MODERN SELF-LUBRICATING COMPOSITES FOR SPACE APPLICATIONS: PGM-HT & SINTIMID 15M

Michael Buttery[(1)], Ben Wardzinski[(1)], Kelly Houton[(1)]

[(1)] *ESTL, Whittle House, Birchwood Park, Warrington, Cheshire, WA3 6FW, UK,*
Email: michael.buttery@esrtechnology.com

ABSTRACT

PGM-HT (PTFE, Glass fiber & Molybdenum Disulphide) is a self-lubricating composite which has been accepted in recent times as a replacement to RT/Duroid 5813; a similar composite no longer in production. The material is used primarily within the space industry as a ball-bearing cage material. It is supplied by the manufacturers pre-treated to a given temperature under either vacuum or air. This paper provides a comparison between these two treatment methods, with regard to the material's ability to lubricate.

Sintimid 15M (known as Tecasint 1391 from April 2009, but referred to as Sintimid here) is an un-reinforced thermoplastic polyimide featuring 15% molybdenum disulphide filler content. It is of particular interest as a European sourced self-lubricating composite alternative to Vespel SP-3 (manufactured in the US and of a similar composition). A series of pin-on-disc tests on this material has been carried out at ESTL to investigate its potential as a lubricant under a variety of conditions.

The paper is split into two sections, each separately covering one of these separate self-lubricating composites

1. BACKGROUND

Self-lubricating composites provide engineers and project managers with simple, inexpensive solutions to certain tribological applications. Through the mechanisms of sacrificial wear and thin film transfer, the contact zone between a self-lubricating composite and a hard counterface can be lubricated without the need for traditional solid or liquid lubrication. Within the space industry these materials are used as bushes, sliding interfaces, guides, ball-bearing cages, slip-rings, motor brushes, and gears.

1.1. PGM-HT

Following the cease in manufacture of the self-lubricating composite RT/Duroid 5813 (Duroid) in the mid-1990's, a replacement was sought. ESTL identified PGM-HT (a composite of PTFE, glass fibre and MoS_2) as "potentially a direct replacement for Duroid" [1]. Subsequently the material became used primarily as a ball bearing cage material – either as a single source of lubricant within a bearing or in conjunction with thin MoS_2 coatings applied to the bearing races and balls.

Some years ago ESTL discovered that PGM-HT shrinks upon returning to room temperature following heating under vacuum [2, 3]. However at elevated temperatures the combination of PGM-HT cage and MoS_2 coatings performed effectively, and could be considered for high-temperature applications.

This shrinking phenomenon was studied and characterized at ESTL, which established that shrinkage could be reduced to acceptable levels by preconditioning the PGM-HT material at 240deg.C **under vacuum** over a 24-hour period [2, 4]. This information was passed onto JPM, the manufacturer of PGM-HT, who procured a vacuum oven to allow the in-vacuum preconditioning of raw PGM-HT material upon request.

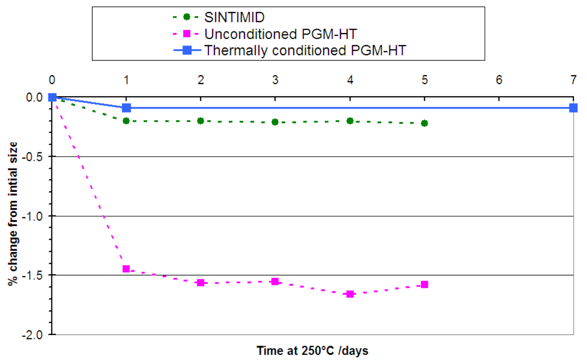


Figure 1. Dimensional change of dummy PGM-HT cages after heating to 250 °C – showing beneficial effects of pre-conditioning on PGM-HT. Sintimid 15M also shown on plot [2]

In addition, these results led to the need to demonstrate that this in-vacuum conditioning of the material does not degrade the material's desirable tribological properties. In this light, ESTL carried out an investigation into the performance of the in-vacuum treated material, carrying out both a repeatability study through the manufacture of pins from multiple blocks of material, and a comparison with the 'un-treated'

'14th European Space Mechanisms & Tribology Symposium – ESMATS 2011'
Constance, Germany, 28–30 September 2011

PGM-HT over a range of test conditions. Tests were carried out using a pin-on-disc tribometer.

This study [5] demonstrated relatively poor repeatability for the in-vacuum treated material, with little consistency either between separately conditioned blocks of PGM-HT, nor within the blocks themselves. Run-in friction values ranged from 0.05 to 0.45, and displayed erratic profiles.

