052 sample cross-sections. c) Cross-sectional Raman spectra for direct comparison between techniques.



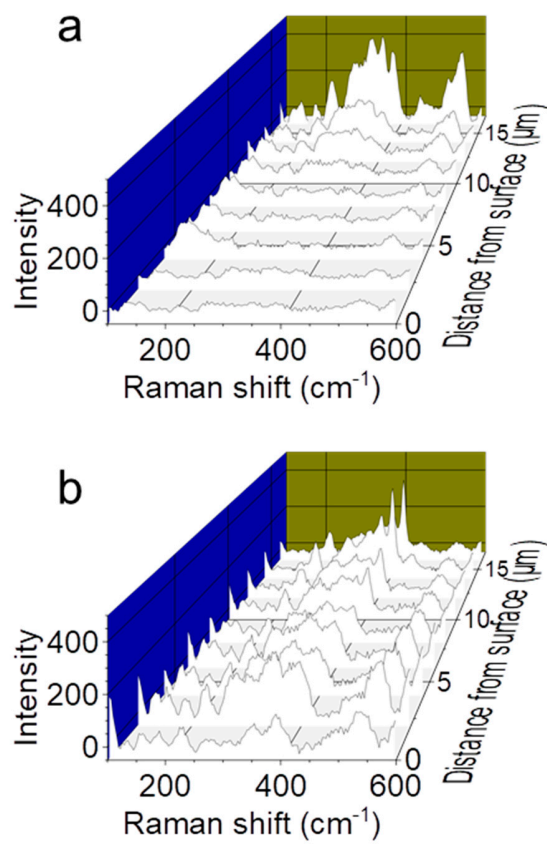
**Figure S15.** Reciprocating scratch tests of 120-minute high-acid anodized Al 5052 with no MoS<sub>x</sub> deposition a) before and b) after heat treatment. Asterisks denote film breakthrough.



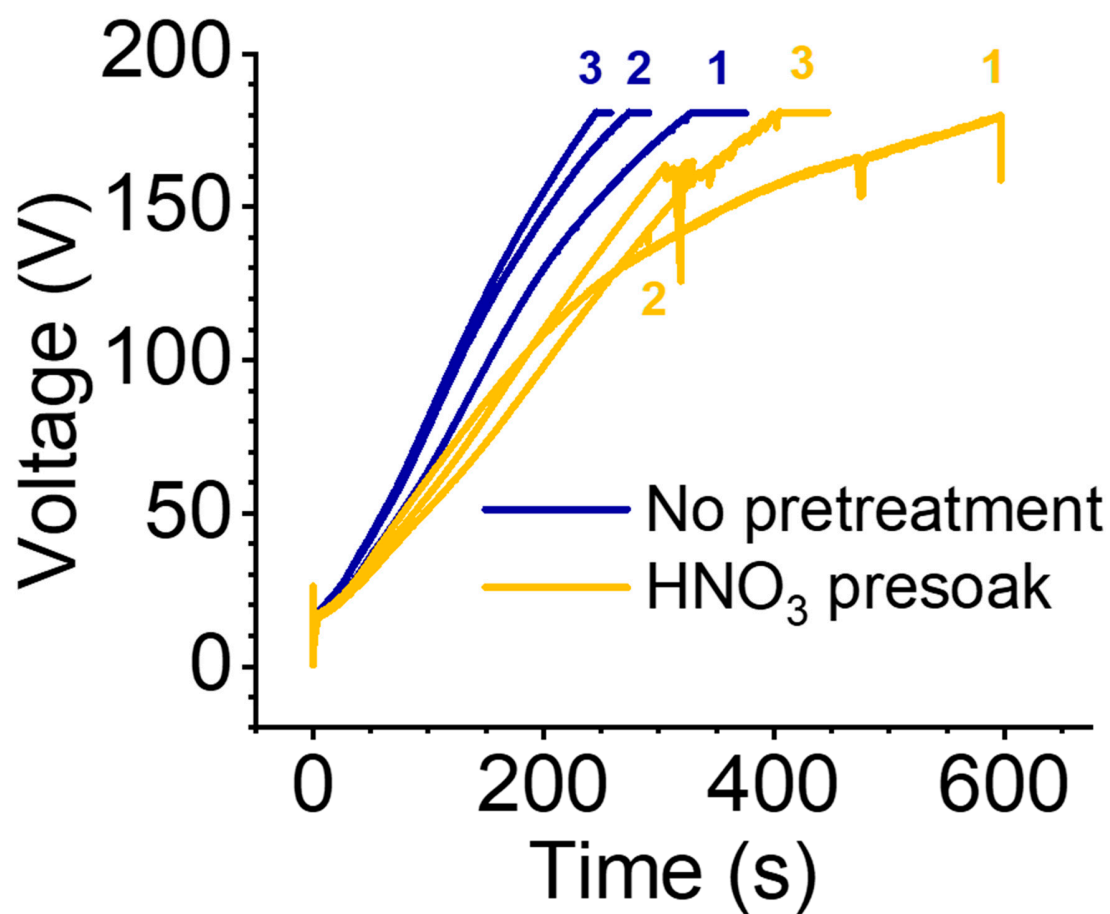
**Figure S16.** Elemental analysis of the center of a commercial MoS<sub>2</sub>/AAO part. a) EDS spectrum. b) X-ray fluorescence results.



**Figure S17.** Example scratch of a commercial MoS<sub>2</sub>/AAO film. a) 3-dimensional optical image of the scratch. b) Example depth profiles across the scratch in various areas.

