Contract for the Operation of the JET Facilities Co-Funded by Euratom

NJOC-PR(16) 15911

P Bunting et al.

Sputtered MoS2 Coatings for JET In-Vessel Fasteners

Preprint of Paper to be submitted for publication in Fusion Engineering and Design

This work has been carried out within the framework of the Contract for the Operation of the JET Facilities and has received funding from the European Unions Horizon 2020 research and innovation programme. The views and opinions expressed herein do not necessarily reflect those of the European Commission.

Sputtered MoS_2 Coatings for JET In-Vessel Fasteners

Authors: P.Bunting[1], V.Thompson[1], V.Riccardo[1]

[1]Culham Centre for Fusion Energy, Culham Science Centre, Abingdon, OX14 3DB, UK

Keywords: Fasteners, seizure, contamination, threads, vacuum, Taguchi, MoS_2

Abstract

JET is a research magnetically-confined thermo-nuclear fusion reactor where a high temperature plasma is created inside a large ultra-high vacuum chamber. The inside of the chamber is protected from the hot plasma with tiles made from beryllium, tungsten, carbon composites and other materials. These tiles are bolted to the vessel wall.

The following study looks at characterising the magnitude of the individual factors affecting the fastener break away torque. The primary aim of this study was to determine whether the application of a low friction coating to the bolt thread would both increase bolt preload and reduce breakaway torque for a controlled tightening torque. This was assessed using a statistical approach, the Taguchi method, to isolate the net effect of individual factors present in a series of tests [1].

The results of this study showed that the Molybdenum-disulphide (MoS_2) coating enhanced bolt performance, increasing bolt preload and reducing break away torque in comparison to uncoated bolts.

Introduction

Events during the 2012 JET shutdown led to a re-examination of the fasteners used to secure diagnostic equipment and protection tiles on the walls of JET.

During the shutdown, several tiles were removed by remote handling (RH) for maintenance and analysis. It was found that on many tiles the breakaway torque on the fasteners (required to loosen them) had increased beyond the initial tightening torque, typically by a factor of two. In addition, one Inconel 718 fastener could not be undone applying a torque of 80Nm, the maximum torque capable from the RH tool. Given the tightening torque was 35Nm, this bolt was assumed to have seized.

As detailed in a previous study [2], it was determined that heat cycling of the fastener assemblies under test conditions caused the preload of the bolts to reduce below that of the previous bolt tightening cycles and the breakaway torque to increase above that seen in the previous cycles.

The current study aims to determine the effect of applying a low friction coating to the threads of the bolt in the fastener assemblies.

Sputtered Molybdenum-disulphide (MoS_2) is a widely used solid lubricant with a layered structure [3]. This is not to be confused with MoS_2 pastes also used in low friction applications. These differ from the solid lubricant since they have no uniform structure and consist of MoS_2 suspended in a solvent [4]. These lubricants are not suitable for use in the JET vacuum vessel as many of the solvents used contain oil which causes problems in obtaining ultra-high vacuum conditions.

The MoS_2 solid lubricant is currently in use in the W-7X fusion reactor [5]. The function of the coating is to prevent seizure between the non-planar coils where NAB (Nickel Aluminium Bronze) mating material is used, the same family of material used for the mating surfaces in standard JET fastener assemblies, making its use applicable to the study.

The structure of solid MoS_2 consists of a layer of molybdenum between two layers of sulphur. The MoS_2 molecules are ordered hexagonally, with strong covalent bonding between molybdenum and sulphur atoms. The low friction characteristic of the coating comes from the poor bonding between the individual MoS_2 layers where only Van de Waals forces exist [3] [5] [6].

The method of deposition is important to lowering the friction coefficient; the avoidance of a columnar structure is required in order to maintain good tribological properties [5]. Two methods that are commonly used to avoid this are magnetron sputtering and a variant on this process, unbalanced closed field magnetron sputtering [5] [6].

Although there is evidence suggesting that MoS_2 coating is a good low friction coating, it has also been found in multiple sources that this coating is sensitive to water intake and is unsuitable for humid environments [3] [5] [6] [7] [8].

The scope of this study is to determine whether MoS_2 would be a suitable bolt coating which allows the JET in vessel bolts to be reliably removed after preloading. All coated bolts have both the threads and surfaces under the head coated.

