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Properties of low friction anti-seize coatings for fusion applications

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ABSTRACT

Remote maintenance in fusion machines such as JET (Joint European Torus) and ITER (International Thermonuclear Experimental Reactor) relies on sliding interfaces such as bolted joints. Experience in JET, where removal torques much higher than installation values with uncoated bolts is commonplace, led to the installation of experimental bolted assemblies in 2015: the first of its kind in JET. These assemblies included some 660B stainless steel ITER Blanket-specific bolts with a solid sputtered coating of MoS_2 as part of an ITER-funded project known as CHEF (Coating and Humidity Experiments on Fasteners). CHEF also includes two ex-vessel activities. The first is a characterisation of the coating fundamental properties including outgassing, friction, wear and sensitivity to humidity: on flat sample discs. The second is the measurement of the coating performance on bolted assemblies subjected to a sequence of tests: initial tightening cycles, thermal vacuum exposure, environmental exposure (high humidity and high temperature), repeated thermal vacuum exposure, and final tightening cycles. This sequence approximates to a possible operation cycle of ITER bolted joints which receive an accidental humidity exposure during operation.

1. Introduction

MoS_2 which is commonly used in paste form as a general fastener lubricant is also available in a thin (˜ 2 μm) sputtered coating which is vacuum compatible [1]. A typical application is for low friction coating of bearings for use in space where the coatings would always be in a dry state. However, sputtered MoS_2 is believed to be moisture-sensitive and could be accidently exposed to moisture in a tokamak. The CHEF project includes humidity exposure of both disks and bolts for physical analysis and application specific hardware respectively.

In addition, MoST – an MoS_2 variant – has also been tested. MoST contains titanium which provides moisture resistance [2], but its vacuum performance is not so well established. The MoST thickness used here is 1.0–1.4 μm and, following recommendation from the coating supplier, is applied on top of a 2–3 μm sputtered under-coat of CrN. The CrN provides a hard skin to protect the relatively weak 660 B substrate from deformation under high surface loads. This arrangement is intended to align the substrate strength (660B yield ˜700 MPa) with the bearing capacity of the coating (˜ > 1500 MPa), and is sometimes known as the “ice on mud” approach.

The sulphur in these coatings makes outgassing measurements essential as its properties (chemical reactivity, high Z (16)) can lead to persistent radiation losses in plasmas. CHEF includes the use of a highly sensitive outgassing facility described in § 3.

2. JET in-vessel tests

There were 36 experimental bolted assemblies exposed inside JET during 2015-16. They were self-contained with spacers and nuts allowing controlled ex-vessel assembly and dis-assembly, Fig. 1. Eight assemblies were part of CHEF using MoS_2 coated 660B bolts and uncoated 660B nuts. The remaining assemblies had a range of JET-relevant features including spiralock® thread form, and uncoated Nimonic and Inconel bolts. The CHEF assemblies performed well after exposure with their high pre-loads maintained and undoing torques similar to their high installation values. Detail examination of these assemblies and the testing of the JET ones are ongoing and will be the subject of a further publication.

3. CHEF vacuum facility

Fig. 2 shows the high sensitivity outgassing vacuum facility which was engineered in the MRF (Materials Research Facility) at UKAEA Culham for the CHEF project.

Sample transfer is via an Ar-purged vacuum load lock chamber and magnetically coupled transfer arm allowing the main sample chamber

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Fig. 1. JET In-Vessel Bolted Assembly.

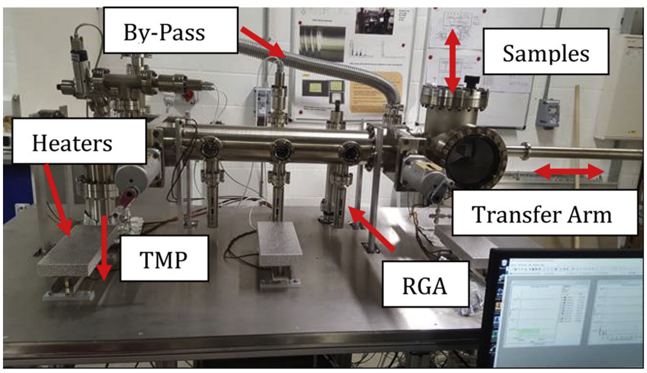

Fig. 2. CHEF Vacuum Facility.