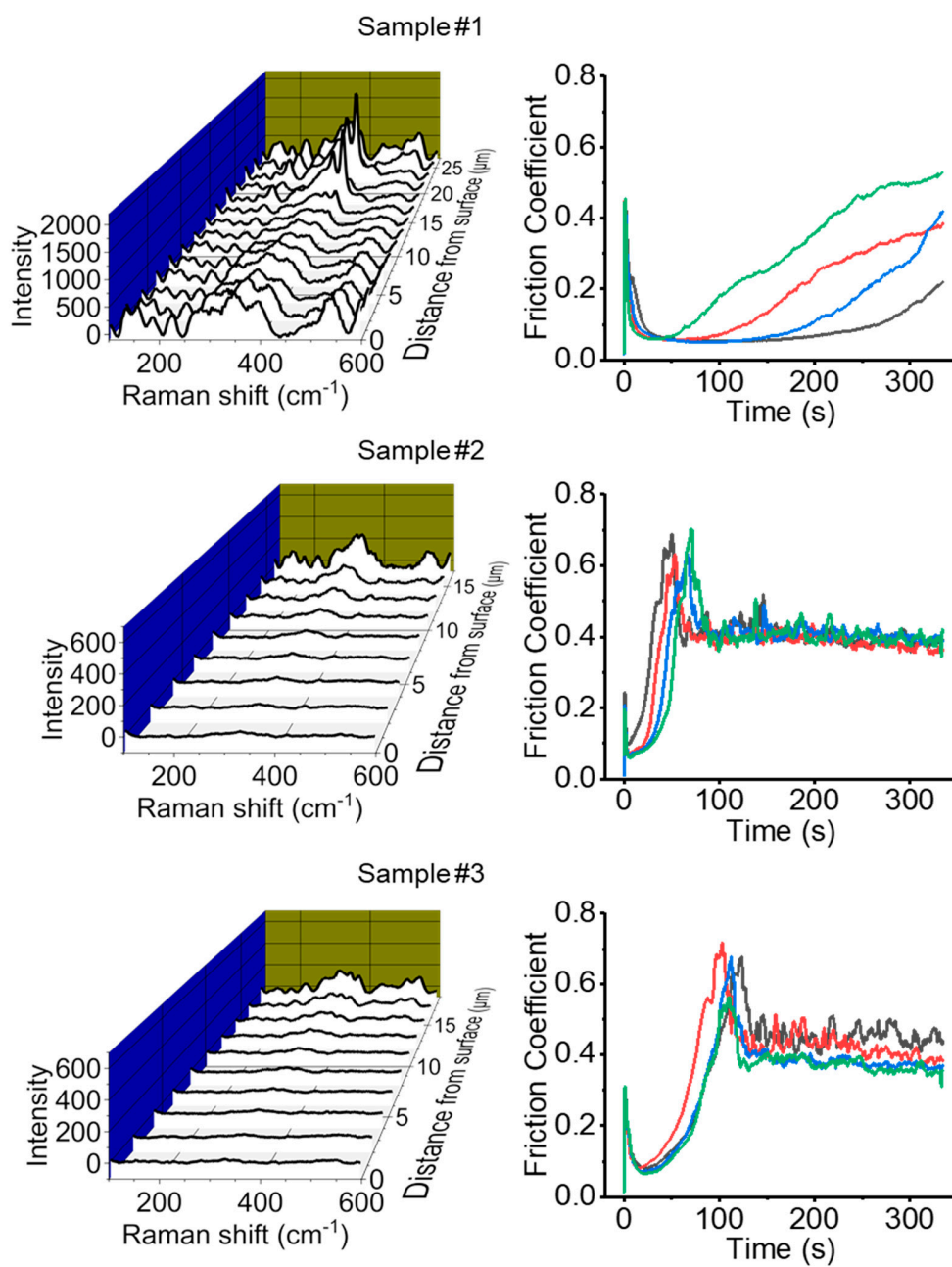


**Figure S18.** Raman linescans of a commercial MoS<sub>2</sub>/AAO film before (a) and after (b) heat treatment.

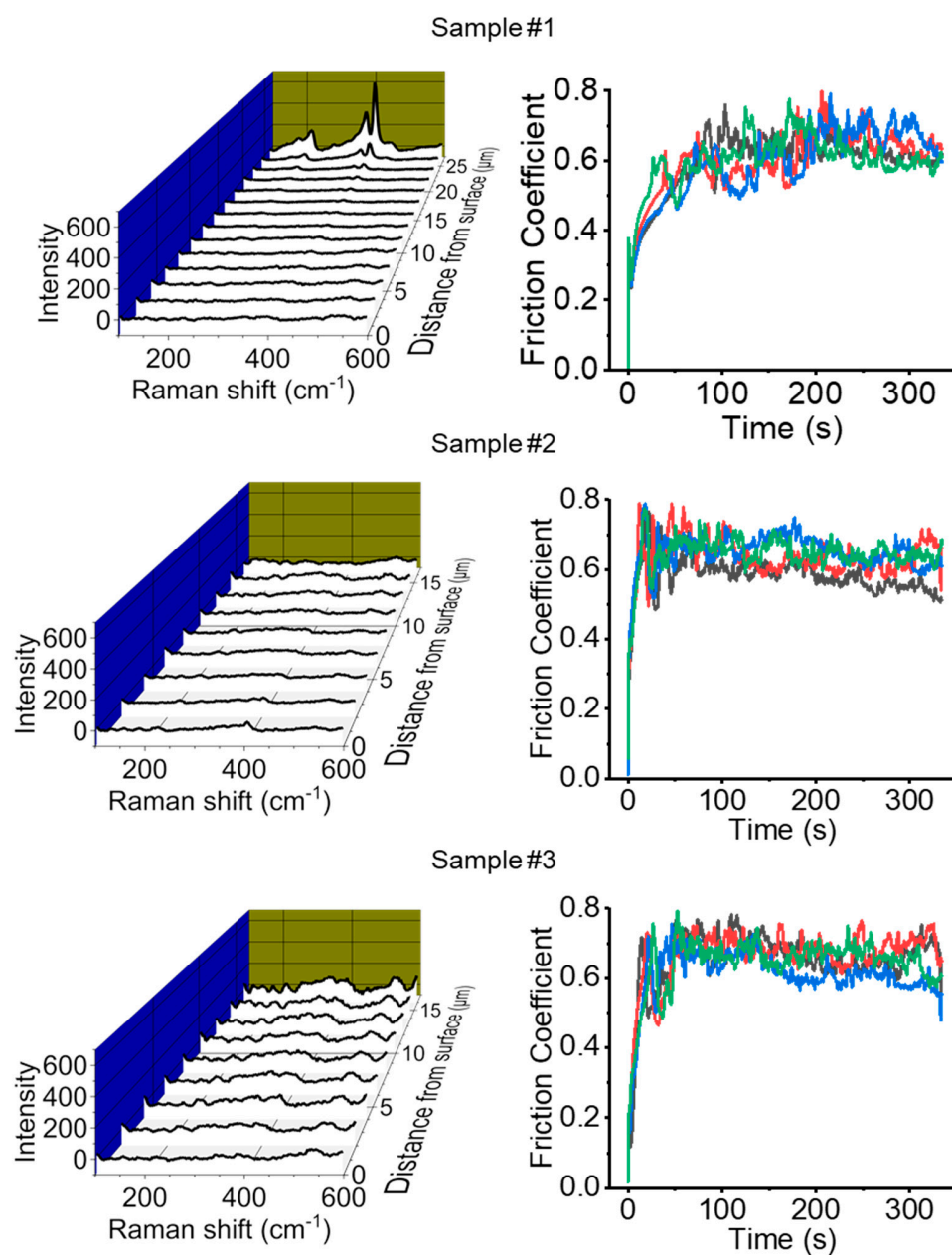


**Figure S19.** V-t curves for MoS<sub>x</sub> deposition on high-acid 120-minute anodized Al 7075, run in triplicate (sample numbers denoted on the graph).

