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PREPARATION OF CHAMELEON COATINGS FOR SPACE AND AMBIENT ENVIRONMENTS (PREPRINT)

C.C. Baker, R.R. Chromik, K.J. Wahl, J.J. Hu, and A.A. Voevodin

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Preparation of Chameleon Coatings for Space and Ambient Environments

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Abstract

Tribological coatings of yttria stabilized zirconia (YSZ), gold, diamond like carbon (DLC) and MoS_2 were synthesized using magnetron assisted pulsed laser deposition (MSPLD). The coatings were synthesized in four-component and three-component combinations that included YSZ/Au/DLC/MoS_2, YSZ/Au/MoS_2, and YSZ/Au/DLC. A range of coating compositions was studied to explore coating optimization for low friction in varying environments (dry, humid and high temperature). For four-component YSZ/Au/DLC/MoS_2 coatings, the optimal compositions for friction adaptation between dry nitrogen and humid air included relatively high concentrations of the soft phase, Au (>20 at. %), and low amounts of the hard phases, DLC and YSZ. *Ex situ* Raman spectroscopy analysis indicates that friction adaptation involves a combination of both lubricating species, MoS_2 and carbon where transitions of DLC to graphitic-carbon and amorphous MoS_2 to its hexagonal phase occurs after cycling between both room temperature humid air and dry nitrogen. In large carbon concentrations (>30 at. %), the DLC component was found to be detrimental for friction in dry nitrogen and humid air, but promoted a longer coating wear life at 500°C. The three-component coating of YSZ/Au/MoS_2 performed well in both dry nitrogen and humid air, suggesting a synergism between Au and MoS_2, where carbon was not necessary for lubrication in humid air.

Keywords: Nanocomposite; Coatings; Tribology; Aerospace

1. Introduction

One current challenge in tribological coatings research is to obtain coatings with low coefficients of friction (c.o.f.) in varying environmental conditions. This is especially important for aerospace applications. Satellites that are designed for hard vacuum in space are also exposed to moisture during launch as well as extreme temperature fluctuations. Solid lubricants that impart low friction behavior in one environment do not generally perform well in another. For example, dichalcogenides like WS_2 and MoS_2, can provide low friction in vacuum conditions, but do not work well in high humidity environments by themselves [1,2]. Similarly, graphite can provide low friction in humid air but not in vacuum. For such applications a composite coating that can adapt itself to provide good friction and wear performance in multiple environments is desirable.

We have previously reported on adaptive "chameleon" nanocomposite coatings where excellent tribological properties are achieved in varying environments [3-10]. These coatings have a combination of solid lubricant components that give low coefficients of friction in both humid environments and in vacuum or dry nitrogen (simulating space environments). The coatings also have nanocrystalline oxide phases that impart high hardness and wear resistance. A nanocomposite system of particular interest includes yttria-stabilizied zirconia (YSZ), diamond like carbon (DLC), and MoS_2. This coating system provided good friction properties in dry and humid conditions and survived up to 10,000 cycles at elevated temperatures [10]. The addition of Au was found to be essential for lowering the coefficient of friction at high temperatures and extending the coating lifetime [10].

Previously, four-component coatings were studied over a relatively small range of compositions [10]. Here we report a study on the properties of a four-component system in a broader range of compositions, maintaining MoS_2 around 14-18% while varying Au, YSZ and DLC content. We also report on the three-component systems YSZ/Au/MoS_2

and YSZ/Au/DLC in order to examine the role of both MoS_2 and carbon for lubrication in this particular coating system. Measurements of the friction coefficient during humidity cycling and separate high temperature experiments were used to determine which coatings were good candidates for aerospace applications.

2. Experimental

YSZ/Au/DLC/MoS_2, YSZ/Au/MoS_2, and YSZ/Au/DLC coatings were prepared in a vacuum chamber using magnetron assisted pulsed laser deposition (MSPLD) [11]. The chamber was initially pumped down to a base pressure of 7×10^{-6} Pa or less. The samples were grown on various mirror-polished 24.5 mm diameter substrates including 440C steel, 52100 steel, M50 steel, (001) silicon, and Inconel 718. The substrates were cleaned in an acetone bath for 30 minutes prior to mounting in the chamber, where they were cleaned with energetic argon $\leq$ 800 V and Ti ions at $\leq$ 650 V. During deposition, substrates were biased at -150V with respect to ground and heated to either 150°C or 300°C. The purpose of the increase in deposition temperature was to promote coating crystallization.

Sectioned targets were used for laser ablation with quarter circles of YSZ (ZrO_2-5 wt.% Y_2O_3), MoS_2, and graphite, while Au was deposited using magnetron sputtering. For the four-component coatings, the target consisted of two quarter sections of YSZ, one quarter section of MoS_2, and one quarter section of carbon. For the three-component coatings the target consisted of two quarter sections of YSZ and one half section of either carbon or MoS_2. The laser ablation was performed with a KrF laser operated with radiation of 248 nm, 800 mJ in pulsed mode with 25 ns wide pulses, and a repetition rate of 40 Hz. Magnetron sputtering was performed with a Au target at powers of 3-6 W/cm^2. Composition control was achieved by varying the magnetron power to obtain between 10%-35% Au to the coatings. Depositions were performed in an Ar atmosphere. A filtered vacuum arc system was used to both clean the substrates and deposit a Ti transition layer between the substrate and coating. This is effective in providing good coating adhesion and film stress reduction [12]. A schematic of the deposition chamber with the vacuum arc

system is given in fig. 1**.** Here a Ti cathode was evaporated with 125 A current, generating a Ti plasma. Titanium ions were guided by an electromagnetic field around a 90° turn to filter out droplets from reaching the substrates. For cleaning, the substrates were biased at ≤ 650 V and bombarded with Ti ions from the arc source. The bias was then reduced stepwise to that of the growth bias, -150V, and a Ti/YSZ/C/MoS_2 transition layer was produced by gradually increasing the laser repetition rate to the YSZ/C/MoS_2 ablation target during deposition with the arc source. Finally, for sample deposition, the arc source was shut off. The Ti layer is generally ~100 nm thick and the graded layer is ~300nm thick. The coatings were generally ~1-2 μm thick.

