

VACUUM TRIBOLOGICAL BEHAVIOUR OF SELF LUBRICANT QUASICRYSTALLINE COMPOSITE COATINGS

F. J. García de Blas, A. Román, C. De Miguel, F. Longo, R. Muelas, A. Agüero
National Institute of Aerospace Technology (INTA), Ctra. De Ajalvir km. 4, 28850 Torrejón de Ardoz (Spain)
Tel.: (+34) 915201509, Fax: (+34) 915201381, e-mail: garciadbj@inta.es

Abstract

High temperature resistant self-lubricant coatings are needed in space vehicles for components that operate at high temperatures and/or under vacuum. Thick composite lubricant coatings containing quasicrystalline alloys (QC) as the hard phase for wear resistance, have been deposited by thermal spray. The coatings also comprise lubricating materials (silver and BaF₂-CaF₂ eutectic) and NiCr as the tough component. This paper describes the vacuum tribological properties of TH103, a coating belonging to this family, with excellent microstructural quality. The coating was deposited by HVOF and tested under vacuum on a pin-on-disc tribometer. Different loads, linear speeds and pin materials were studied. The pin scars and disc wear tracks were characterized by EDS-SEM. A minimum mean steady friction coefficient of 0.32 was obtained employing a X-750 Ni superalloy pin in vacuum conditions under 10 N load and 15 cm/s linear speed, showing moderate wear of the disc and low wear of the pin.

1. Introduction

High temperature resistant self-lubricant coatings are needed in space vehicles for components that operate at high temperatures and/or under vacuum. These coatings should exhibit low shear strength, maintaining their chemical stability at extremes temperatures and in the space environment¹.

An in-space instrument (TriboLAB) capable of evaluating some tribological properties as well as the durability of solid lubricant coatings has been built as part of a joint spanish-british effort supported by the European Space Agency (ESA)². This instrument will be integrated onto EuTEF (European Technology Exposure Facility) at the International Space Station. The coatings presented in this work are being optimised to be tested at TriboLAB. If successful, these coatings could be employed in re-usable space plane applications, such as elevon hinges, where temperatures of 700 °C are reached during re-entry into the Earth's atmosphere¹. These coatings should also be capable of providing effective lubrication at lower temperatures since "cold start" operation may be necessary³, even in the space environment.

Self lubricant composite coatings containing solid lubricants in a hard matrix can be designed for adequate behaviour in the above mentioned conditions. For instance, NASA's Lewis Research Centre has developed

self lubricating composite coatings for terrestrial use comprising hard materials like chromium carbide and solid lubricant additives such as silver and BaF₂-CaF₂ eutectic on a NiCr matrix. These coatings are applied by plasma and HVOF thermal spray and significantly reduce friction coefficient improving wear resistance over a wide temperature range^{4,5,6}.

Thick composite lubricant coatings containing quasicrystalline alloys (QC) as the hard phase for wear resistance have been deposited by plasma spray and HVOF. QCs show promising tribological characteristics⁷ exhibiting a combination of adequate anti-friction properties: low friction coefficient, high hardness and high yield strength under compression^{8,9}, thermal expansion coefficients close to that of metals, high thermal stability, low thermal conductivity and good oxidation and hot corrosion resistance¹⁰. The coatings also comprise lubricating materials (silver and BaF₂-CaF₂ eutectic) and NiCr as the tough component.

Composite coatings with different composition have been developed and optimised in order to improve the microstructure of the coatings and the tribological behaviour^{2,11,12}. This paper describes the vacuum tribological properties of TH103 (AlCoFeCr, NiCr, Ag, CaF₂, BaF₂), deposited by HVOF, which so far has shown the best tribological properties. As previously described, TH103 exhibits excellent microstructural quality (low porosity and uniform phase distribution) and the main phases present in the powder are maintained in the coating². The coating was tested under vacuum on a pin-on-disc tribometer at AMTT-ARC (Aerospace and Space Materials Technology Test House – Austrian Research Centres), in Seibersdorf (Austria). Different loads, linear speeds and pin materials were studied.

2. Experimental

2.1. Materials

The discs substrate was X750 Ni superalloy.

The spray powder for TH103 was made mixing AlCoFeCr powder (SNMI Cristome BT1, 20-53 µm), NiCr (Sulzer Metco 43F-NS, < 56 µm), Ag (SEMP, < 56 µm) and BaF₂-CaF₂ eutectic (in house made from Aldrich fluorides, < 45 µm)

2.2. Deposition

The coating was deposited by a Sulzer Metco Diamond Jet Hybrid unit (model A-3120) mounted on a 6 axes robot (ABB) and fed by a twin rotation powder feeder.