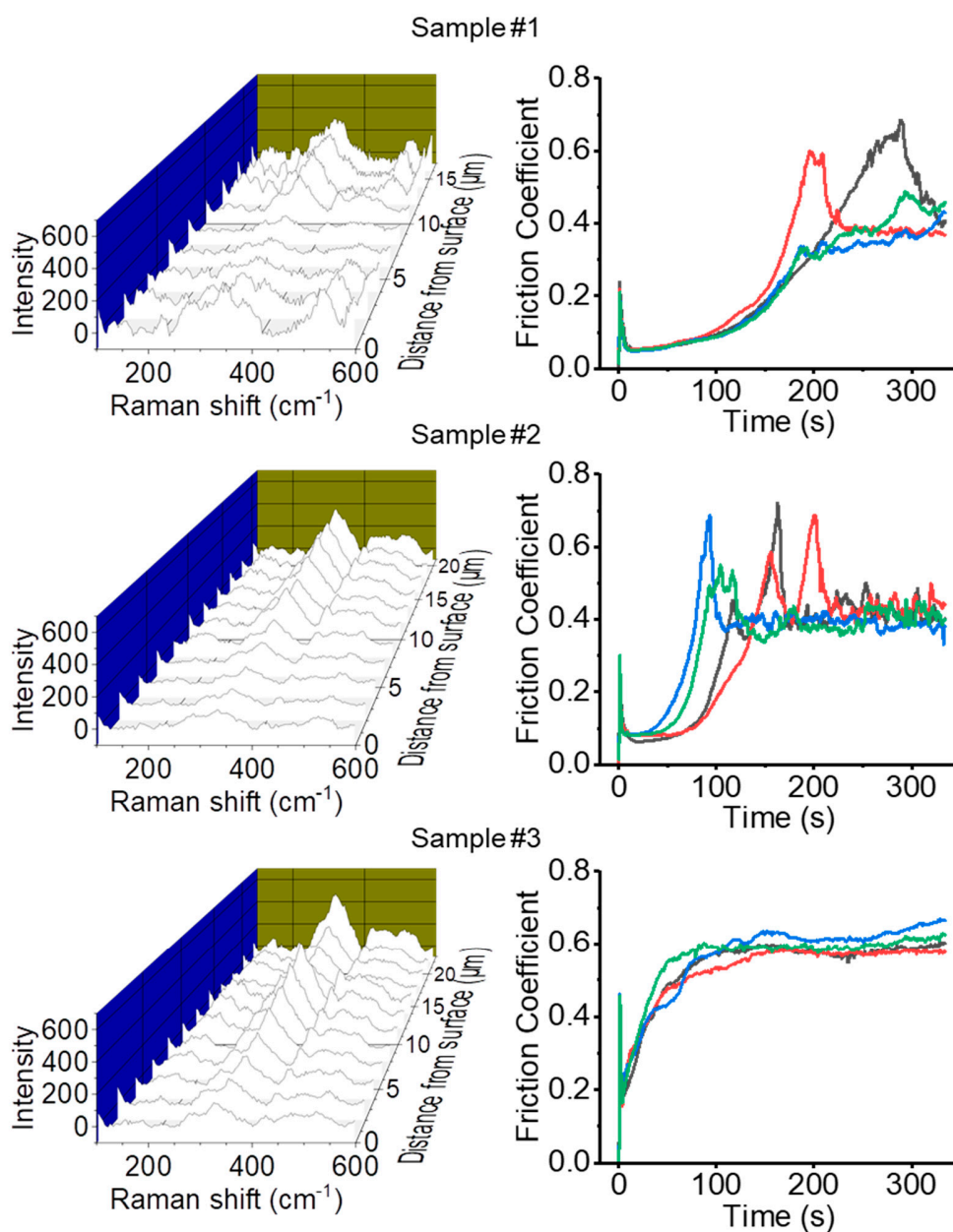




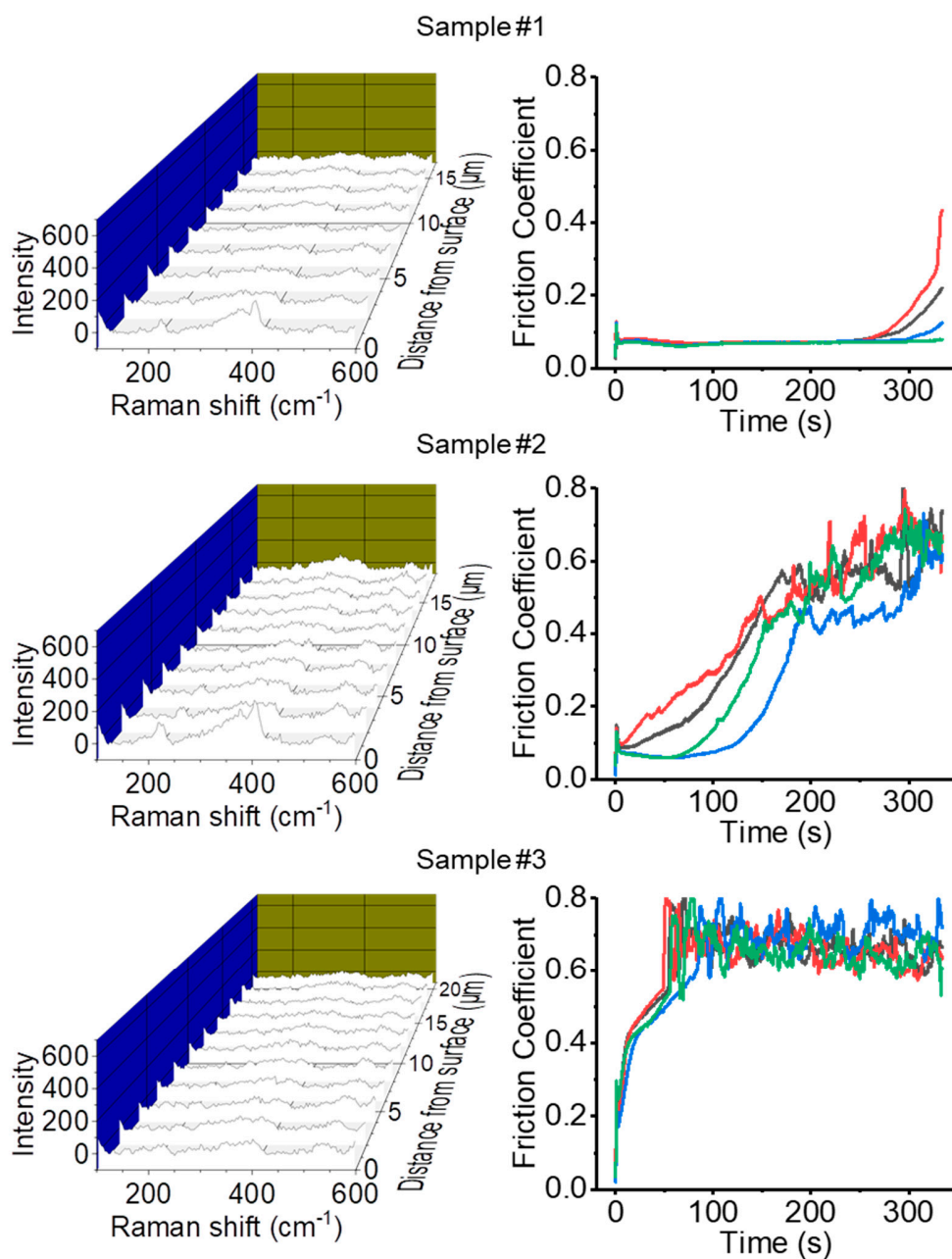
**Figure S20.** Raman-tribology comparison of 120-minute high-acid anodized Al 7075 samples with no acid pretreatment before heat treatment. Four scratches were performed per sample.