Due to the results seen in a previous study [2], the heat cycling of the bolts was not carried out. The method of heat cycling which would have been used was shown to not be an accurate representation of the fasteners' typical life cycle since. The purpose of the previous study was to replicate in-vessel conditions and cause some bolt assemblies to seize, thus replicating the mechanism seen in the original component failure.

The bolt material has been varied to determine whether Nimonic 80A may be more suitable than Inconel 718.

The examination of the effects of the experimental parameters was achieved by using a statistical approach based on a Taguchi method of examining interactions between test factors [1]. This approach reduces the number of tests and reduces the cost and time required for the study.

For this particular study we will examine the effect of 4 factors each of which have two states. In Figure 1, the "+" and "-" states define which of the two parameters states are used in each test.

		Parameters/Effects						
		1	2	3	4	5	6	7
Tests	1	-	-	+	-	+	+	-
	2	+	-	-	-	-	+	+
	3	-	+	-	-	+	-	+
	4	+	+	+	-	-	-	-
	5	-	-	+	+	-	-	+
	6	+	-	-	+	+	-	-
	7	-	+	-	+	-	+	-
	8	+	+	+	+	+	+	+

Figure 1 - L8 Orthogonal Array – Modified Layout

A full experimental study would thus require 16 tests (2^4). The Taguchi method guides the selection of a reduced number of factor combinations to be used in a smaller number of tests – 8 in this case – to maximise the output from the tests. The Taguchi factor selection is defined by orthogonal arrays and here the appropriate array, L8, is shown in Figure 1.

Four of the columns in Figure 1 represent the test factors and the rest are left un-assigned. The rows represent separate tests with particular configurations of factors and the matrix itself is made up of elements which are either "+" or "-" corresponding to the binary state of the relevant factor.

Here the columns were assigned as follows:

- 1 (A) – Bolt material (Nimonic 80A / Inconel718)
- 2 (B) – Bolt Thread coating (MoS_2 / None)
- 4 (D) – Insert Material (Inconel 625 / NAB)
- 5 (E) – Insert thread form (ISO Standard / Spiralock®)

Spiralock® is a thread form developed to provide resistance to loosening of fastener assemblies.

Materials and Methods

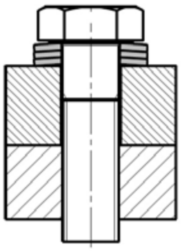

Figure 2 – Section Drawing of Bolt Assembly

The experimental testing involved the following test procedure:

1. Bolt relaxed – Length measured
2. Bolt tightened to 35Nm – Length measured
3. Bolt loosened – Length and breakaway torque measured
4. Bolt tightened to 35Nm – Length measured
5. Bolt loosened – Length and breakaway torque measured
6. Bolt tightened to 35Nm – Length measured
7. Bolt loosened – Length and breakaway torque measured

The tightening and loosening procedure was repeated 3 times for each bolt assembly to determine any cyclic deterioration of bolt performance.

At each stage of the procedure the tightening/loosening torque of the bolted assembly was measured using a digital torque wrench which records the maximum values. In addition to this, the lengths of the bolts were measured using a micrometer at each stage to enable the inference of a bolt preload prior to loosening. To obtain the most accurate length measurement, the bolts were modified before testing began to have machined flat surfaces on the top and bottom faces. When used in conjunction with stiffness values obtained through both hand calculation and finite element analysis, the preload of the bolt may be inferred from the measured length. The loads were kept well below yield so elastic behaviour could be assumed.

Results

The baseline parameters represent the standard fastener configuration used in JET, with the addition of the MoS_2 coating on the bolt thread.

The test results in Figure 3 and Figure 4 are taken from one of the three replicates from the labelled baseline tests. The baseline case parameter states were:

- Bolt material – Inconel 718
- Insert Material - Nickel-Aluminium-Bronze (NAB)
- Insert thread form - Spiralock®

However, to more effectively illustrate the difference between the coated and uncoated bolt assemblies, both results have been displayed.