Fig. 3. CHEF Friction Discs During Soak Test (MoST).

(100 mm diameter x 600 mm long) to be permanently under vacuum ($< 10^{-2}$ mBar during transfer (mostly Ar) and $< 3 \times 10^{-10}$ mBar during operation). The chambers and closely coupled equipment can be baked to 200°C by enclosing the whole facility with insulated panels and using 3 x 1 kW heaters mounted on the bench. There is an RGA (Residual Gas Analyser) with remote electronics and a bakable sensor inside the thermal enclosure. A dedicated PC monitors the RGA, four vacuum gauges (two cold cathode and two convection Pirani), seven thermocouples, and has an encrypted network connection so that key operations (e.g. temperature checking and RGA switching) can be done remotely.

A 40 kVA UPS supplies all the electrical equipment – except for the heaters – giving high reliability continuous operation. The tests last typically 4 days (coated discs) or 28 days (bolted assemblies) and the facility has been continuously operating for over 12 months.

4. Friction disc tests

To simplify the humidity exposure for the disc tests, two extreme conditions were used. In the first "dry" condition, the samples were stored dry and when exposed, the RH was monitored to ensure that it never exceeded 65%. In the second "wet" condition, the discs were soaked in distilled water for 1 h before drying naturally for 24 h. The resulting four test types are designated MoSTD, MoSTW, MoS2D, MoS2W where D and W stand for dry and wet respectively. Fig. 3 shows the MoSTW discs at the start of drying – note how the coating repels the water. Both sets of wet samples fully dried in due course without any visible residue.

Each test included a CO (Chamber Only) and ST (Sample Test) phase. The CO phase preceded the ST phase in order to provide the outgassing background. The thermal profiles were approximately 4 h dwell at 20 °C, 12 h ramp up to 150 °C, 24 h dwell at 150 °C and 12 h ramp back to 20 °C.

The RGA recorded the ion currents for all masses in the range 1–200 amu and was normally operated in "bar chart" mode with one measurement per amu taken every 10 s to monitor trends. At key points, including the start and end of the 150 °C dwell, the RGA was switched to "analogue" mode with 32 samples per amu taken to provide more detail. Fig. 4 shows a typical result (MoS2W end of 150 °C dwell) where the CO (blue) and ST (red) phases are overlaid so that the effect of the samples can be highlighted.

The expected presence of H_2, H_2O and CO_2 in similar quantities in both phases is evident by the ion current peaks at 2, 17–18, and 44 respectively. There is also the commonly observed peak at mass 28 which could be due to N_2, CO or other gases as discussed later. The difference due to the presence of the samples is clear evidence of sulphur gases coming from the coatings with peaks at 32–34 (H_2S), 64 and 48 (SO_2) and 76 (CS2).

There are also other peaks in the spectra which are less easy to quantify and so a procedure has been written to de-convolve the mass spectra into parent molecule ions. The procedure takes a library of 26 candidate molecule cracking patterns and performs a least-squares fit with the measured spectra. The result is a predicted molecular ion current for each of the candidates and Fig. 5 illustrates the results for the case above (ST phase). The fit uses all the masses from 1 to 100 (results above 100 were insignificant) and the figure illustrates the 25 most significant along with the 12 most significant molecules. The ratio of RMS error (measured – fit) to the RMS measured value over all masses was generally < 5% showing a good quality fit.