Chemical analysis of the coatings was investigated with a Surface Science Instruments M probe x-ray photoelectron spectrometer (XPS). Prior to analysis the samples were sputter cleaned with 1000 eV Ar^+ ions. Coating compositions were calculated using intensity factors from area analysis of C 1s, O 1s, Zr 3d doublet, Au 4f doublet Mo 3d doublet, S 2p doublet, and yttria 3d doublet peaks. Coating microstructure was analyzed with x-ray diffraction (XRD) using a Rigaku diffractometer and Cu k_α radiation in θ-2θ mode. Transmission electron microscopy (TEM) and selected area diffraction (SAD) was performed on a Philips CM200 microscope to identify phases present in the coatings. Micro-Raman spectroscopy was performed on a Renishaw Ramascope 2000 equipped with a 514.5 nm laser. Raman shift scattering was recorded from 200 to 2000 cm^{-1}.

Friction coefficient was measured using ball on disc tribometers with a 1 N load applied to M50 steel balls (radius = 3.175 mm) and a sliding rate of 0.4 m s^{-1}. The Hertzian contact stress was estimated to be ~0.43 GPa for steel vs. steel. The tests were run during cycling in air at 40% RH, nitrogen at <1% RH. High temperature tests (in air at 500°C) used a Si_3N_4 ball (radius = 3.175 mm) loaded to 1 N and sliding speed of 0.2 m/s.

Nanoindentation was performed using a Hysitron Ubi scanning indenter. Indentations were conducted on sections of wear tracks that had been run for 100 cycles. These areas were used because there was minimal wear and the surface was smoother than the as-

prepared coating. An exception was the YSZ/Au/DLC coating, which was much smoother than the other coatings; indentation experiments on this coating were done on the as-prepared sample. Indentation load functions consisted of loading at 0.2 mN/s to either 1.0 mN or 2.0 mN maximum load, a hold segment of 2 seconds, followed by unloading at 0.2 mN/s. The load function with 2.0 mN maximum load resulted in depths of penetration on the order of 120 nm, or approximately 5 – 15% of the total coating thickness. Arrays of indents were spaced 5 μm apart and were followed by post-indentation imaging using the scanning capability of the indenter system. Indents found to be near coating defects or obvious track damage or debris were discarded from each dataset. Average hardness and modulus values reported here were calculated from at least 30 indents, all analyzed using the standard Oliver and Pharr method [13]. The indenter tip was a diamond Berkovich with a nominal tip radius of 100 nm. Calibration of the tip area function was conducted on a fused silica specimen.

3. Results

3.1 Preparation, Chemistry, Structure and Mechanical Properties

Eight different coating compositions were studied and are listed in Table 1. The coatings are classified as “high Au” and “low Au”. The high Au coatings had 23-34 at. % Au, and the low Au coatings had 10-14 at. % Au as determined by XPS. Note that among the high Au coatings are the three-component coatings YSZ/Au/MoS_2 and YSZ/Au/DLC.

XPS results were examined in more detail to determine the chemical state of the various elements in the coatings. The C 1s peak was found at binding energies of 284.5 eV for all coatings which is consistent for C-C bonds with no evidence for carbide formation. The Zr $3d_{5/2}$ peak was consistent with ZrO_2 which has a binding energy of 182.4 eV, and the Au doublet was consistent with literature values for metallic Au. The sulfur 2p doublet had a binding energy of 161-163 eV, consistent with a sulfide. However, we note that the Mo $3d_{5/2}$ peak was found at 228-228.3 eV indicating that Mo exists with a stoichiometry as

MoS_x with x<2. This may be a result of preferential sputtering of the sulphur during sample cleaning in the XPS chamber [14].

A HRTEM image corresponding to a low Au sample ($(YSZ)_{0.33}(Au)_{0.14}(MoS_2)_{0.15}(C)_{0.36}$) is shown in fig. 2 along with a selected area electron diffraction pattern. The image shows dark regions composed of Au nanocrystals in an amorphous matrix. The interplanar spacing is highlighted and also indicates Au. From these TEM studies only Au was found to exist in nanocrystalline form and carbon, YSZ, and MoS_2 exist in a mostly amorphous state within the region examined. In the SAD pattern rings are indexed to Au.

XRD results for two samples are presented in fig 3. The patterns correspond to a high Au sample ($(YSZ)_{0.42}(Au)_{0.31}(MoS_2)_{0.16}(C)_{0.11}$) and a low Au sample ($(YSZ)_{0.59}(Au)_{0.10}(MoS_2)_{0.18}(C)_{0.11}$). Broad peaks corresponding to Au are evident for both coatings. For the low Au sample with 59 % YSZ the (111) ZrO_2 peak is evident at $2\theta \approx 30°$, which gives a close match to the P4m2 tetragonal symmetry with lattice parameters a=0.512 nm and c=0.525 nm [15,16]. This sample represents the maximum in YSZ obtained for all coatings in this study. All other samples showed no diffraction peak for YSZ and thus these elements were part of the amorphous matrix, consistent with prior studies of YSZ/Au naoncomposites [9,17]. Note that a small peak for metallic molybdenum is also evident. This may be the result of occasional Mo droplets from the PLD process [10].