Figure 3 represents the raw breakaway torque data, combined with the calculated k-factor data for the baseline case. The k factor may be obtained for values of applied torque, bolt diameter and bolt preload.

$$T = k \times F \times D$$

T = Applied torque k = Bolt torque factor

F = Bolt preload D = Bolt diameter

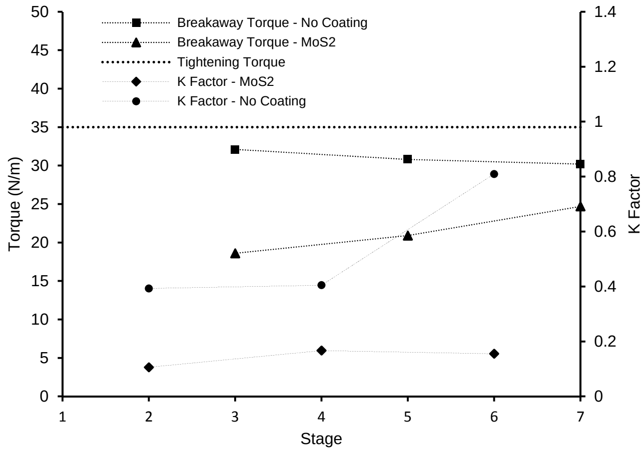

Figure 3 - Sample Torque and K factor data for baseline cases

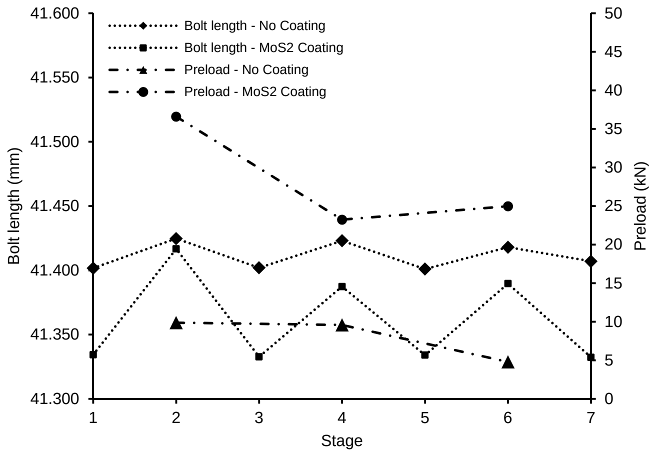

Figure 4 - Sample Bolt Length and Preload data for baseline cases

Figure 4 represents the bolt length during the testing stages and the inferred bolt preload. For each test, two replicate tests are carried out to mitigate the effect of an anomalous result. The results shown in Figure 4 are typical results from individual replicates from their respective tests.

The results of all three repetitions for a particular test are averaged to give the response used in the effects plot.

The use of the Taguchi based method allows the production of an effects plot for each set of test responses. For each parameter an effect on test response may be present. The effects plot displays this as a net effect for each parameter.

In this case, effects plots were produced from the measured data of both breakaway torque and bolt preload responses.

The effects plots are based on the average values of bolt preload and breakaway torque at each stage of the tests taken from the 3 repetitions of each test. These results are the test responses from which the effects plots for bolt preload and breakaway torque were produced and are shown in Figure 5 and Figure 6.

To obtain the net effect of a particular parameter, the average value of all the responses with a "+" must be subtracted from all the average responses with "-". This then gives the range of the net effect between the two states of the chosen parameter.

Each net effect is plotted centred over the average value of all the responses for that data set. The plots are labelled to illustrate which of the binary states for each parameter produced the higher or lower value of net effect.

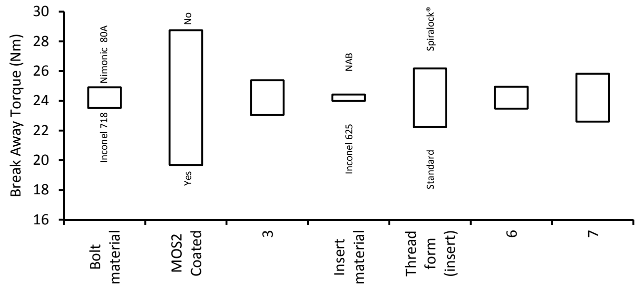

Figure 5 – Breakaway Torque Effects Plot

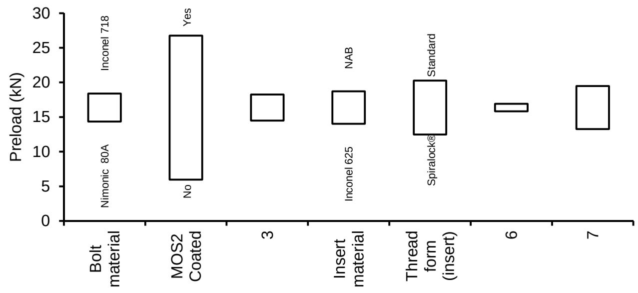

Figure 6 – Bolt Preload Effects Plot

Discussion

Table 1 - Factor Interaction Table

Column No.	1	2	3	4	5	6	7
Effect	[A]	[B]		[D]	[E]		
Measured 2 FIs	[DE]		[AB]	[AE]	[AD]	[BD]	[BE]
Measured 3 FIs			[BDE]			[ABE]	[ABD]