The open columns represent the measured results and the coloured ones the fitted values. Where more than one parent molecule contributes to a single mass e.g. 28, the total coloured column is to the same logarithmic scale as the measured values, but the division within

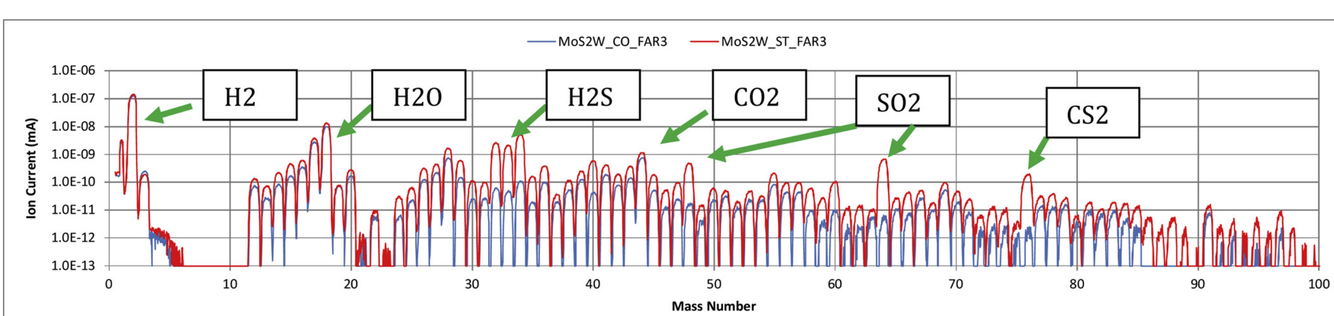

Fig. 4. Analogue Scans (End of 150 °C Dwell). MoS2W.

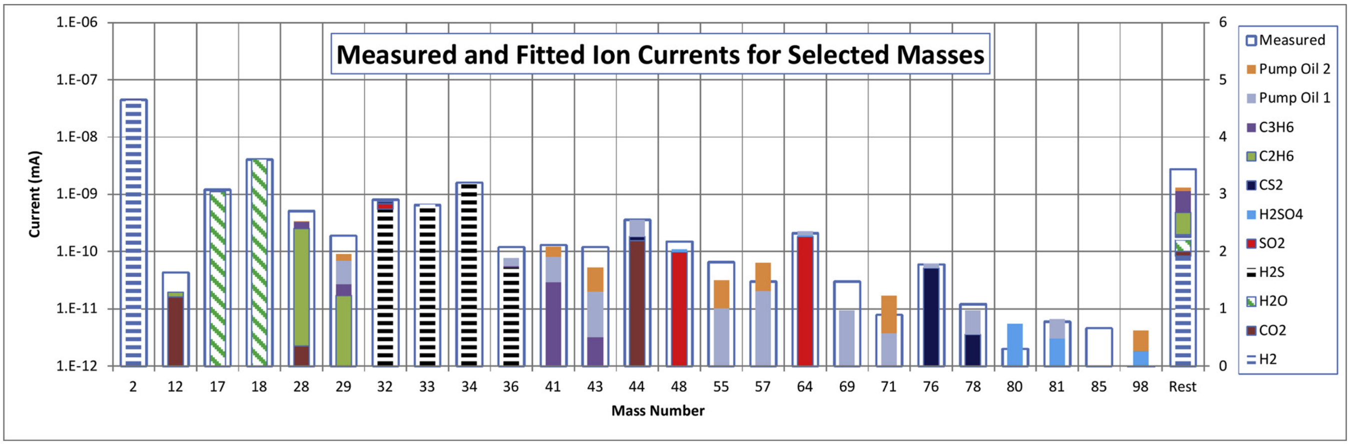

Fig. 5. Measured and Fitted Ion Currents. (MoS2W ST Phase End of 150 °C Dwell).