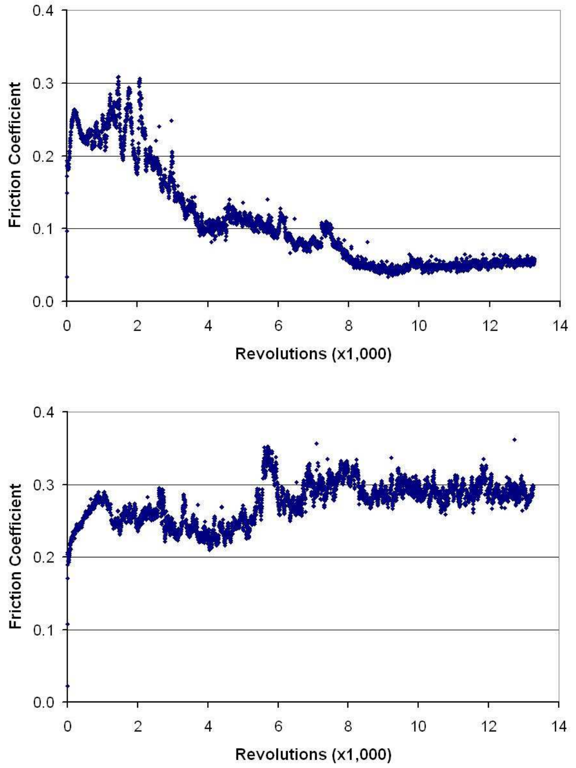


Figure 2. Two friction plots of in-vacuum conditioned PGM-HT from the same block (sliding against 52100 steel, under vacuum, 5N load, 0.1ms^{-1} sliding speed)

Measured wear rates varied, ranging from 1 to 12 x 10^{-15} m^3/Nm. There also appeared to be a correlation between friction coefficient and wear rate of the in-vacuum material [5], and a comparison with data from un-conditioned material taken from [1] showed similarities.

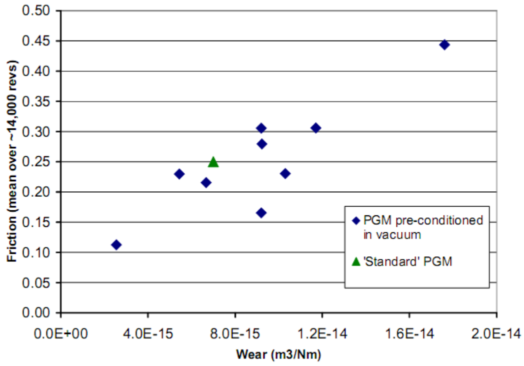


Figure 3. Integrated friction against wear rate. In-vacuum data from [5], 'Standard' data from [1]. Test performed under vacuum, load 5N, speed 0.1ms^{-1}

Recently it has come to our attention that JPM will, unless specifically requested to carry out in-vacuum pre-conditioning, subject PGM-HT material to heating **in air** at 140deg.C for 8 hours. However JPM are uncertain as to when this practice started, and as a result we are unable to state whether the PGM-HT tested in [1] had been subjected to this in-air heating.

Thus for the avoidance of doubt, testing was carried out to compare the tribological properties and repeatability of in-air conditioned PGM-HT against in-vacuum conditioned PGM-HT.

1.2. Sintimid 15M

Sintimid 15M (renamed as Tecasint 1391 in 2009) is a self lubricating material comprising polyimide (un-reinforced thermoplastic) with 15% MoS_2 filler content. It is manufactured within Europe by Ensinger, and is the European equivalent to DuPont's Vespel SP-3.

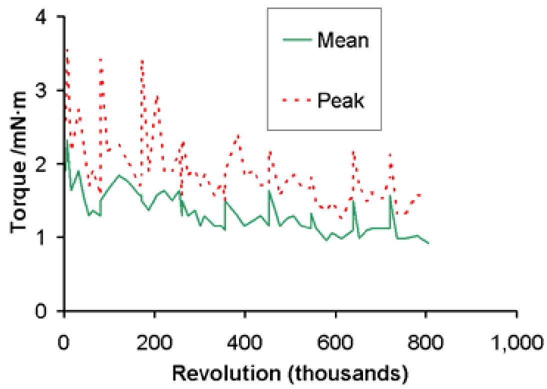


Figure 4. Lifetest results of bearing lubricated with MoS_2 lubrication (balls & races) and titanium-Sintimid cages, running at 275deg.C under vacuum [2]

Tests at ESTL have demonstrated that TECASINT 1391 has strong potential as a bearing cage material, performing well over a wide range of temperatures under vacuum, and in particular for bearings lubricated with sputtered MoS_2 coatings [2]. Good behavior was also observed for tests without lubricant coating on the balls or races (lubrication provided by the cage only) at high temperatures.