Due to the interactions in each column, summarised in Table 1, it can be determined that the large net effect seen in column 2 for the MoS_2 coating is a pure effect, i.e. not the result of confounding 2 factor interactions. This suggests that the coating is the cause of the largest increase in bolt preload out of all the effects studied.

The bolt preload effects plot in Figure 6 shows that the MoS2 coated bolts have increased preload, up to 5 times higher than their uncoated counterparts.

Figure 5 shows that the greatest effect on breakaway was caused by the change in bolt coating. This indicates that by having the MoS_2 coating applied to the thread and under head surfaces the breakaway torque is reduced. This is also true of the results seen in the baseline cases in Figure 3.

Figure 3 also shows that the breakaway torque for the coated bolts remains near constant with each tightening cycle. This is also true for uncoated bolts. Whilst this may suggest that with further cycles this values for both may continue to remain constant, this is unlikely to occur.

The second largest effect on the breakaway torque is the insert thread form. The standard insert thread form was shown to reduce the breakaway torque. When compared to the results seen in other studies, this is in agreement with what has already been observed about the effect of insert thread form on breakaway torque [2].

As more loading cycles were performed, the preload of the coated bolts begins to drop off but still not down to the level of the uncoated bolts.

The coating of bolts in MoS_2 has resulted in the preload being increased above that of the uncoated bolts. The bolt breakaway torque has also been reduced below that of the uncoated bolts. These effects have been demonstrated to be reliable after multiple uses.

The use of the MoS_2 coating on the bolts has significantly increased their overall performance, by both increasing the preload for the tightening torque of 35Nm and reducing the breakaway torque even after such high loading.

These characteristics meet the aims of the study and identify the MoS_2 coated bolts as a possible candidate for future use in the JET vessel providing that other requirements are met in further testing which is out of the scope of this study, for example the compatibility with humidity.

The results of this study have also addressed the sub aim of the study, to explore the possibility of Nimonic 80A being used as an alternative bolt material.

The trend previously seen was that the Nimonic 80A bolt had a lower breakaway torque than the Inconel 718 making the justification of which was more suitable difficult since the Inconel bolts had a higher preload [2].

However, now with a low friction coating, the difference in breakaway torque between the two is reversed with the Inconel 718 bolts having the lower breakaway torque.

From Figure 6, the preload seen in coated Inconel 718 bolts has been shown to be higher than that of the coated Nimonic 80A bolts. The net effect was approximately 4kN.

The advantage of using effects plots for the statistical analysis is that the plots should highlight any of the second order effects between the factors. Figure 5 and Figure 6 both show small, but not negligible 2 factor interactions present in column 3. Column 3, as described in Table 1, is the interaction between the bolt material and the coating of the bolt thread.

The presence of the interaction shows that there are combinations of material and coating which perform better than others. One of the limitations of the effects plot is that it is not able to specify which combinations these are. But because the effects plot has highlighted this, the specific data can then be reviewed.

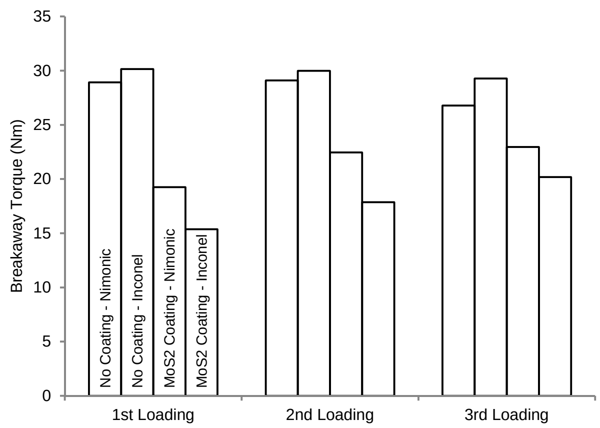

Figure 7 - Average Breakaway Torque for Coated Bolts

Figure 7 and Figure 8 represent the average breakaway torque and bolt preload of all the tests at the 1st, 2nd and 3rd loading stages of the tests.