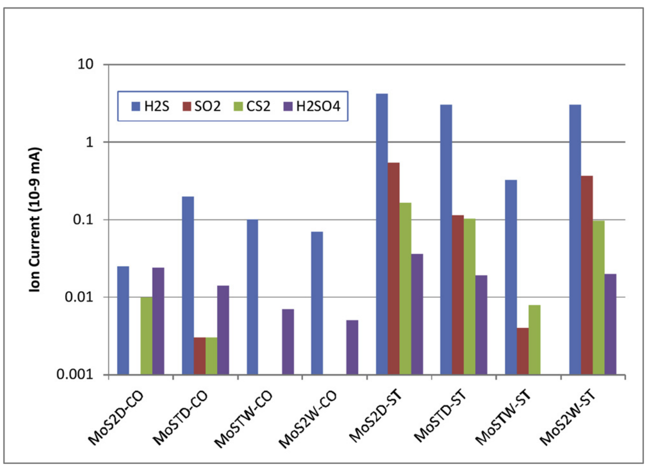

Fig. 6. Sulphur Gas Ion Current Comparison.

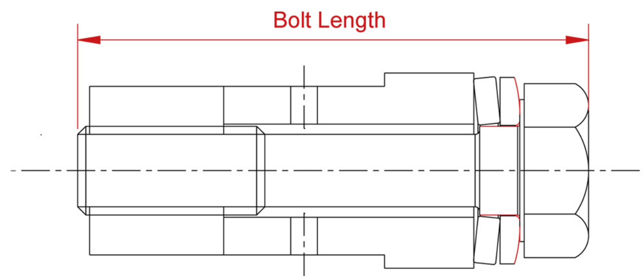

Fig. 7. CHEF Bolted Assembly.

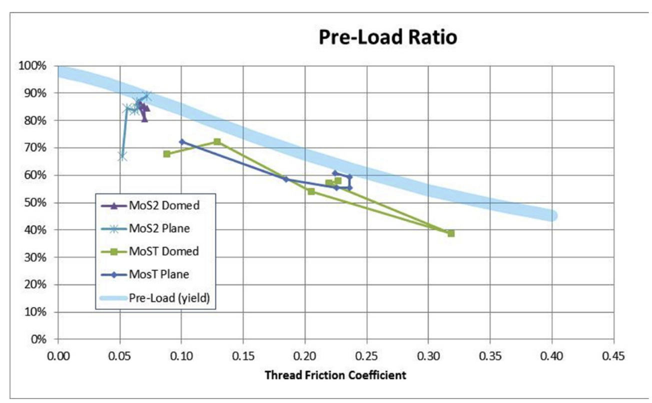

Fig. 8. Ratio of Pre-Load to Yield.

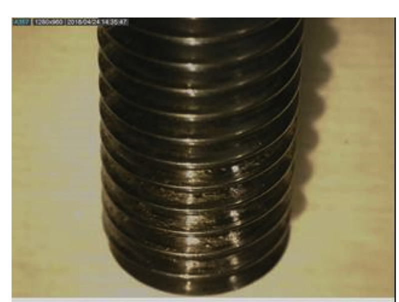

Fig. 9. MoST Coating Damage After 6 Cycles.

Table 1
PoD Results.

Env.	Coat	Exp.	Temp.	μ(init.)	Revs to μ	
			°C		> 0.1	> 0.3
Vac.	MoST	Dry	22	0.033	41	329
Vac.	MoST	Dry	150	0.061	54	395
Air	MoST	Dry	22	0.081	**5000**	**5000**
Vac.	MoST	Wet	22	0.091	78	139
Vac.	MoST	Wet	150	0.046	179	355
Air	MoST	Wet	22	0.051	**5000**	**5000**
Vac.	MoS_2	Dry	22	0.060	48	94
Vac.	MoS_2	Dry	150	0.051	82	194
Air	MoS_2	Dry	22	0.082	27	97
Vac.	MoS_2	Wet	22	0.052	31	83
Vac.	MoS_2	Wet	150	0.072	30	60
Air	MoS_2	Wet	22	0.051	46	111

Bold values represent the maximum values obtained.

the column is linear to represent the true proportions of separate contributions. As H_2 has a single dominant peak at mass 2, a good fit is guaranteed. But there are also good fits for H_2O, H_2S and SO_2 where multiple cracking fragments are present. The results also indicate traces of C_2H_6 (ethane), C_3H_6 (propylene) and vacuum pumping oils. As both the turbo and scroll pumps are oil-free, the likely source of these vapours is residual contamination from the un-baked bypass system used during the sample transfer. They are present in both phases and so are unlikely to be from the samples. The bulk of the measured ion current at mass 28 is accounted for by C_2H_6 and CO_2 rather than N_2 and CO which, along with O_2, have very low fitted values.