Nanoindentation measurements of the hardness and modulus of the low and high Au coatings revealed distinct differences between the two coating sets (see Table 1). The high Au YSZ/Au/DLC/MoS_2 coatings were found to have moduli of about 70-90 GPa and hardness values between 3 and 4 GPa. The YSZ/Au/MoS_2 coating had similar mechanical properties to the four-component high Au coatings. The YSZ/Au/DLC coating was much harder and stiffer than the other high Au coatings. This coating was more similar in mechanical properties to the low Au coatings, which were found to have moduli of roughly 100 – 130 GPa and hardness values of 5 – 6 GPa.

3.2 Tribological Properties

3.2.1 Four-component Coatings

In Table 2 we present the average friction coefficient results for all coatings. The values were determined after 10000 sliding cycles in humid air and dry nitrogen. The table also gives values for the friction and cycles to failure in air at 500°C for several coatings. Friction spiking and coating failure are also indicated with asterisks.

From all of the coatings studied, the four-component coatings with high Au content had the lowest and most stable friction. On the other hand, low Au coatings with either high carbon or high YSZ content were found to have friction spikes and in one case an extended run-in stage at high friction (μ ~0.4). Spiking and extended run-in occurred in dry nitrogen for the coating with 36 at. % carbon and low Au. Spiking also occurred in humid air for two coatings with high YSZ content ($\geq$ 45%) and low Au. An example of two coatings showing spiking in humid air and extended run-in and spiking in dry nitrogen and is given in figs. 4a and 4b respectively.

In tribological testing of the nanocomposites at 500°C, low friction of ~0.1-0.3 was observed for the coatings tested (Table 2). Wear lives at this temperature were between 700 and 6200 sliding cycles.

In order to simulate an aerospace application, friction tests were performed in varying humidity environments. For these experiments, the friction coefficient was recorded for up to 120,000 sliding cycles while the environment was repeatedly cycled between dry nitrogen at <1% RH and air at 40% RH. A typical example is given for a four-component coating in Figure 5, where cycling between the two conditions occurred every 20,000 cycles. The coefficient of friction for an optimal coating was observed to be ~0.02-0.03 in dry nitrogen and 0.1-0.15 in humid air. The coating systems have been found to exhibit

these low friction properties from 100,000 to 120,000 cycles depending on coating thickness.

Typical Raman spectra from the as-deposited coating, the wear track surface and the transfer film formed on the ball counterface for sample $(YSZ)_{0.33}(Au)_{0.34}(MoS_2)_{0.14}(C)_{0.17}$ taken after sliding in both environments is given in Fig**.** 6. Before sliding, only a weak carbon signature was obtained from the coating. After sliding, the wear track and transfer film showed peaks consistent with MoS_2 and graphite-like carbon, especially the transfer film. The broad overlapping peaks centered at 1380 and 1530 cm^{-1} correspond to the D and G bands found in graphite-like carbon, while the peaks found in the region between 380-410 cm^{-1} correspond to hexagonal MoS_2 [18]. With both species found in the spectrum after cycling between the two environments, the friction is likely controlled by both species acting synergistically.

3.2.2 Three-component Coatings

To test the endpoints of the friction behavior as a function of coating composition, coatings were deposited without carbon ($YSZ/Au/MoS_2$) and without MoS_2, (YSZ/Au/DLC). Both coatings were cycled in varying environments and the coefficient of friction measured. The YSZ/Au/DLC coating failed immediately in dry nitrogen, where the c.o.f. was 0.4. This coating also failed after only several hundred sliding cycles in humid air as well, with the c.o.f greater than 0.2. Conversely, the $YSZ/Au/MoS_2$ coating had a friction performance that was comparable to the best $YSZ/Au/DLC/MoS_2$ coatings in this study in either environment as well as cycling tests (Fig. 7**)**.

High temperature results for the three-component coatings are also included in table 2. In this case the friction for both three-component coatings was comparable to the four-component coatings; however, the YSZ/Au/DLC coating had a slightly longer wear life than the $YSZ/Au/MoS_2$ coating.

4. Discussion

The high Au four-component coatings had the best friction performance in cycling between dry nitrogen (c.o.f = 0.01-0.04), and humid air (c.o.f.= 0.1-0.13). These coatings had moduli of 70-90 GPa and hardness values between 3 and 4 GPa. The low Au coatings were harder, had higher elastic moduli, but exhibited friction spiking. *In situ* tribometry studies have shown that these spikes are the result of transfer film instabilities initiated by plowing events in the coating [19]. Increasing the Au content of the coatings eliminated the friction spiking and decreased the c.o.f. in dry nitrogen. While the increased Au content decreased hardness and modulus, it also likely decreased fracture toughness, as shown previously [10]. How this impacts friction spiking is not clear, although it is possible that it may have improved transfer film formation and adherence [20]. The improved room temperature performance for high Au content coatings did not translate to better performance at high temperature.

The YSZ/Au/MoS_2 coating was found to have nearly identical friction behavior as the optimal four-component coatings in both dry nitrogen and humid air environments. We note that in a prior study, YSZ/MoS_2 films without Au showed poor friction performance in air, which was improved by the addition of Au to the matrix [10]. These results are consistent with evidence for a synergistic effect for MoS_2 lubrication when Au is present. This is consistent with previous work on Au-doped MoS_2 films [21-23], as well as studies showing that metal dopants in MoS_2 enhances the tribological performance in both dry [23-27], and humid air [28-30]. Interestingly, the YSZ/Au/MoS_2 film from this study outperformed prior similar films in room temperature conditions (both dry and humid environments) [10]. The three component coating without MoS_2 (YSZ/Au/DLC) showed poor friction performance at room temperature conditions. The performance of this coating contrasts previous studies where friction coefficients of 0.25-0.35 were recorded in ambient conditions. The carbon and Au content of the present film (> 60%) is far greater than the previous films (~20% [10]), suggesting that the total C and Au content should be limited in this composition range for coatings without MoS_2 .