Figure 7 shows that at each stage of loading the coated Inconel bolts consistently have a lower breakaway torque. This is in contrast to what is seen in the uncoated bolts, where the Nimonic bolts have a the lower breakaway torques. This indicates that the coating has a greater effect on the Inconel bolts than the Nimonic bolts in reducing the breakaway torque. This corresponds to the effect seen in column 3 of Figure 5.

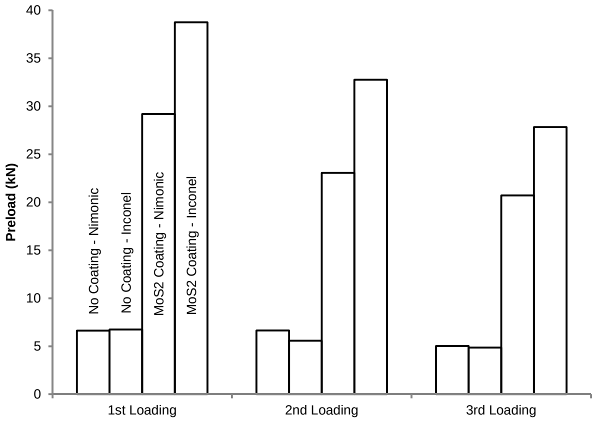

Figure 8 - Average Preload for Coated Bolts

Figure 8 shows that at each stage of loading the coated Inconel bolts consistently have a higher preload. This differs from the uncoated bolts which have very similar preloads. The effect seen in column 3 of Figure 6 can be attributed to this.

Based on the results from both effects plots and the additional data from the raw data, the preferred material for the bolts is Inconel 718 with both higher preload and lower breakaway torque when compared to the Nimonic 80A bolts.

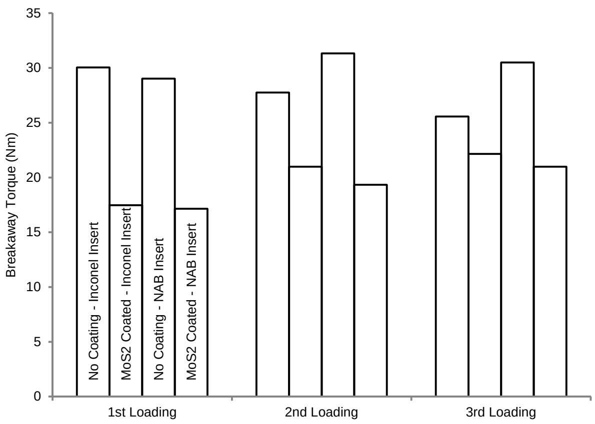

Figure 9 - Average Breakaway Torque for Coated and Uncoated Bolts with Varying Insert Material

It should be noted that further 2 factor interaction are present in both effects plots, Figure 5 and Figure 6. Column 6 shows the 2 factor interaction between bolt coating and insert material, and the 3 factor interaction between bolt material, bolt coating and thread form. It is unlikely that the 3 factor interaction is the reason for this measured effect, which suggests that there is a small interaction between bolt coating and insert material.

This interaction may be explained with Figure 9. There is a large difference between coated and uncoated bolts, and a small difference between the insert materials used. The interaction shown may be where the uncoated bolt behaviour varies with respect to the insert material. The uncoated bolt with a NAB insert is higher than its equivalent with an Inconel insert. Whereas, with the coated bolts this is reversed and the Inconel insert has a higher breakaway torque. This behaviour is not reflected in the preload results, which may suggest why it is more prevalent in the breakaway torque results.

A further 2 factor interaction present in the effects plots can be seen in Figure 5 and Figure 6 in column 7. Using Table 1, column 7 corresponds to a 2 factor interaction between bolt coating and insert thread form. A 3 factor interaction may also be present in this effects plot, however like column 6 it is unlikely to be the source of the measured response.

Figure 10 shows the average result for the coated and uncoated bolts with the two different insert thread forms. This shows that the combination yielding the lowest breakaway torque is the coated bolt with a standard thread, which is in agreement with the results seen in Figure 5.