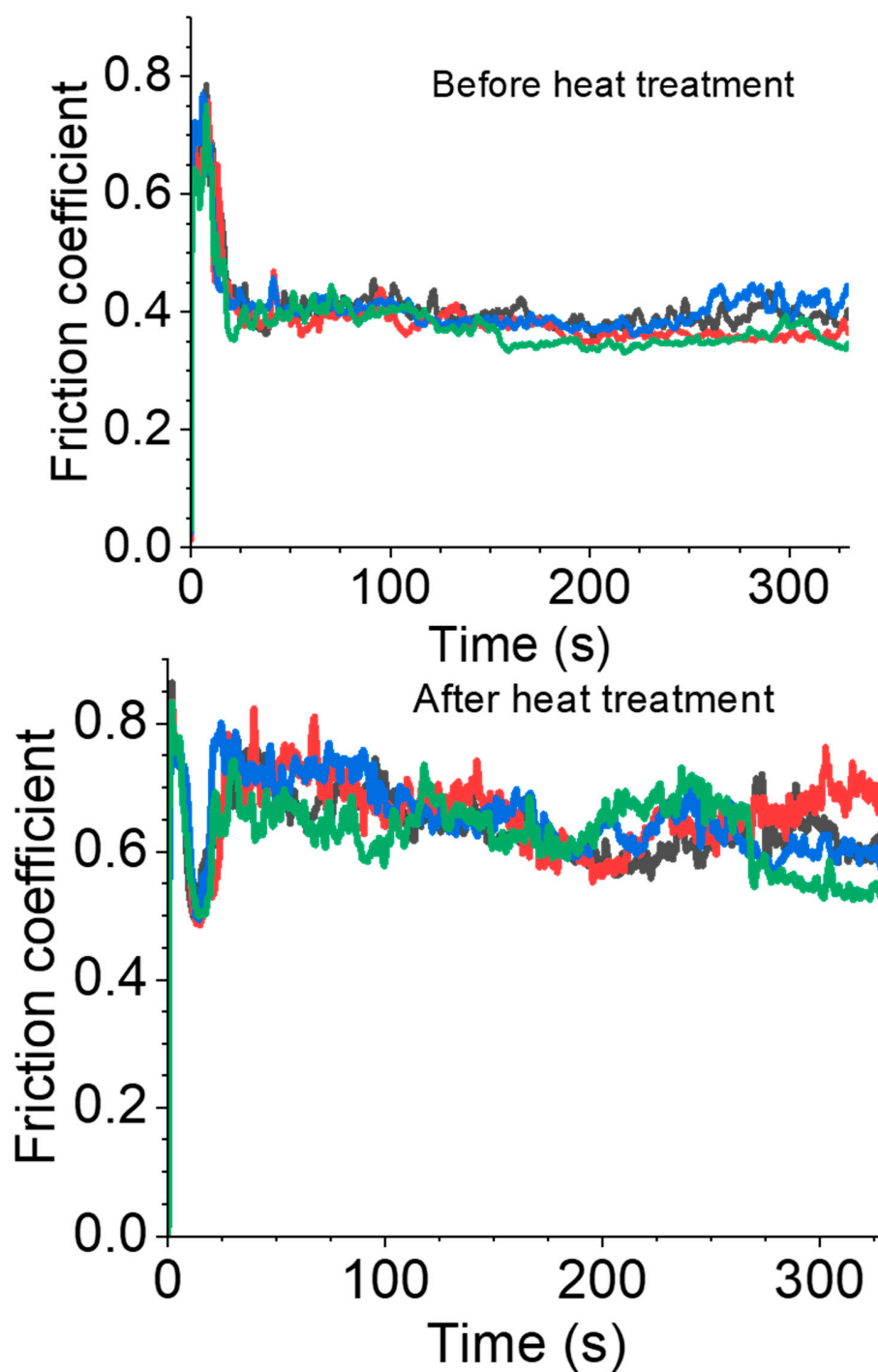
**Figure S21.** Raman-tribology comparison of 120-minute high-acid anodized Al 7075 samples with no acid pretreatment after heat treatment. Four scratches were performed per sample.



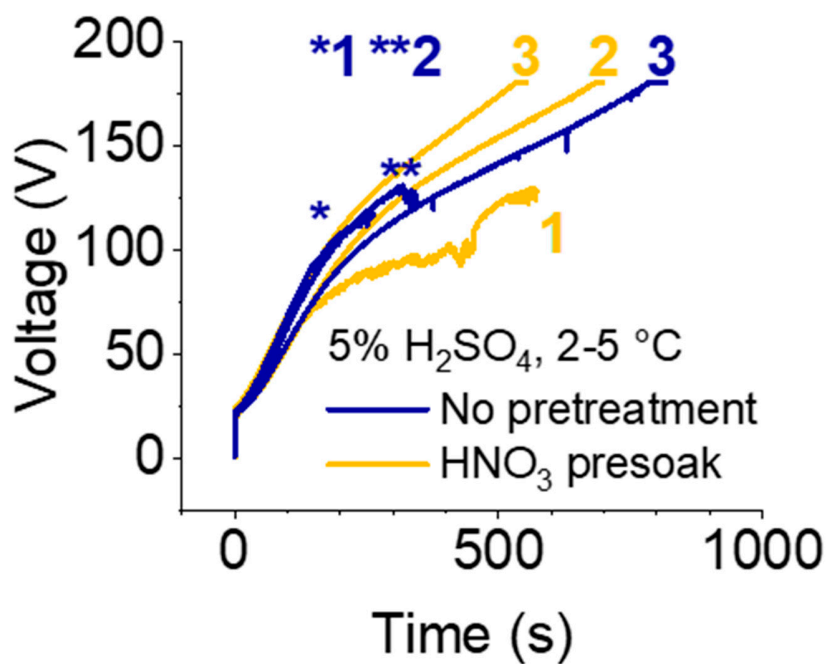
**Figure S22.** Raman-tribology comparison of 120-minute high-acid anodized Al 7075 samples with  $\text{HNO}_3$  presoaking before heat treatment. Four scratches were performed per sample.