Fig. 6 shows the sulphur gas ion currents at the end of the 150 °C dwell for the four CO tests (LHS) and four ST tests (RHS). H_2S dominates the coating outgassing with little differences between coatings and pre-conditioning (wet versus dry). There are also clear indications of SO_2 and CS_2 from all but the dry MoST case. H_2SO_4 was expected for the MoS2 cases: but the levels were low and not significant.

After vacuum testing, the coating friction coefficients, μ, were measured using a PoD (Pin on Disc) tester with the discs rotated beneath uncoated pins at a contact pressure selected to be representative of highly loaded bolts (1500 MPa). There were 12 tests to cover the four disc types with three test conditions each: air at 22 °C, vacuum at 22 °C and 150 °C. The rig monitored μ continuously up to 5000 revolution or a value $\mu > 0.3$ (indicative of complete coating wear-through): whichever occurred first. The material combinations and surface finishes (Ra) replicated those in the bolted assemblies: 660B, ˜0.1 μm, coated (discs and bolts) and 660B, ˜ 0.5 μm, uncoated (pins and nuts).

Table 1 includes the number of revolutions to reach $\mu = 0.1$ which is the desired limiting value for the ITER bolt coating. The results show initial μ ˜ 0.06 as expected. The lifetimes vary enormously with MoST giving the longest particularly when tested in air: but even the shortest lifetime of 27 revolutions is more than sufficient for typical bolting applications.

5. Bolted assembly tests

The CHEF bolted assembly is similar to the JET in-vessel case and comprises a bolt coated on threads and washer face, plane washer coated on the top face, disc spring washer, spacer and un-coated nut, Fig. 7. Standard M10 threads were used. The bolt ends were faced so that the length could be accurately measured and used to deduce bolt preload due to elastic stretching when combined with the calculated bolt stiffness.

The assemblies are currently going through their second thermal vacuum exposure and so results here are for the un-exposed prototypes.

To challenge the coating performance in these early tests, the bolts were tightened to a target pre-load of 80% of tensile yield (and a coating contact pressure ˜1000 MPa). The pre-load was controlled via bolt length measurements at intermediate stages and the coating μ (average of head and thread) was deduced from torque measurements. Fig. 8 shows how the pre-load ratio varies with μ for 6 tightening cycles. Two types of coated washer were tested – domed and plane – and both the MoS_2 cases show excellent performance with μ staying around 0.05-0.07 (similar to the disc PoD value) and pre-loads staying close to the 80%.target.

However the MoST cases reached only about 70% initially, dropping to 40–60% with cycles as μ increased to about 0.25–0.3.

This behavior is due to the slightly higher initial friction in the bolt threads causing torsion in the bolt shaft to reduce the available tension. The blue band indicates the theoretical tension yield when combined with torsion and the MoST case travels down this line as the bolt suffers damage due to yielding and coating damage leads to ever higher friction as attempts were made to reach the target pre-load.

Fig. 9 shows one of the MoST cases where there is clear damage to the coating. It is likely that the MoS_2 case was close to reaching this runaway situation and so for the subsequent endurance tests, the target pre-load was reset to 65%.

6. Summary

Experimental bolted assemblies exposed inside JET during 2015-1016 demonstrated the low friction and anti-seize properties of MoS_2 applied as a solid sputtered coating onto 660B bolts representing an application for ITER blanket modules. Ex-vessel disc tests were used to measure the key properties of outgassing, humidity sensitivity and wear rates of this coating along with a titanium containing variant: MoST. Additional ex-vessel tests on bolted assemblies have so far revealed the durability of these coatings to repeated tightening cycles.

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