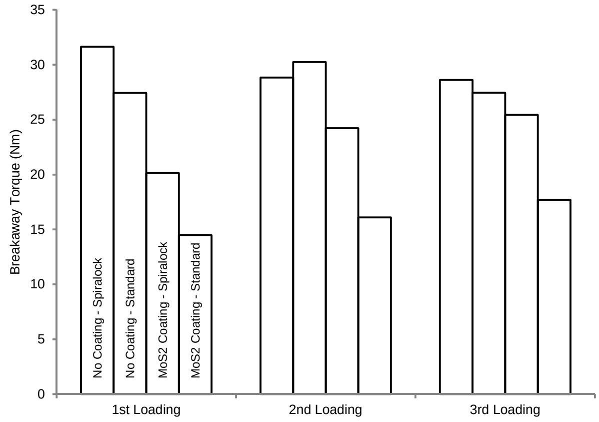

Figure 10 - Average Breakaway Torque for Coated and Uncoated Bolts with Varying Insert Thread Form

It has previously been established through Figure 5 that coated bolts have lower breakaway torques. However Figure 10 shows that the relationship between the insert thread forms for coated bolts is different to that seen with the uncoated bolts. For uncoated bolts the Spiralock thread form gives lower breakaway torque, however once the bolts are coated this is not true and the standard thread form has the lower breakaway torque. This may be the effect being measured in column 7.

Conclusions

In conclusion to this study, it has been determined that the application of a MoS_2 coating applied to the thread and under head region of Inconel 718 and Nimonic 80A bolts, the preload for a set torque of 35Nm is increased and the breakaway torque is reduced when compared to uncoated counterparts.

Of the two bolt materials, Inconel 718 was found to be the preferred materials for use in the JET vacuum vessel. This was determined due to its higher preload and lower breakaway torque when compared to Nimonic 80A.

Further to this investigation it was recognised that to assess the compatibility of the MoS_2 coating with the UHV conditions of the JET vacuum vessel additional testing will need to occur. These tests will need to determine how the coating is affected by moisture before they are used in their operating environment. The work should determine whether the exposure to different levels of moisture deteriorates the tribological properties or causes the out gassing at low pressures of gases incompatible with JET operating conditions.

Acknowledgments

This work has been carried out within the framework of the Contract for the Operation of the JET Facilities and has received funding from the European Union's Horizon 2020 research and innovation programme. The views and opinions expressed herein do not necessarily reflect those of the European Commission.

Bibliography

[1] D. M. Grove and T. P. Davis, Engineering Quality & Experimental Design, Singapore: Longman Singapore Publishers Pte Ltd, 1992.

[2] P. Bunting, V. Thompson and V. Riccardo, “Fastener Investigation in JET,” *Fusion Engineering and Design,* vol. 112, no. 1, pp. 42-46, 2016.

[3] N. M. Renevier, J. Hampshire, V. C. Fox, J. Witts, T. Allen and D. G. Teer, “Advantages of using self-lubricating, hard, wear-resistant MoS2-based coatings,” *Surface and Coatings Technology,* Vols. 142-144, no. no issue, pp. 67-77, 2001.

[4] Dow Corning, “Molykote Industrial lubricants,” Dow Corning corporation, No location, 2007.

[5] F. Koch, R. Nocentini, B. Hinemann, S. Lindig, P. Junghanns and H. Bolt, “MoS2 coatings for the narrow support elements of the W-7X nonplanar coils,” *Fusion Engineering and Design,* vol. 82, no. 5-14, pp. 1614-1620, 2007.

[6] D. G. Teer, J. Hampshire, F. Fox and V. Bellido-Gonzalez, “The tribological properties of MoS2/metal composite coatings deposited by closed field magnetron sputtering,” *Surface and Coatings Technology,* Vols. 94-95, no. no issue, pp. 572-577, 1997.

[7] D. G. Teer, “New solid lubricant coatings,” *Wear,* vol. 251, no. 1-12, pp. 1068-1074, 2001.

[8] D.-Y. Yu, J.-A. Wang and J.-L. O. Yang, “Variations of properties of the MoS2-LaF3 cosputtered and MoS2-sputtered films after storage in moist air,” *Thin Solid Films,* vol. 293, no. 1-2, pp. 1-5, 1997.