In humid air, carbon is generally thought to be the predominant species in the transfer film that imparts low friction, while in dry nitrogen MoS_2 is thought to be the most active species determining the friction behavior. Past researchers have reported possible synergistic effects for combinations of Graphite/MoS_2 [31], PTFE/MoS_2 [32], and for WC/DLC/WS_2 systems [6, 33-35], where it was shown that these mixtures contributed to improved lubrication and provide longer wear life in humid environments. Our Raman spectra results show that for the four-component YSZ/Au/DLC/MoS_2 coatings both species are generally present after cycling between the two environments, similar to that observed by Wu et al. [34]. Thus we find that both the four-component YSZ/Au/DLC/MoS_2 and three-component YSZ/Au/MoS_2 coatings provided effective lubrication in all three environments tested. For these coatings, the value of carbon in the coatings may lie more in longer wear life at elevated temperatures than for lubrication in humid environments. Finally, we conclude that although the three-component YSZ/Au/MoS_2 performed very well in dry nitrogen and humid air, the four-component YSZ/Au/MoS_2/DLC coatings had the broadest range of applicability for lubrication and extended wear life in all environments including high temperature.

5. Summary

Coatings of YSZ/Au/DLC/MoS_2, YSZ/Au/MoS_2 and YSZ/Au/DLC were synthesized using a hybrid magnetron assisted pulsed laser deposition process. Based on our results, the following summary is made:

Four-component coatings

- Coatings with Au composition > 20 at. % (high Au) performed the best by giving low friction in both dry nitrogen and humid air environments. These coatings had lower moduli and hardness suggesting that mechanical properties are also a factor in friction cycling performance.

- Coatings with low Au and with carbon content of ≥ 30 at. % or with YSZ content of ≥45 at. % performed poorly in either dry nitrogen or humid air environments.
- Upon environmental cycling, Raman analysis showed peaks indicating that both MoS_2 and graphite-like carbon were formed in the transfer film, indicating that they work together to lower friction in room temperature environments.
- The hard coating samples had the greatest cycles to failure in 500 °C air.

Three-component Coatings

- Coatings with MoS_2 and no carbon performed optimally in cycling environments of dry nitrogen and humid air. These coatings did not perform as well at high temperatures as coatings with carbon and no MoS_2.

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Table Captions

Table 1. Chameleon coating compositions and hardness and modulus. The coatings are classified as “high Au” and “low Au”.

Table 2. Average friction coefficient results in humid air, dry nitrogen, and air at 500°C. Coating cycles to failure at 500°C is also given.

Figure Captions

Fig. 1. MSPLD deposition chamber to produce chameleon nanocomposite coatings.

Fig. 2. TEM and diffraction pattern of coating $(YSZ)_{0.33}(Au)_{0.14}(MoS_2)_{0.15}(C)_{0.36}$ showing Au grains.

Fig. 3. XRD patterns for two nanocomposites coatings.

Fig. 4a. Friction coefficient vs. sliding cycles showing friction spikes for coating $(YSZ)_{0.59}(Au)_{0.10}(MoS_2)_{0.18}(C)_{0.11}$ tested in humid air.

Fig. 4b. Friction coefficient vs. sliding cycles showing extended run-in and friction spikes for sample $(YSZ)_{0.33}(Au)_{0.14}(MoS_2)_{0.15}(C)_{0.36}$ tested in dry nitrogen.

Fig. 5. Friction coefficient vs. sliding cycles for coating $(YSZ)_{0.33}(Au)_{0.34}(MoS_2)_{0.14}(C)_{0.17}$ during cycling between dry nitrogen and humid air.

Fig. 6. Raman spectra for coating $(YSZ)_{0.33}(Au)_{0.34}(MoS_2)_{0.14}(C)_{0.17}$ both as prepared and for a wear track after humidity cycling from dry to humid to dry. Also, the Raman spectrum from a transfer film formed on the steel counterface during the same test.

Fig. 7. Coefficient of friction vs. sliding cycles for a three-component YSZ/Au/MoS_2 coating tested during cycling between dry nitrogen and humid air.

Tables

Sample Composition	Growth Temp °C	Hardness GPa	Reduced Modulus GPa
High Au			
$(YSZ)_{0.34}(Au)_{0.24}(MoS_2)_{0.17}(C)_{0.24}$	150	3.0 ± 0.4	66 ± 7
$(YSZ)_{0.42}(Au)_{0.31}(MoS_2)_{0.16}(C)_{0.11}$	300	3.9 ± 0.2	90 ± 4
$(YSZ)_{0.33}(Au)_{0.34}(MoS_2)_{0.14}(C)_{0.17}$	150	3.2 ± 0.3	91 ± 8
$(YSZ)_{0.41}(Au)_{0.23}(MoS_2)_{0.35}$	150	3.4 ± 0.5	74 ± 4
$(YSZ)_{0.39}(Au)_{0.28}(C)_{0.31}$	150	7.0 ± 1.0	110 ± 10
Low Au			
$(YSZ)_{0.33}(Au)_{0.14}(MoS_2)_{0.15}(C)_{0.36}$	150	4.8 ± 0.3	105 ± 9
$(YSZ)_{0.45}(Au)_{0.10}(MoS_2)_{0.14}(C)_{0.30}$	150	6.3 ± 0.8	121 ± 12
$(YSZ)_{0.59}(Au)_{0.10}(MoS_2)_{0.18}(C)_{0.11}$	300	5.2 ± 0.7	133 ± 11

Table 1.