**Figure S23.** Raman-tribology comparison of 120-minute high-acid anodized Al 7075 samples with  $\text{HNO}_3$  presoaking after heat treatment. Four scratches were performed per sample.

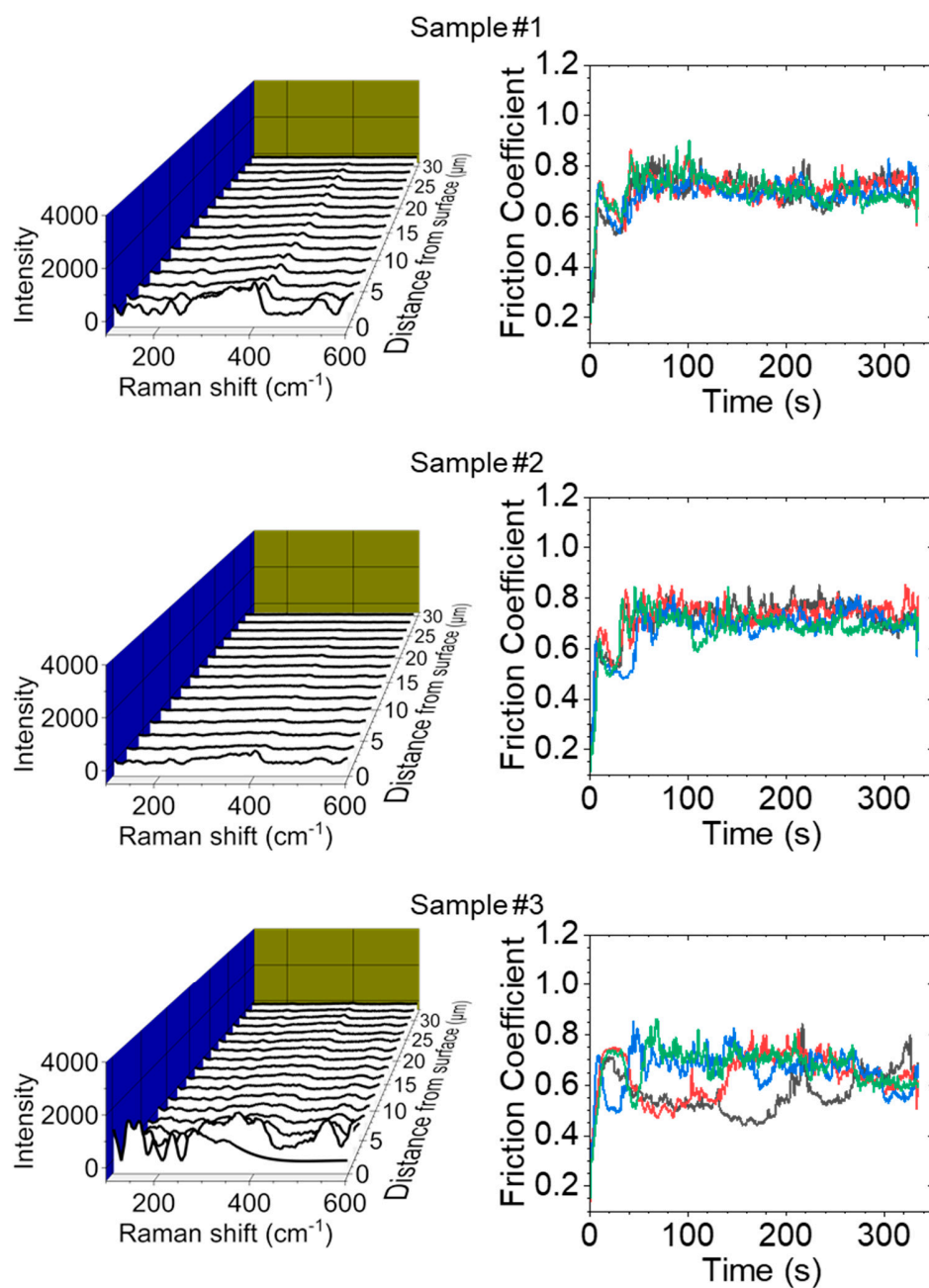


**Figure S24.** Reciprocating scratch tests of 120-minute high-acid anodized Al 7075 with no MoS<sub>x</sub> deposition a) before and b) after heat treatment. Asterisks denote film breakthrough.