The material has also been demonstrated to be thermally stable, displaying minimal shrinkage when subjected to thermal cycling [2], as shown in Fig. 1.

Given Sintimid 15M's potential, it seems likely that it will be used increasingly as a material for European space applications as an alternative to Vespel SP-3 and PGM-HT, both of which are produced in the US. Given this predicted increase in use, and the repeatability issues with in-vacuum treated PGM-HT raised previously [5], it was decided to make an appropriate check on the repeatability of the friction and wear properties of Sintimid 15M.

2. TRIBOLOGICAL TESTING OF PGM-HT

2.1. Test details

Testing was separated into two stages: a first stage in which the batch-to-batch repeatability of the in-air conditioned material was assessed, and a second stage in which the tribological behaviour of the material was studied with regard to environment, sliding speed, and contact stress.

First stage – repeatability

Hemispherically ended pins of PGM-HT were manufactured from three independently pre-conditioned blocks of in-air treated material. Three pins were manufactured from each block, giving a total number of nine tests. Pins were run against 52100 steel discs of surface roughness ~0.1µm R_a using a pin-on-disc tribometer. Motion was carried out uni-directionally over a sliding distance of 1,000m (~13,260 disc revolutions at a track radius of 12mm) under high vacuum (5 x 10^{-6} mbar). A sliding speed of $0.1ms^{-1}$ and a 5N load (10MPa mean contact stress with 18mm pin radius) was employed for all repeatability tests. Note, these conditions are identical to Test 1 of the characterisation tests, in Tab. 1 below.

Second stage – characterisation

Identical pins were manufactured from a single block of in-air conditioned PGM-HT for the characterisation tests. These were tested on a pin-on-disc tribometer in a similar way to the repeatability tests, following Tab. 1.

Table 1. Test conditions for characterisation tests of in-air conditioned PGM-HT

Test ID	Test environment	Sliding speed (m/sec)	Contact stress
1	High vacuum (5 x 10^{-6} mbar)	0.1	S1
2			S2
3		0.01	S1
4	Laboratory air (class 10,000)	0.1	S1
5			S2
6		0.01	S1
7	Dry nitrogen	0.1	S1

Notes: S1 equates to a mean contact stress of 10MPa (pin load 5N, PGM-HT pin radius 18mm)
S2 equates to a mean contact stress of 16MPa (pin load 5N, PGM-HT pin radius 18mm)

These conditions were chosen to be identical to those in [5].

2.2. Pin-on-disc methodology

Test specimens were ultrasonically solvent cleaned prior to testing. All testing was carried out using ESTL's pin-on-disc tribometer. The experimental setup is shown in Fig. 5, and consists of a pin mounted on a balanced arm, and loaded against the disc by a deadweight. The disc is rotated by a motor positioned outside the vacuum chamber. The frictional force is measured via the deflection of the arm, and recorded using a PC-based data acquisition system. Calibration was checked using a pulley system to apply known loads to the tribometer arm.

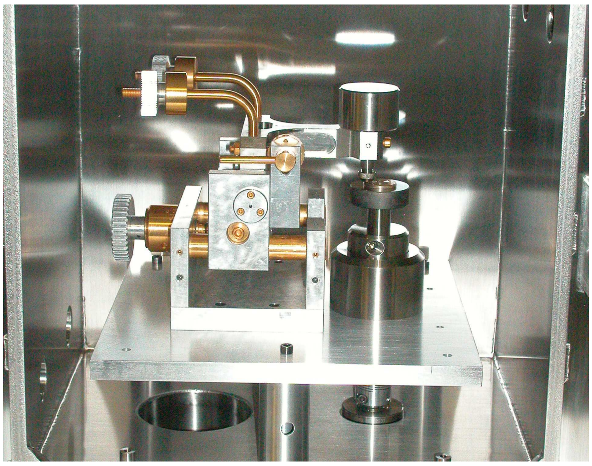


Figure 5. Vacuum pin-on-disc tribometer

2.3. Test results - repeatability

Repeatability results for in-air conditioned PGM-HT were fairly encouraging, with a noticeably greater level of consistency when compared with the in-vacuum treated material, considering both the steady-state friction coefficient as well as the specific wear rate. Friction coefficients typically increased to between 0.3 and 0.35 over the first few thousand revolutions, before settling down to between 0.05 and 0.1 at around 10,000 revolutions.