Sample Composition	Thickness	C.O.F humid air	C.O.F dry nitrogen	C.O.F Air 500°C	Cycles to Failure 500°C
High Au					
$(YSZ)_{0.34}(Au)_{0.24}(MoS_2)_{0.17}(C)_{0.24}$	2.67	0.1-0.13	0.02	------	-----
$(YSZ)_{0.42}(Au)_{0.31}(MoS_2)_{0.16}(C)_{0.11}$	1.73	0.1-0.11	0.03-0.04	0.2-0.3	700
$(YSZ)_{0.33}(Au)_{0.34}(MoS_2)_{0.14}(C)_{0.17}$	1.45	0.1-0.11	0.02-0.03	------	-----
$(YSZ)_{0.41}(Au)_{0.23}(MoS_2)_{0.35}$	3.01	0.1	0.01-0.02	0.1-0.3	1200
$(YSZ)_{0.39}(Au)_{0.28}(C)_{0.31}$	1.19	**	**	0.15	1800
Low Au					
$(YSZ)_{0.33}(Au)_{0.14}(MoS_2)_{0.15}(C)_{0.36}$	1.65	0.1-0.13	0.05*	0.1-0.25	6200
$(YSZ)_{0.45}(Au)_{0.10}(MoS_2)_{0.14}(C)_{0.30}$	2.15	0.1*	0.05	-----	-----
$(YSZ)_{0.59}(Au)_{0.10}(MoS_2)_{0.18}(C)_{0.11}$	-----	0.1*	0.05	0.15-0.25	1500

*Indicates friction spikes

**Indicates coating failure before 10000 sliding cycles

Table 2.

Figures

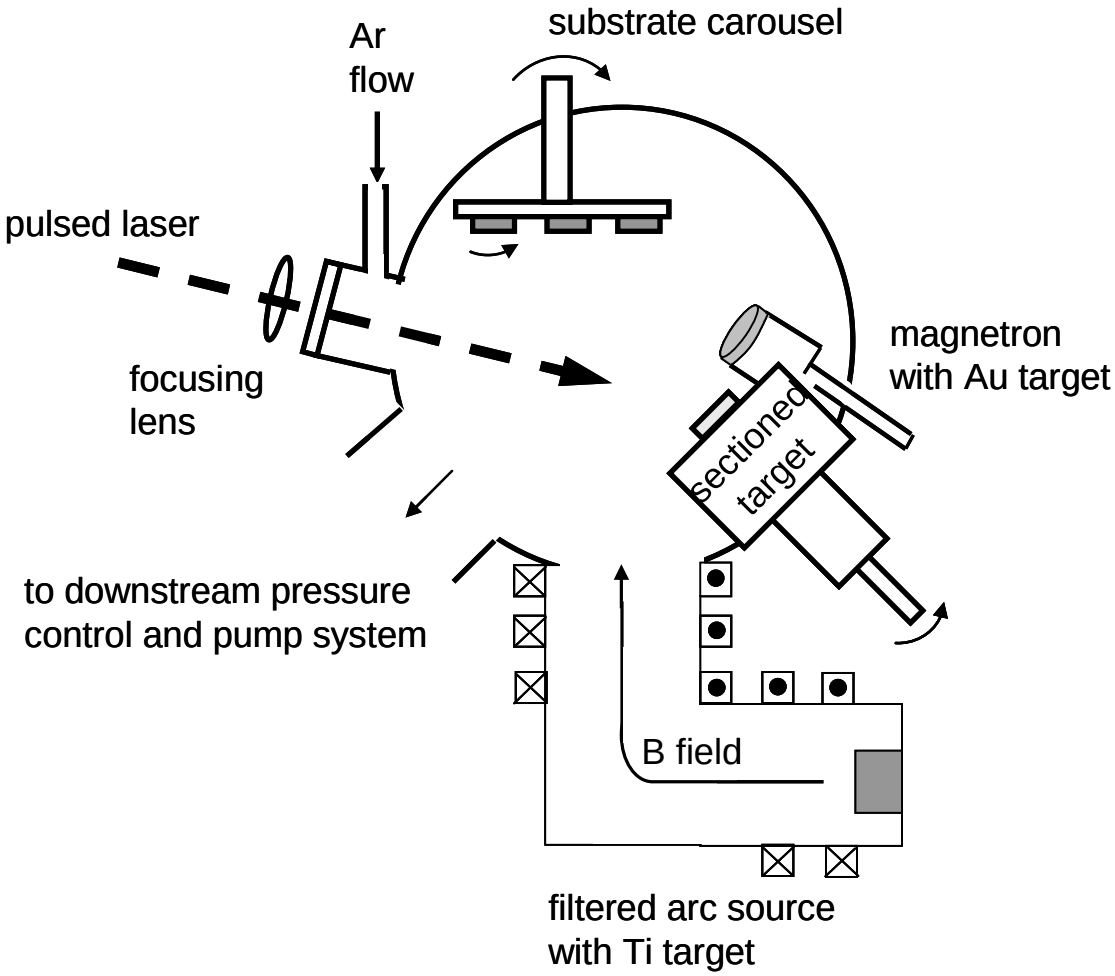

Fig. 1.