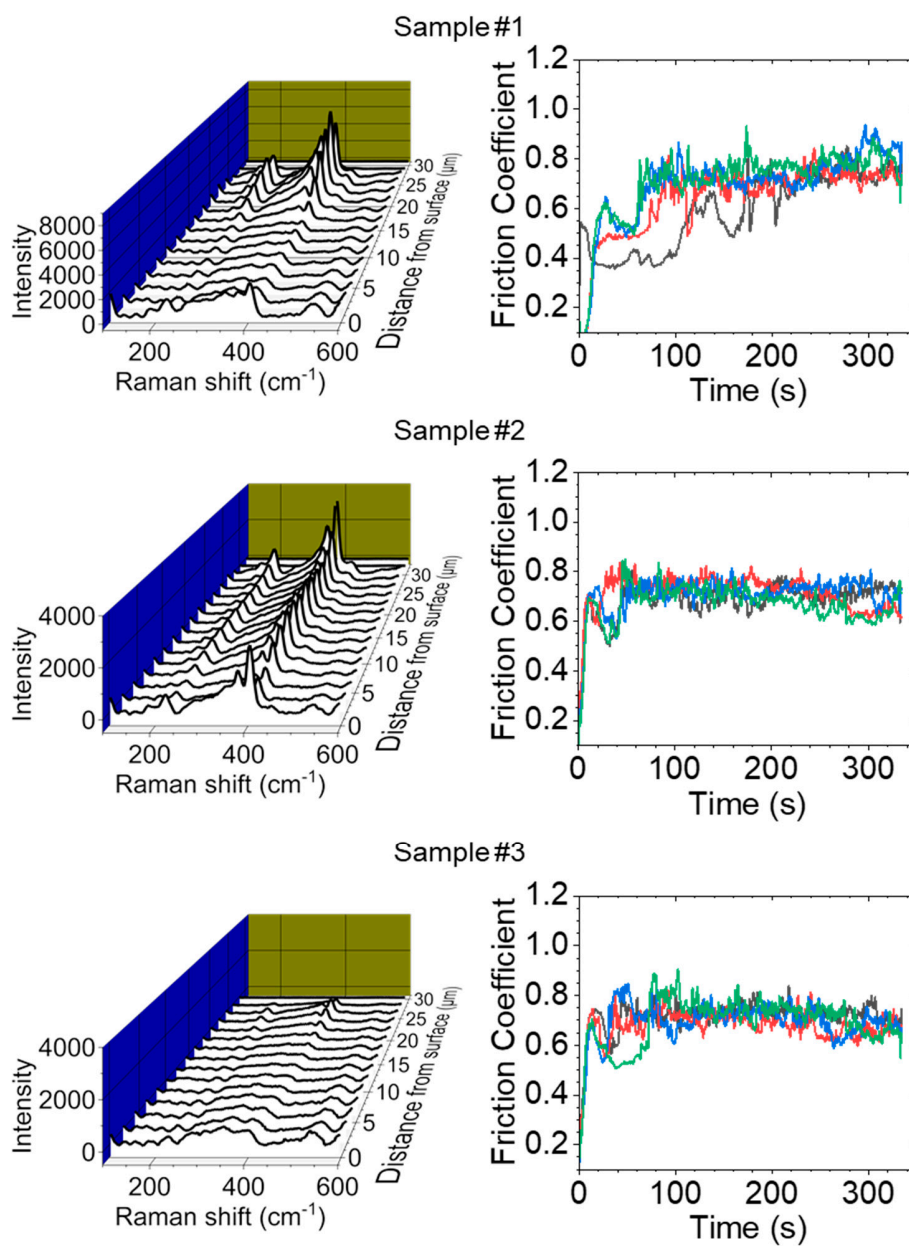


**Figure S25.** V-t curves for MoS<sub>x</sub> deposition on low-acid anodized Al 5052 (5% H<sub>2</sub>SO<sub>4</sub> at 2-5 °C) for 120 minutes. All samples were run in triplicate (sample numbers denoted in the figure).



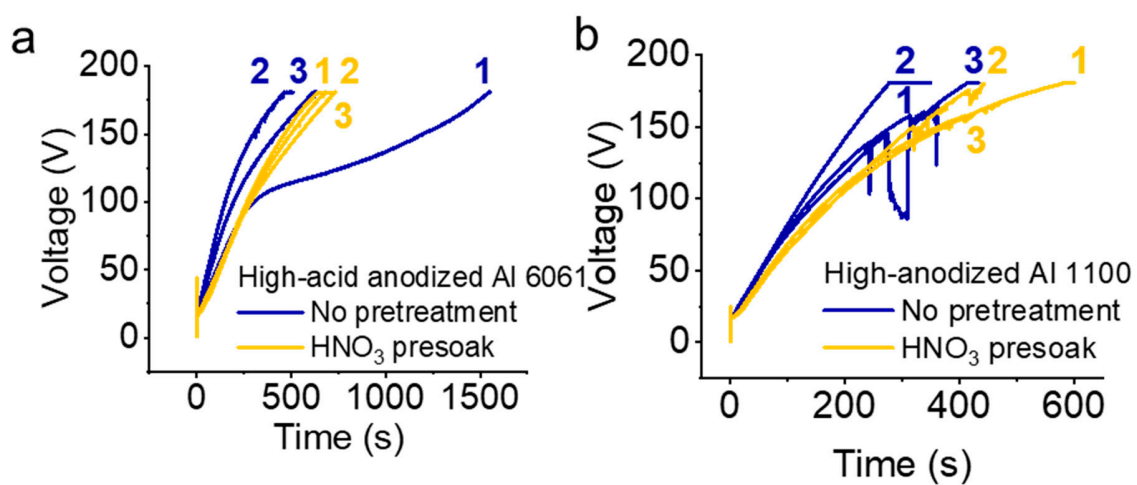


**Figure S26.** Raman-tribology comparison of 120-minute low-acid anodized Al 5052 samples with no acid pretreatment. Four scratches were performed per sample.