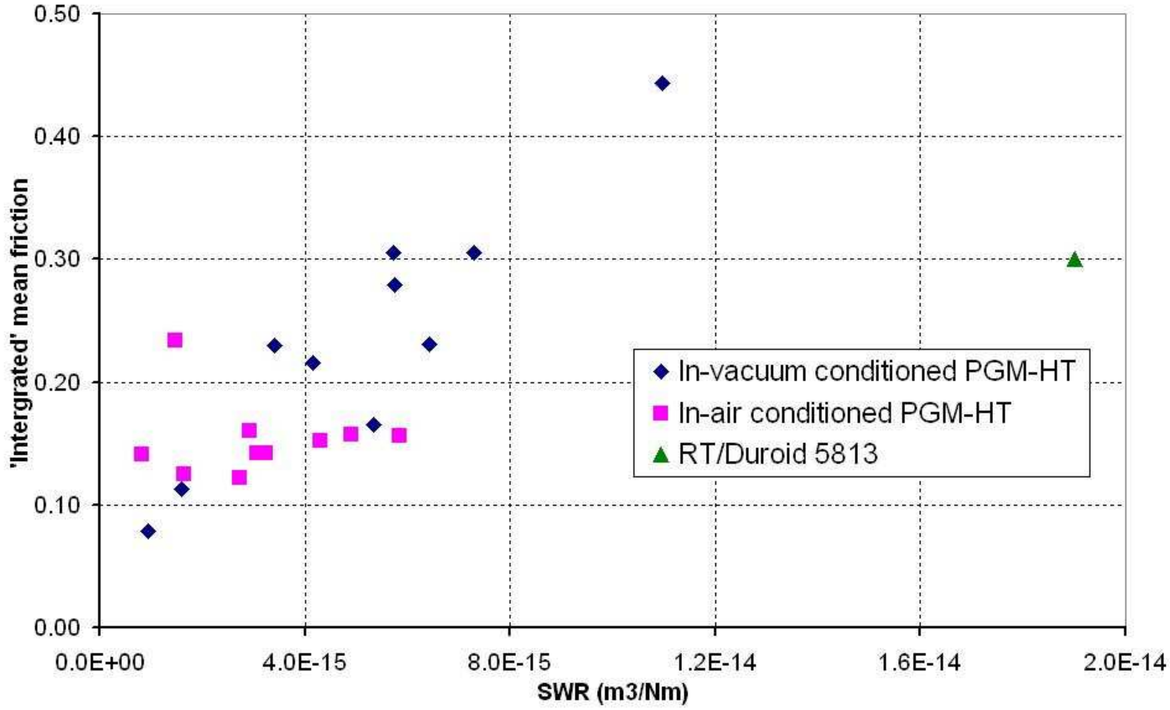


Figure 6. Integrated friction against wear rate for in-air conditioned PGM-HT, in-vacuum conditioned PGM-HT, and RT/Duroid 5813

Table 2. Summary of repeatability results for in-air and in-vacuum treated PGM-HT

Properties	In-air PGM-HT	In-vac PGM-HT [5]
Number of tests	9 (3 x 3 blocks)	9 (3 x 3 blocks)
Test conditions	Vac, 5N, $0.1ms^{-1}$	Vac, 5N, $0.1ms^{-1}$
Start-up friction	~0.20	~0.20
Steady-state friction mean	0.09	0.25
Steady-state friction range	0.06 – 0.14	0.05 – 0.45
Specific wear rate mean ($x\ 10^{-15}m^3/N.m$)	2.8	5.6
Specific wear rate range ($x\ 10^{-15}m^3/N.m$)	0.8 – 4.9	1.6 – 11.0

If we plot the integrated friction over the 1,000m against wear rate for both populations of PGM-HT we observe a separation in the populations. It is apparent from this method that the in-air PGM-HT provides greater consistency under these test conditions.

If we include on the same plot the value for Duroid (taken from [1]) we see a clear separation from both populations of PGM-HT, with the Duroid displaying an increased wear rate for a given friction coefficient (Fig. 6). As self-lubricating composites rely on sacrificial wear for their mode of operation, this result may be an indication that the rate of material transfer to a counterface is greater for Duroid than that for PGM-HT.