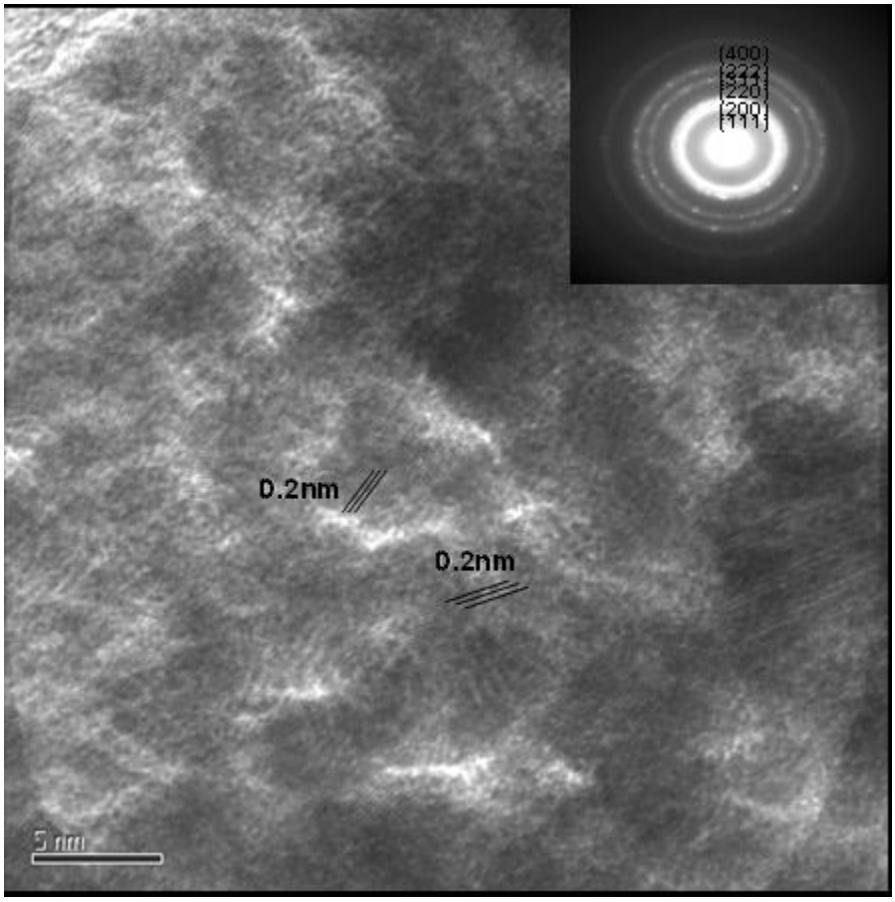

Fig. 2.

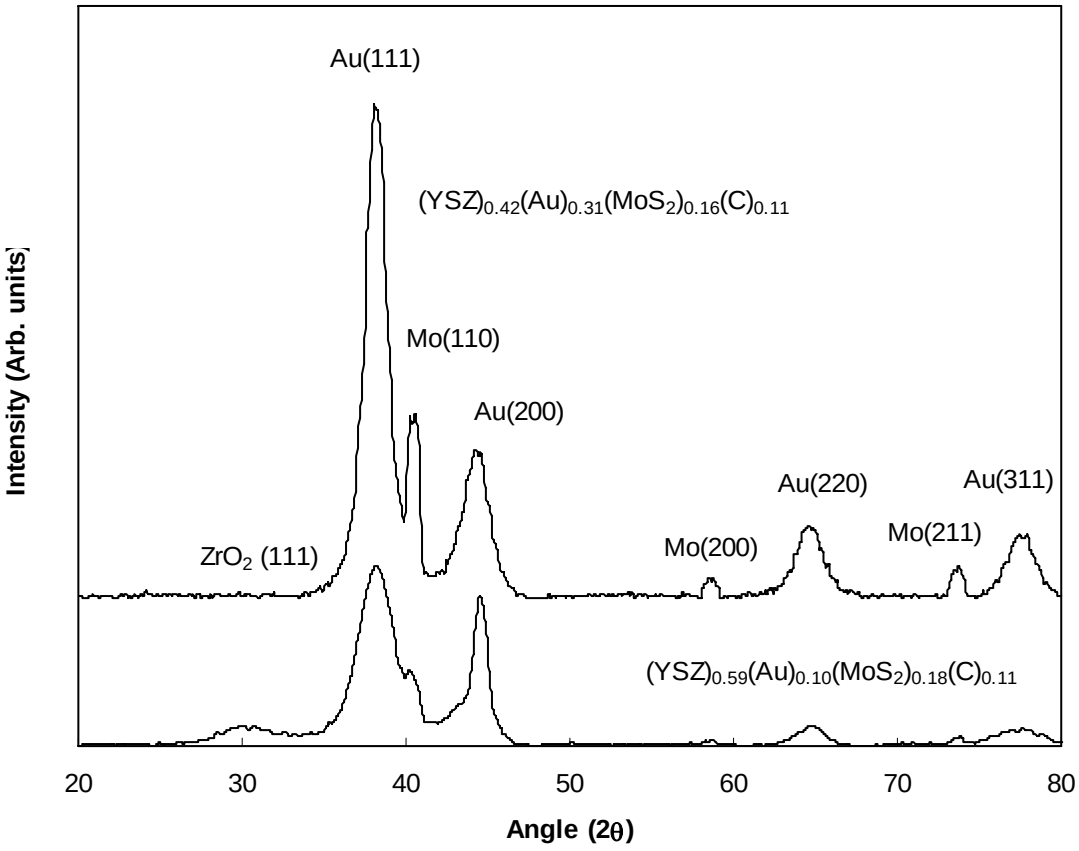
Au(111)
(YSZ)0.42(Au)0.31(MoS2)0.16(C)0.11
Mo(110)
Au(200)
Au(220)
Au(311)
Mo(211)
Mo(200)
ZrO2 (111)
(YSZ)0.59(Au)0.10(MoS2)0.18(C)0.11
Intensity (Arb. units)
20
30
40
50
60
70
80
Angle (2θ)

Fig. 3.

