lubricants

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Supplementary Materials

High Temperature Microtribological Studies of MoS_2 Lubrication for Low Earth Orbit

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Supplementary Information 1 – Environmental Control Box Design Notes

SI 1.1 Physical Description

The environmental control box used in this study, shown in Figure SI1, was designed to provide a controlled temperature and gas environment. The temperature range was designed to reach 120°C while minimizing leaks to control local environment. The box primarily consisted of an aluminum base (a) and an acrylic lid (b**)**, machined and laser-cut respectively. The sample was attached to a ceramic disk-shaped sample stage via double-sided carbon tape and placed directly on top of the heating stage consisting a water jet-cut aluminum plate and the resistive heater (c). The heating stage was secured to the ECB via five PEEK screws to minimize vibration due to gas flow. The heating stage was insulated on five sides by polyurethane foam to isolate from the aluminum structure to ensure localized heating and protect the AFM stage from high temperatures. Of particular note, additional polyurethane foam was placed in front of the gas inlet (d) as a porous diffuser to further improve stability by preventing vibration due to gas flow. Flanges on the sides of the base (**e)** enabled the box to be held in place on top of the AFM stage by two rectangular magnets. The lid, attached to the base via screws for sample access, has a hole for the AFM head to access the sample. The lid features a rubber gasket (f) which is slightly smaller than the size of the bottom of the AFM head. This gasket deforms to allow the AFM piezo-electric stage to move the sample in X,Y during scanning with minimal gas leaking. Despite not being a perfect seal, the nitrogen environment consistently reached <1% RH during operation. The ambient temperature and humidity sensor was mounted on the face opposite to the gas inlet (g) to generate a representative measure of the humidity measurement. Self-adhesive thermocouple temperature sensors were attached to the heater plate and the side of the ECB near the contact with the AFM stage. Thermal simulations performed in ANSYS Workbench as well as tests performed inside the AFM confirmed that the temperature of the aluminum heating stage is statistically similar to the sample itself.

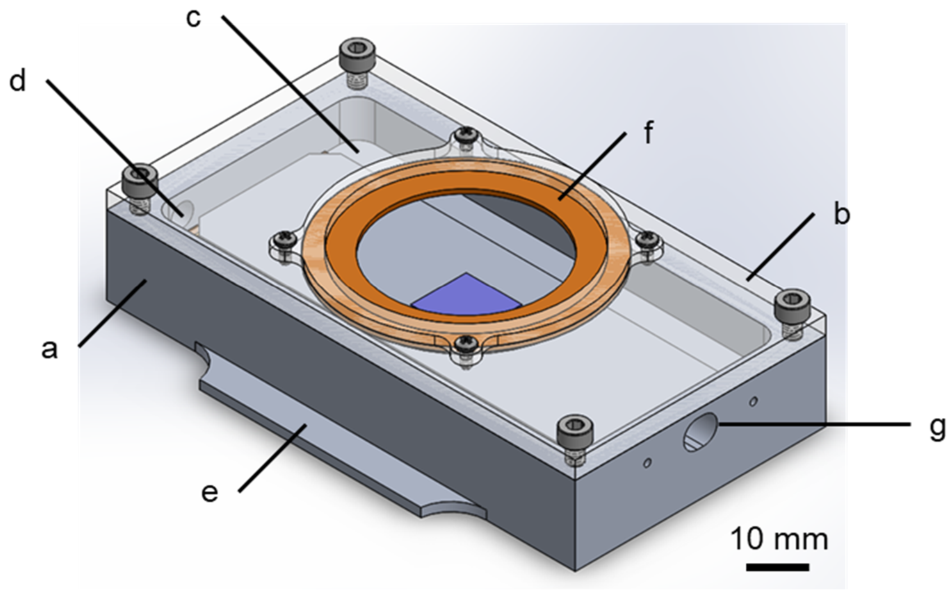

Figure SI1. Annotated CAD model of the environmental control box used in this study, including the: (**a**) aluminum base; (**b**) acrylic lid; (**c**) heater stage; (**d**) gas inlet; (**e**) flanges to hold the box in place via magnets; (**f**) hole for the AFM head and rubber gasket to prevent leaks; and g) hole for the temperature and humidity sensor. Not shown here: the polyurethane foam insulation and porous diffuser underneath the heater plate and thermocouple locations.

SI 1.2 Control System

The ECB was controlled via two independent systems: sample temperature control as well as emergency shut off to protect the AFM and user. The sample temperature control system was an open-loop controller consisting of the heater, a DC power supply, a K type thermocouple, and an Arduino Uno microcontroller. A standard curve of temperature as a function of voltage was measured during testing and fitted to obtain the empirical control equation $V(S) = -0.001642S^2 + 0.4715S - 10.81$, where V is the applied voltage in V and S is the set point in °C. An additional curve of temperature was created for the nitrogen tests to account for the cooling brought on by the lower temperature and the convection of the nitrogen. A similar approach was taken with respect to methodology and the resulting curve was found to be $V(S) =-0.0015x^2 +0.4374x-3.084$. The user then determined the voltage requirement for each temperature and the local temperature of the heater stage was displayed and recorded through the associated Arduino controller. Any deviations from the sample setpoint alerted the user via display messages. The emergency control system consisted of a K type thermocouple and the heater connected to a standard temperature controller. The K type thermocouple ensures the external temperature of the box never exceeds 50°C to protect the AFM and user from damaging temperatures. This emergency shut off was not required through the temperature range used herein.

Supplementary Information 2 – Numerical Analysis of System Instability

The system instability may be suggestive of an increased duration for the run-in phase until steady state operation is achieved. However, the standard deviation for friction is found to have little correlation with the duration of run-in as seen in Table SI1. This is especially apparent looking at the tests performed under dry nitrogen where the friction average and standard deviation both increase while the cycles to steady state decreases. As such, the duration of the run-in is more likely attributed to the temperature and the thermal energy lowering the barrier to optimal reconfiguration of the coating for lubrication.

Table SI1. Numerical analysis of the steady state friction average, standard deviation, and ratio, and duration of the run-in phase.

	Air							N_2		
	25 °C	**50 °C**	**70 °C**	**90 °C**	**100 °C**	**110 °C**	**120 °C**	**25 °C**	**100 °C**	**120 °C**
SS Friction Average	0.449	0.217	0.160	0.063	0.068	0.025	0.092	0.060	0.092	0.086
SS Friction StDev	0.022	0.032	0.009	0.005	0.006	0.003	0.011	0.003	0.008	0.005
StDev/Avg	0.049	0.149	0.058	0.072	0.085	0.113	0.117	0.054	0.088	0.060
Cycles to SS	61	48	43	28	30	21	33	35	18	20

SS = Steady State