A similar claim has been made regarding PGM-HT and Duroid caged bearings, in which two mechanisms successfully qualified with Duriod underwent new lifetests with PGM-HT caged bearings [6]. These tests indicated torque instabilities and a lack of lubricant transfer when PGM-HT cages were employed.

2.4. Test results - characterisation

From the characterisation pin-on-disc tests it is apparent that the lowest friction coefficients are generated under vacuum, with greater values in air. The single nitrogen test did not display a steady-state during the 1,000m. Wear rates for in-air conditioned PGM-HT were not seen to be particularly dependant upon environment.

Similarly, no great dependence was observed on either the friction coefficient or wear rate if we consider sliding speed, or contact stress.

Table 3. Friction and wear results of in-air conditioned characterisation tests

Conditions	Steady-state friction	Wear rate (x $10^{-15}m^3$/N.m)
Vac, S1, $0.1ms^{-1}$	0.08	5.9
Vac, S2, $0.1ms^{-1}$	0.16	5.6
Vac, S1, $0.01ms^{-1}$	0.12	2.4
Air, S1, $0.1ms^{-1}$	0.20	3.3
Air, S2, $0.1ms^{-1}$	0.22	3.9
Air, S1, $0.01ms^{-1}$	0.19	2.3
N_2, S1, $0.1ms^{-1}$	0.21 – 0.27	1.7

These results are broadly comparable to those of the in-vacuum conditioned PGM-HT [5], with no consistent differences in either wear rate or friction coefficient.

3. SINTIMID 15M

4. TRIBOLOGICAL TESTING OF SINTIMID 15M

4.1. Test details

As with the PGM-HT, tests on Sintimid 15M were separated into two stages; repeatability and a detailed characterisation.

First stage – repeatability

Repeatability tests were performed in the same way as were carried out for PGM-HT; unidirectional sliding on 52100 steel for a distance of 1,000m. Three hemispherically ended pins of radius 18mm were machined from three independent blocks of Sintimid 15M, giving nine pins in total. Testing was carried out under the same conditions as for PGM-HT, though the mean Hertzian contact stresses were varied for the two materials due to their different elastic moduli. For clarity, repeatability tests were performed under identical conditions as those described in Test 8 in Tab. 4 below.

Second stage – characterisation

Characterisation tests were also carried out under similar conditions to those for PGM-HT. Test conditions are described below.

Unlike the PGM-HT tests, due to material constraints the pins were not machined from a single block of Sintimid 15M. Tests 16.B, 17.B, 18.B and 19.B were machined from a second block, with all other test items originating from the same block.

Table 4. Test conditions for characterisation tests of Sintimid 15M

Test ID	Test environment	Sliding speed (m/sec)	Contact stress
8	High vacuum (5×10^{-6} mbar)	0.1	S3
9			S4
10		0.01	S3
11			S4
12	Laboratory air (class 10,000)	0.1	S3
13			S4
14		0.01	S3
15			S4
16.A	Dry nitrogen	0.1	S3
16.B			
16.C			
17.A			S4
17.B			
18.A		0.01	S3
18.B			
19.A			S4
19.B			

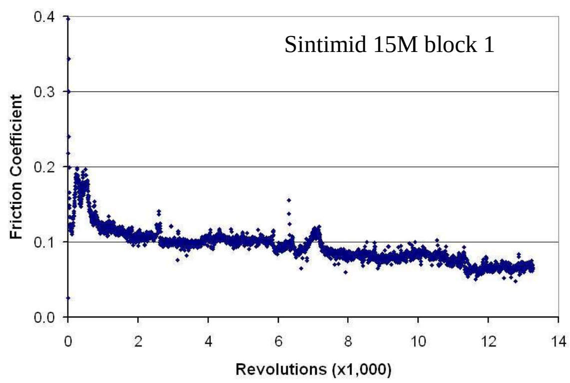
Notes: S3 equates to a mean contact stress of 23MPa (pin load 5N, Sintimid 15M pin radius 18mm)
S4 equates to a mean contact stress of 37MPa (pin load 5N, Sintimid 15M pin radius 18mm)

4.2. Pin-on-disc methodology

Testing was performed using identical kit to that previously described (section 3.2)

4.3. Test results - repeatability

The behaviour of all tests was broadly similar, demonstrating high friction initially, before quickly falling and displaying low friction throughout the remainder of the test. Some variance was noticed in the number of revs required to achieve steady-state friction, as with the friction coefficient values themselves, but not to any great extent. No great difference was observed in the results from different blocks (examples in Fig. 7).