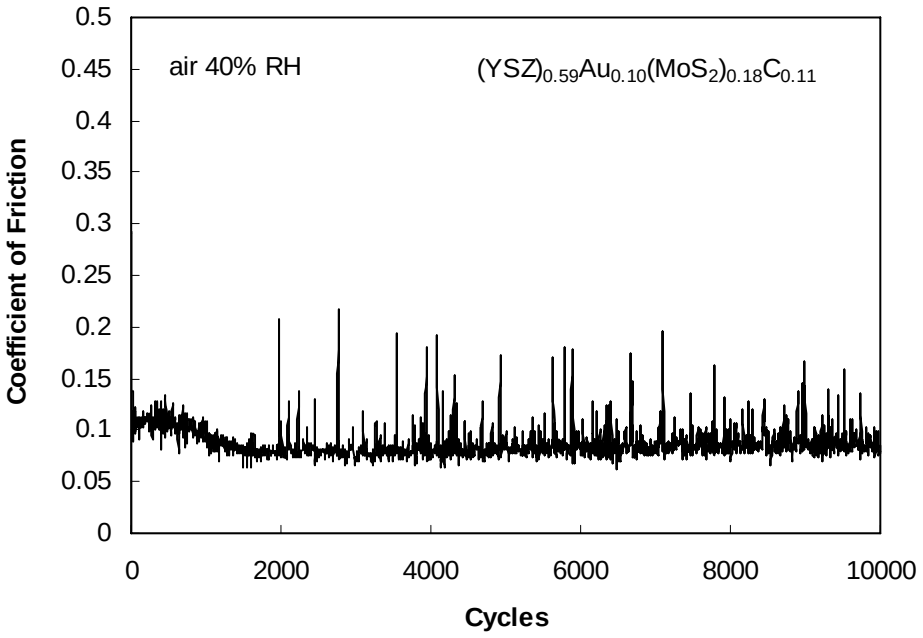
air 40% RH
$(YSZ)_{0.59}Au_{0.10}(MoS_2)_{0.18}C_{0.11}$
Coefficient of Friction
Cycles
0.5
0.45
0.4
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
0
2000
4000
6000
8000
10000

Fig. 4a.

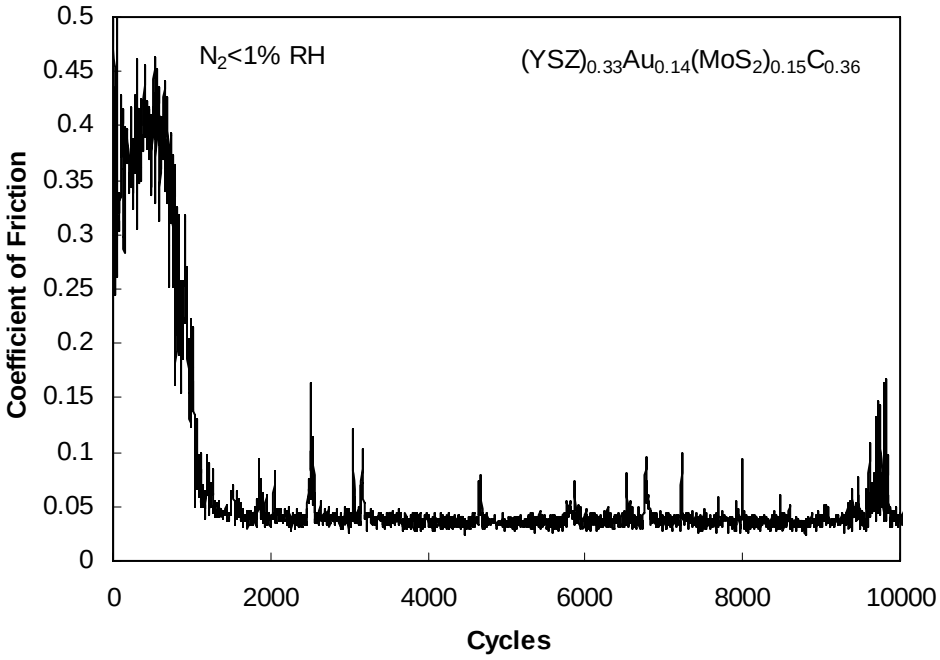
0.5
0.45
0.4
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
Coefficient of Friction
N2<1% RH
(YSZ)0.33Au0.14(MoS2)0.15C0.36
0
2000
4000
6000
8000
10000
Cycles

Fig. 4b.

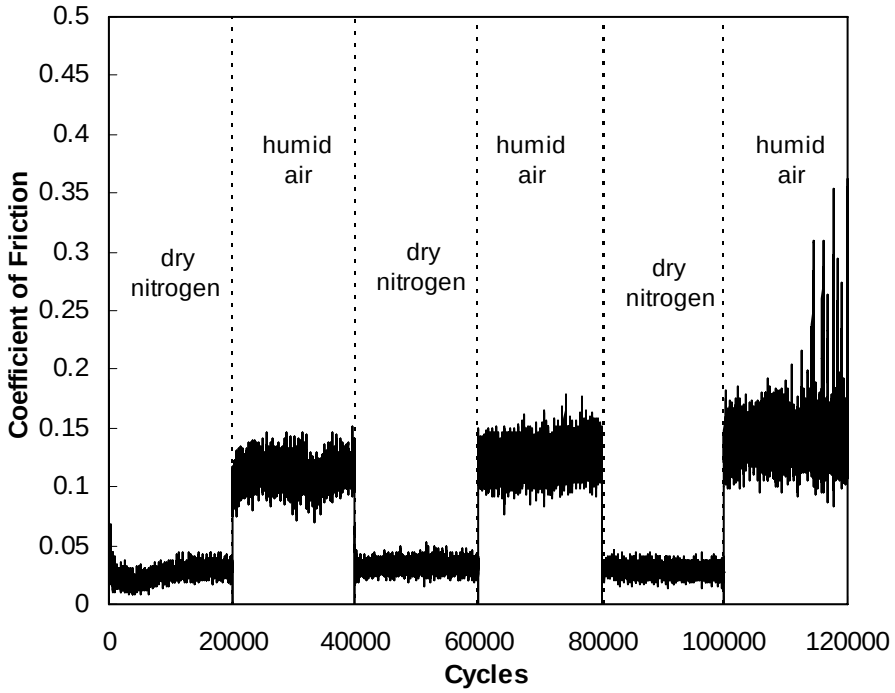
Coefficient of Friction
0.5
0.45
0.4
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
dry nitrogen
humid air
dry nitrogen
humid air
dry nitrogen
humid air
0
20000
40000
60000
80000
100000
120000
Cycles