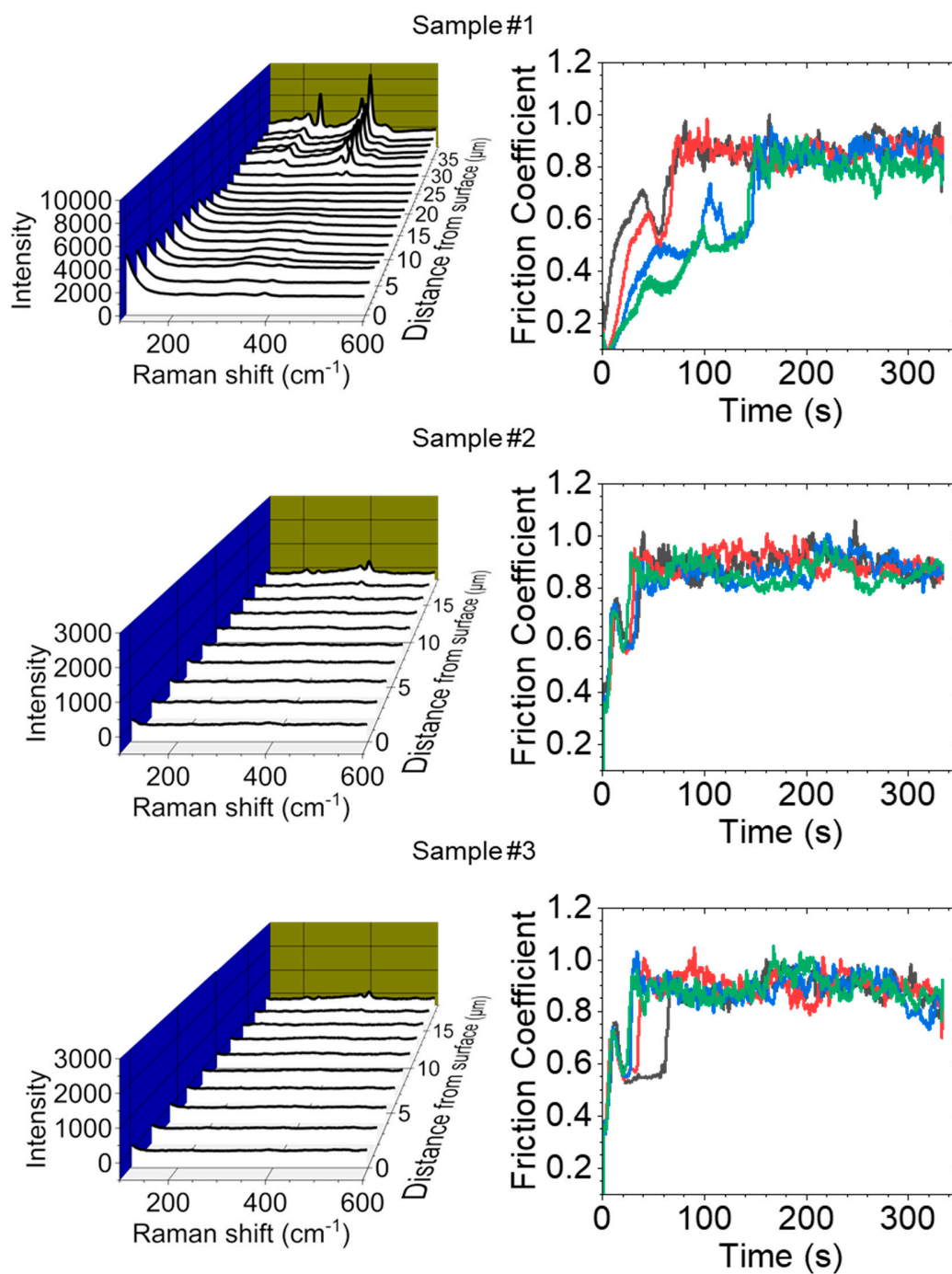


**Figure S27.** Raman-tribology comparison of 120-minute low-acid anodized Al 5052 samples with  $\text{HNO}_3$  pretreatment. Four scratches were performed per sample.