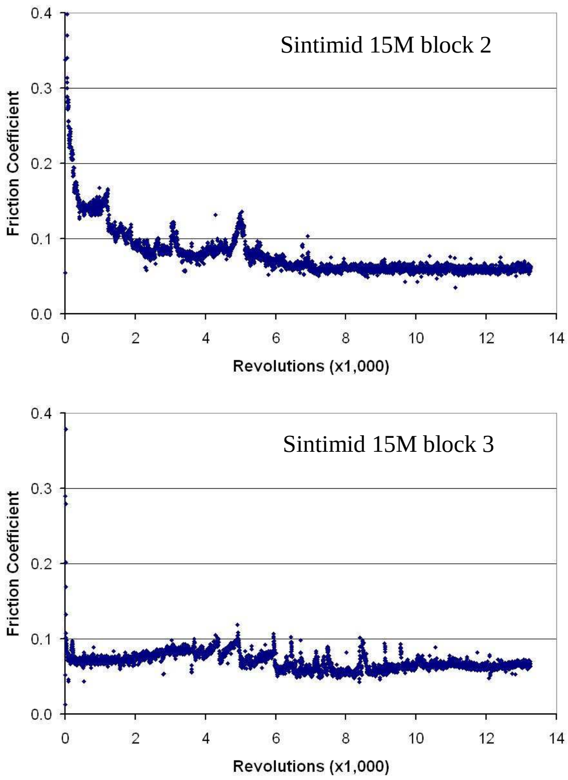


Figure 7. Friction plots from each block of Sintimid 15M material (sliding against 52100 steel, under vacuum, 5N load, 0.1ms^{-1} sliding speed)

Initial peak friction values ranged from 0.54 to 0.74 with a mean of 0.65, and were approximately 10-times higher than the steady-state friction coefficients, measured at 0.06 to 0.09 with a mean of 0.07. Wear rates were measured to be in the range 0.30 to 2.38 x 10^{-15}m^3/N.m, with a mean of 1.37 x 10^{-15}m^3/N.m.

The behaviour of these tests are similar enough for us to state that Sintimid 15M gives repeatable friction and wear rates both within, and between individual blocks of material.

4.4. Test results - characterisation

The effect of increasing load, in the cases of air and vacuum environments, was to slightly decrease steady-state friction, a behaviour previously observed [7]. In a nitrogen environment however the inverse is true, with steady-state friction increasing with increasing contact stress. No strong dependency on load was noted when considering either start-up friction or specific wear rate.

Table 5. Friction and wear results of Sintimid 15M characterisation tests

Conditions	Start-up friction	Steady-state friction	Wear rate (x 10^{-15}m^3/N.m)
Vac, S3, 0.1ms^{-1}	0.70	0.08	1.58
Vac, S4, 0.1ms^{-1}	0.68	0.07	0.31
Vac, S3, 0.01ms^{-1}	0.50	0.12	0.46
Vac, S4, 0.01ms^{-1}	0.66	0.08	0.94
Air, S3, 0.1ms^{-1}	N/A	0.81	6.75
Air, S4, 0.1ms^{-1}	N/A	0.71	5.97
Air, S3, 0.01ms^{-1}	N/A	0.75	13.94
Air, S4, 0.01ms^{-1}	N/A	0.90	50.42
N_2, S3, 0.1ms^{-1}	N/A	0.91	3.62
N_2, S4, 0.1ms^{-1}	N/A	1.06	2.19
N_2, S3, 0.01ms^{-1}	N/A	0.56	0.63
N_2, S4, 0.01ms^{-1}	N/A	0.99	0.64

Note: Tests in air and N_2 did not display initial peak friction values, but rather displayed a slower increase over the first few hundred revolutions of the test.

The effect of sliding speed on steady-state friction is not clear. Whilst in nitrogen we find a definite increase in friction with increasing sliding speed, this is not as well defined for air and vacuum testing. In air increasing the sliding speed causes a significant decrease in the specific wear rate, whilst in nitrogen an increase is observed for the greater rotation speeds.