Fig. 5.

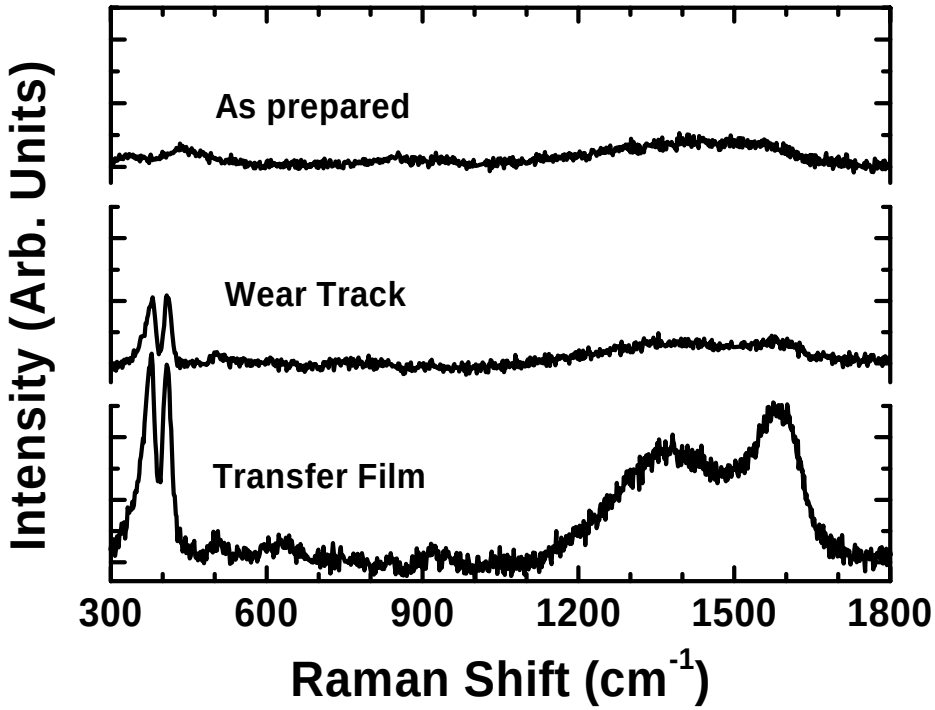
As prepared
Wear Track
Transfer Film
Intensity (Arb. Units)
300
600
900
1200
1500
1800
Raman Shift (cm-1)

Fig. 6.

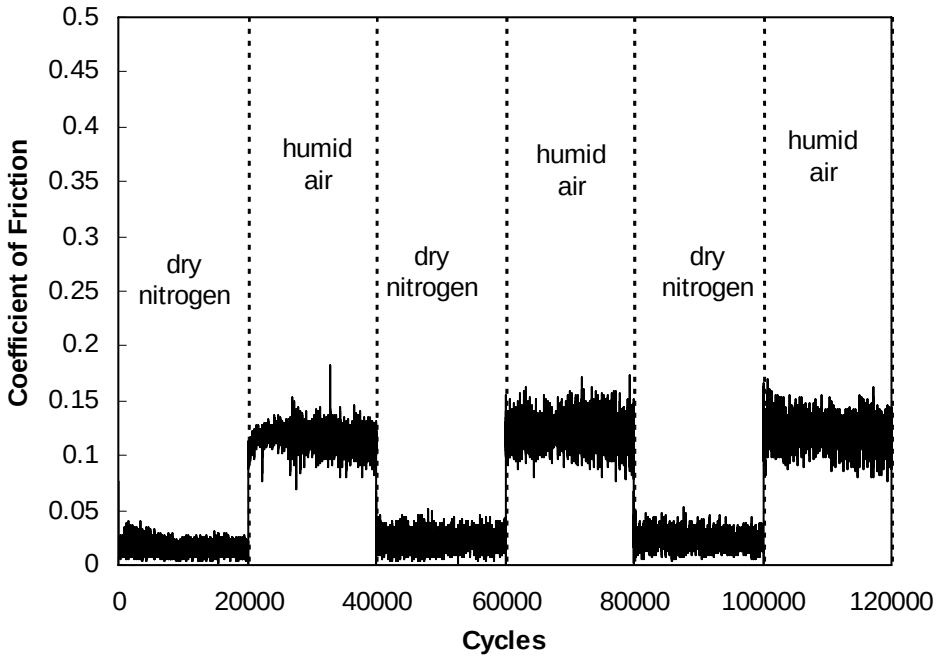
Coefficient of Friction
0.5
0.45
0.4
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
dry nitrogen
humid air
dry nitrogen
humid air
dry nitrogen
humid air
0
20000
40000
60000
80000
100000
120000
Cycles

Fig. 7.