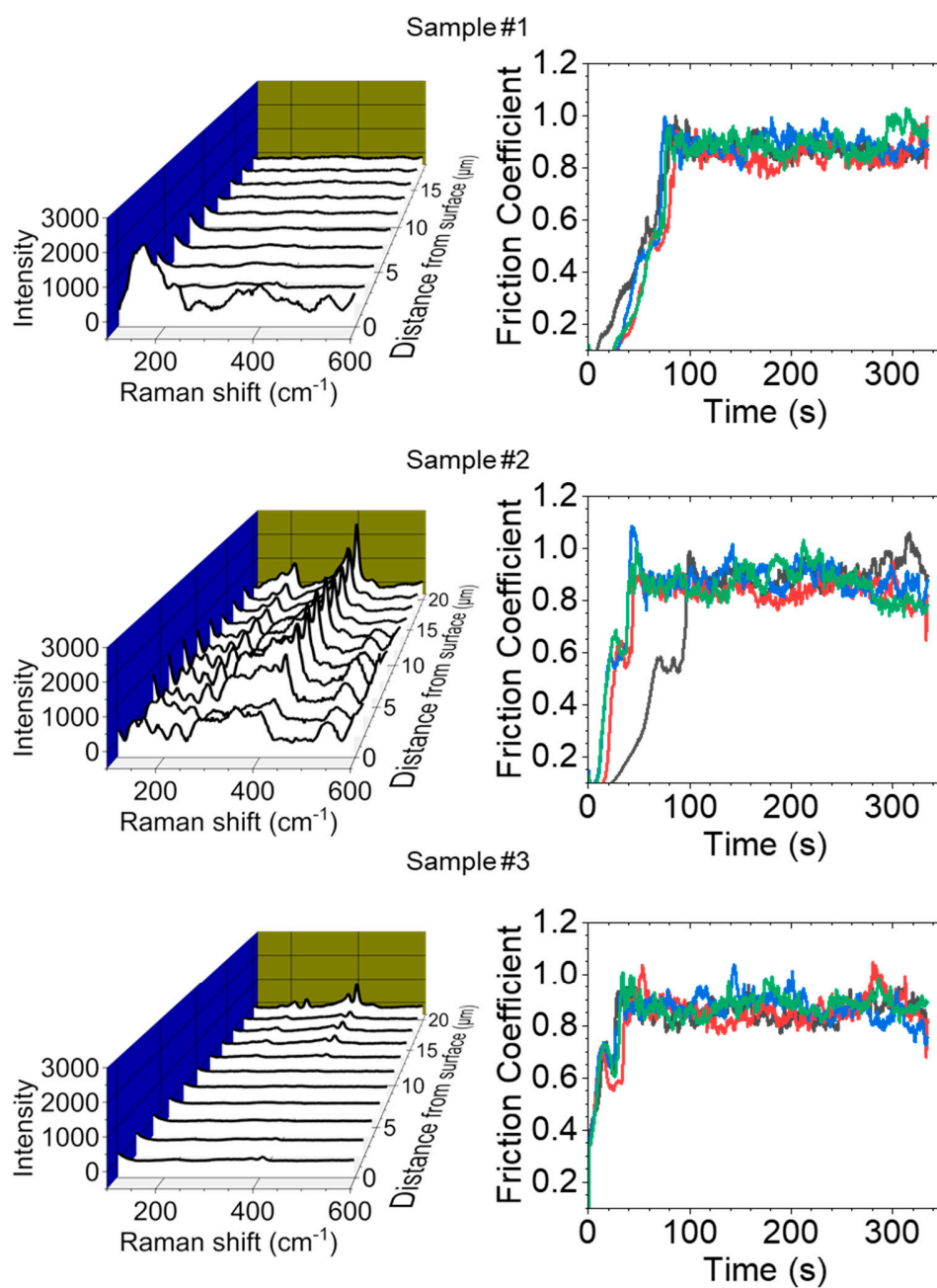




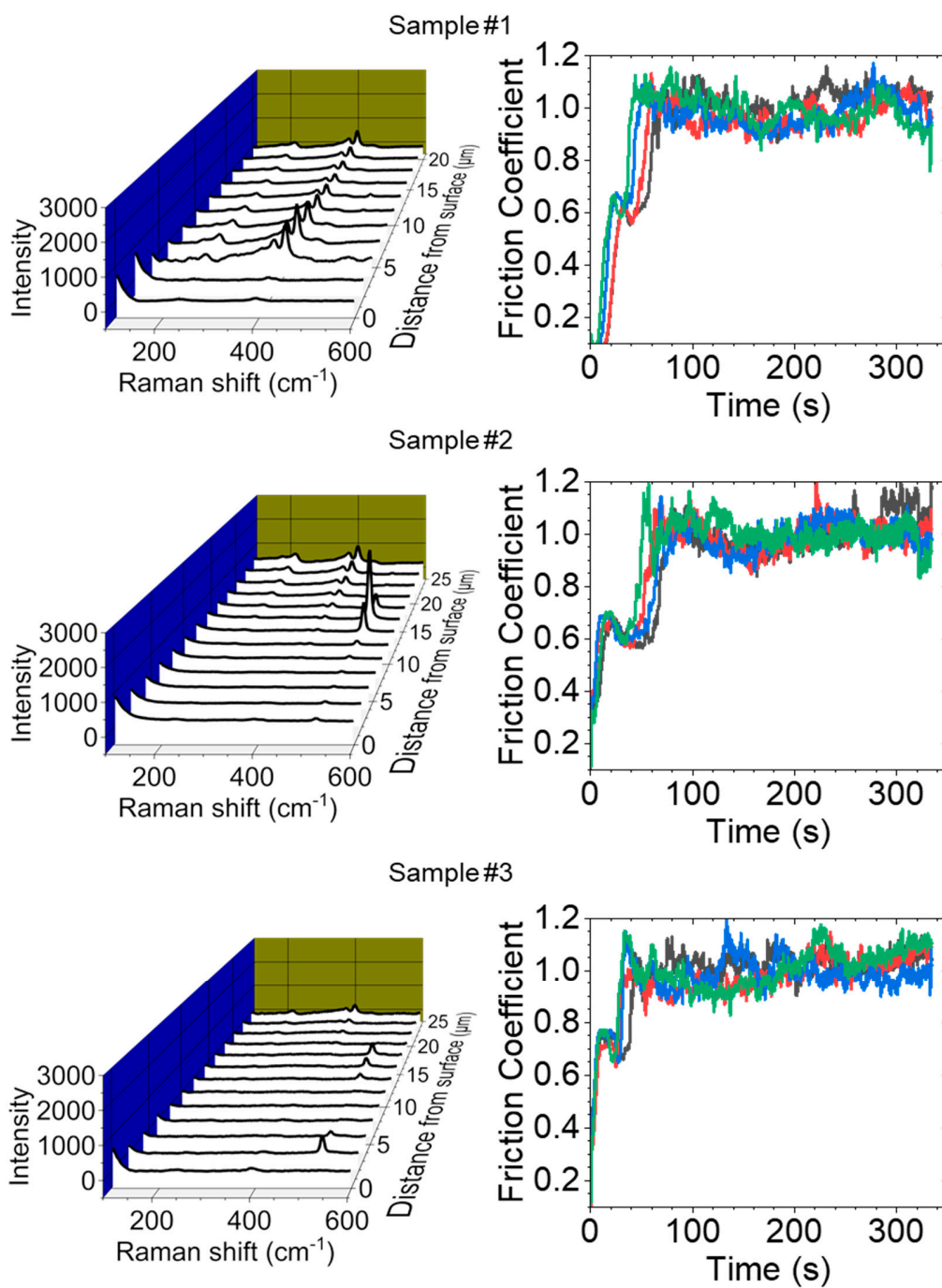
**Figure S28.** V-t curves for MoS<sub>x</sub> deposition on a) Al 6061 and b) Al 1100. All samples were run in triplicate (sample numbers denoted in the figure).



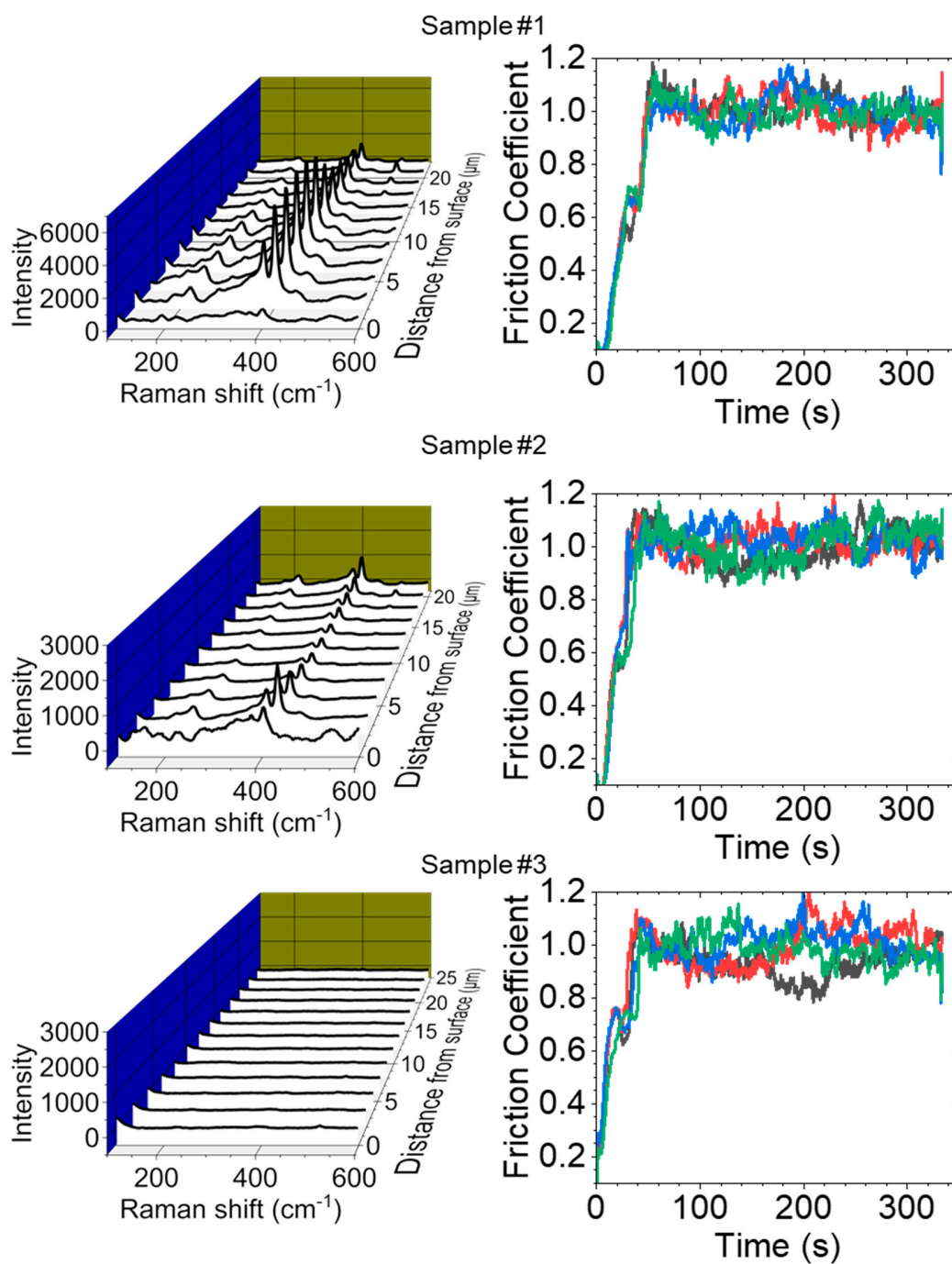
**Figure S29.** Raman-tribology comparison of 120-minute high-acid anodized Al 6061 samples with no acid pretreatment. Four scratches were performed per sample.



**Figure S30.** Raman-tribology comparison of 120-minute high-acid anodized Al 6061 samples with  $\text{HNO}_3$  pretreatment. Four scratches were performed per sample.



**Figure S31.** Raman-tribology comparison of 120-minute high-acid anodized Al 1100 samples with no acid pretreatment. Four scratches were performed per sample.



**Figure S32.** Raman-tribology comparison of 120-minute high-acid anodized Al 1100 samples with  $\text{HNO}_3$  pretreatment. Four scratches were performed per sample.