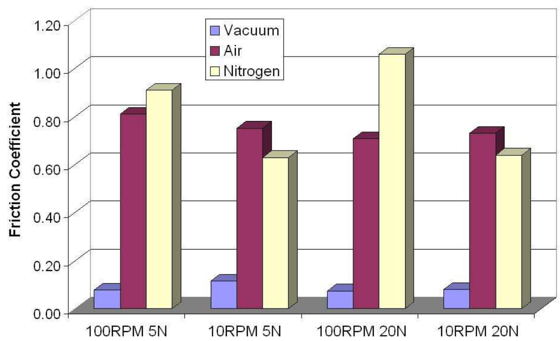


Figure 8. Effect of environment on steady-state friction of Sintimid 15M

The material was found to be very sensitive to environment. Sliding in air produced greatly elevated steady-state friction coefficients in comparison to vacuum. Specific wear rates were similarly increased in comparison to air. Considering dry nitrogen, steady-state friction coefficients were comparable to, and in some cases greater than, those found in air, but wear rates were comparable to those found under vacuum under similar conditions.

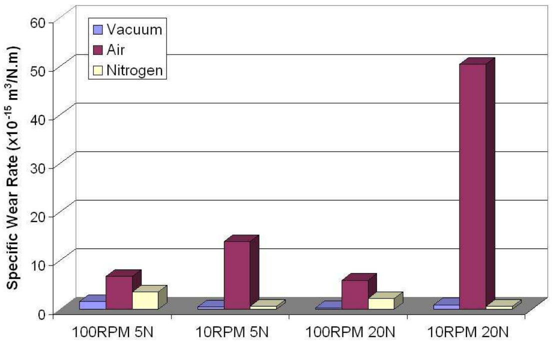


Figure 9. Effect of environment on wear rate of Sintimid 15M

4.5. Comparison with other self-lubricating composites

We now present a comparison of these results against Vespel SP-3, a similar polyimide to Sintimid 15M also containing 15% MoS_2 filler, as well as in-vacuum and in-air conditioned PGM-HT.

Table 6. Comparison of Sintimid 15M with other common self-lubricating composites

Conditions	Sintimd 15M	Vespel SP-3 [8]	In-air PGM-HT	In-vac PGM-HT [5]
	Steady-state friction coefficient			
Vac, 5N, 0.1ms^{-1}	0.08	0.05	0.08	0.06
Vac, 20N, 0.1ms^{-1}	0.07	-	0.16	0.05
Vac, 5N, 0.01ms^{-1}	0.12	-	0.12	0.08
Air, 5N, 0.1ms^{-1}	0.81	0.65	0.20	0.22
Air, 20N, 0.1ms^{-1}	0.71	-	0.22	0.21
Air, 5N, 0.01ms^{-1}	0.75	-	0.19	0.20
N_2, 5N, 0.1ms^{-1}	0.91	-	0.21 – 0.27	0.20 – 0.32
Conditions	**Sintimd 15M**	**Vespel SP-3 [8]**	**In-air PGM-HT**	**In-vac PGM-HT [5]**
	Specific wear rate x 10^{-15} m^3/N.m			
Vac, 5N, 0.1ms^{-1}	1.58	3.8	5.9	0.9
Vac, 20N, 0.1ms^{-1}	0.31	-	5.6	2.4
Vac, 5N, 0.01ms^{-1}	0.46	-	2.4	1.3
Air, 5N, 0.1ms^{-1}	6.75	10.7	3.3	2.7
Air, 20N, 0.1ms^{-1}	5.97	-	3.9	2.6
Air, 5N, 0.01ms^{-1}	13.94	-	2.3	2.2
N_2, 5N, 0.1ms^{-1}	3.62	-	1.7	3.0

The behaviour of Sintimid 15M in vacuum and laboratory air is broadly similar to that of Vespel SP-3, with a similar dependence upon environment.

Friction coefficients are also generally high (>0.6) for Vespel SP-3 during the first few revolutions in vacuum, behaviour observed here for Sintimid 15M. However Vespel has been shown to display high friction for long sliding distances before reducing to <0.1 [8]. Conversely, Sintimid 15M reduced from an initial peak much sooner (typically less that 40 revolutions, 3-meters sliding distance at ~12mm track radius), rapidly reducing to a friction coefficient of 0.1 to 0.2, before a much steadier drop over several thousand revolutions to <0.1.

Previous studies into polyimides indicate that these materials are capable of low friction and wear only at high temperatures or in environments of low water vapour [9, 10]. It is thought that the cause of this is the presence of adsorbed H_2O molecules hydrogen bonding to the polyimide chains, and preventing the formation of a thin shear surface offering low friction and wear. This can be overcome by removing the water vapour available, after which the polyimide can provide good lubrication. This effect is apparent in tests performed on Sintimid 15M under vacuum, where an initial period of high friction (providing frictional heating) is then followed by a stable period of low friction. This behaviour has previously been seen for polyimides sliding under vacuum [11].

However, this description does not explain the high friction observed for Sintimid 15M sliding under nitrogen – in such a dry environment we would expect the behaviour to be similar to that in vacuum. The root cause of this is not clear, but it is noted that high friction when sliding in dry nitrogen has been observed by ESTL previously for Vespel SP-3 [12], indicating that this behaviour may be common among polyimides.

5. CONCLUSIONS

PGM-HT

During this study, the performance of in-air conditioned PGM-HT was compared to that of in-vacuum conditioned material. Pin-on-disc testing under a variety of different environments and conditions demonstrated no significant differences between these materials. However repeat testing revealed greater consistency in the performance of the in-air pre-conditioned material. These observations would lead us to tentatively suggest that for ambient temperature applications, in-air conditioned PGM-HT is to be preferred.

However we have also observed that the wear rate for both these materials is reduced in comparison to RT/Duroid 5813. A previous investigation has highlighted the lack of material transfer with PGM-HT in comparison to Duroid when used as a bearing cage material [6], and it may be that these findings are related.

Sintimid 15M

These findings lead us to conclude that Sintimid 15M / Tecasint 1391 has potential as a viable European-sourced alternative to the US-sourced Vespel SP-3, provided consideration is given to adequately run-in the material in vacuum to overcome the initial high friction. The material performs well under vacuum, but is not suitable for use as a lubricant in air, due to high friction and excessive wear. Performance under nitrogen gives a combination of high friction with low wear. Therefore, as dry nitrogen does not replicate the performance in vacuum, any truly representative testing of this material must take place under vacuum.

Table 7. Summary of effect of environment on Sintimid 15M

Environment	Start-up friction	Steady-state friction	Wear rate
High vacuum (5×10^{-6} mbar)	High	Low	Low
Laboratory air (class 10,000)	N/A	High	Med/High
Dry nitrogen	N/A	High	Low

6. ACKNOWLEDGEMENTS

Funding for this work was provided by ESTEC through the Tribology Applications Program (TAP).

7. REFERENCES

[1] Anderson, M. J., *'Tribometer characterisation tests and definition of bearing screening test plan'*, ESA-ESTL-TM-226 01-, 1999

[2] Hampson, M. R. et al, *'Towards the effective solid lubrication of ball bearings operating at high temperature'*, Proc. 12th ESMATS Liverpool, 2007

[3] Mürer, J. A., Harper, R. & Anderson, M. J., '*High temperature antenna pointing mechanism for BepiColombo mission*', Proc. 11th ESMATS Lucerne, 2005

[4] Cropper, M., *'Cage material stability of PGM-HT and Sintimid'*, ESA-ESTL-TM-0045 01-, 2008

[5] Buttery, M. & Anderson, M. J., *'The friction and wear properties of thermally conditioned PGM-HT'*, Proc. 13th ESMATS Vienna, 2009

[6] Sicre, J. et al, *'PGM-HT as RT/Duroid 5813 replacement? Lifetime results on STD Earth scanning sensor and polder bearing shaft'*, Proc. 13th ESMATS Vienna, 2009

[7] Merstallinger, A. et al, '*Self lubricating composites for medium temperatures in space based on polyimide SINTIMID*', Proc. 11th ESMATS Lucerne, 2005

[8] Cunningham, J. & Swales, G., '*Assessment of the friction & wear performance of self lubricating polymers identified as possible replacements to RT/Duroid 5813*', ESTL-TM-196 01-, 1997

[9] Fusaro, R. L., '*Molecular relaxations, molecular orientation and the friction characteristics of polyimide films*', ASLE Trans. 20 (1), 1977

[10] Fusaro, R. L., '*Effect of atmosphere & temperature on wear, friction & transfer of polyimide films*', ASLE Trans. 21 (2), 1978

[11] Fusaro, R. L., '*Evaluation of several polymer materials for use as solid lubricants in space*', Tribology Trans. 31 (1), 1988

[12] Cunningham, J. M., '*Tribometer round-robin: A practical assessment of data repeatability & reproducibility in tribometer tests for space applications*', ESTL-TM-170 01-, 1995