2.3. Characterisation

The samples were characterised by optical and electron microscopy (JEOL JSM-840 equipped with a KEVEX EDS microanalyser).

Hardness measurements were carried out with a Future-Tech Vickers indenter under a 200 g-load, on polished cross-sections.

2.4. Wear tests

The coating was tested on an ultra high vacuum pin-on-disc tribometer at AMTT-ARC (Aerospace and Space Materials Technology Test House – Austrian Research Centres), in Seibersdorf (Austria), employing four different loads: 1, 2, 5 and 10 N, and two linear speeds: 1.5 and 15 cm/sec at 10^{-5} mbar. Three different pin materials (X-750, 100Cr6 and Al_2O_3) were used.

The roughness of the pins and discs and the pin scars and disc wear tracks were measured with a Taylor-Hobson pneumo-profilometer with a 2 microns diamond cone stylus tip.

The pin scars and disc wear tracks were also studied by EDS-SEM.

3. Results and discussion

3.1. Microstructure of the coating

Several composite coatings were developed and optimised in order to improve the microstructure of the coatings and the tribological behaviour^{2,11,12}. Among these coatings HVOF TH103 was the best one (figure 1).

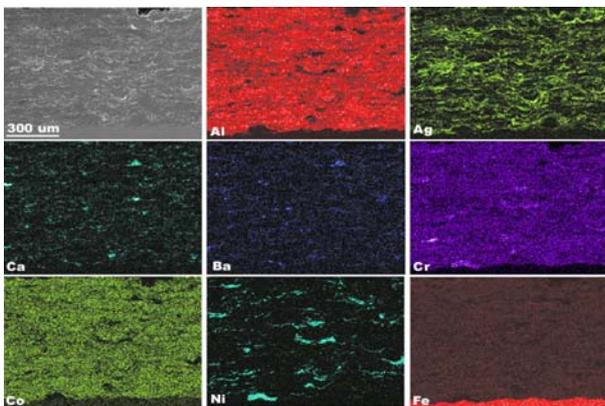


Figure 1. Microstructure of HVOF TH103 coating

TH103 coating shows excellent microstructural quality (low porosity and uniform phase distribution) and the main phases present in the powder are maintained in the coating².

3.2. Tribological behaviour of the coating

Microhardness of the coating ranges from 600 to 625 $HV_{0.2}$, confirming previous results¹¹.

Table 1 shows the wear and friction coefficients (k and μ) for the tested coating under different conditions as well as the Hertzian Pressures, P_{Hertz} , calculated dividing the applied load by the contact surface. The wear coefficients of the coated discs and pins (k_d and k_p respectively) were calculated dividing the measured worn volume by the load applied and the sliding distance.

Three different pin materials (X-750, 100Cr6 and Al_2O_3) were used. 100Cr6 is a steel commonly used in tribological applications and Al_2O_3 is a ceramic material with potential uses in high temperature tribological applications.

Pin material	L (N)	P_{Hertz} (GPa)	V (cm/s)	Cycles	μ_{st}	k_d	k_p
						$10^{-6} \text{ mm}^3/\text{N.m}$	
*X750	1	0.66	1.5	6167	0.80	N.D.	8.6
X750	1	0.66	1.5	6006	0.67	407	10.4
X750	5	1.14	1.5	6003	0.59	509	10.5
X750	10	1.43	1.5	3071	0.49	400	36.7
X750	2	0.84	1.5	1612	0.57	290	3.5
100Cr6	2	0.82	1.5	1817	0.57	339	2.1
Al_2O_3	2	1.00	1.5	6005	0.65	305	2.9
X750	5	1.14	1.5	6003	0.59	509	10.5
X750	10	1.43	1.5	3071	0.49	400	36.7
X750	10	1.43	15	39875	0.32	76.2	1.0
X750	5	1.14	15	31810	0.50	539	11.3

Table 1. Pin-on-disc wear data. *Test at atmospheric pressure; P_{Hertz} : hertzian pressure; μ_{st} : mean steady friction coefficient; k_d : disc wear coefficient, k_p : pin wear coefficient; N. D.: non detectable

The friction coefficient for the X750 material rubbing against an X750 pin on a pin-on-disc test is 0.9 under atmospheric conditions, whereas for a TH103 coated disc also rubbing against an X750 pin the friction coefficient decreases to 0.8, indicating that the coating behaves as a lubricant.

3.2.1. Influence of pressure and applied load

The load influence on the friction coefficient of HVOF TH103 as a function of the number of cycles is shown in figure 2 employing X750 pins. The atmospheric pressure (A.P.) friction coefficient at a load of 1 N is also included.

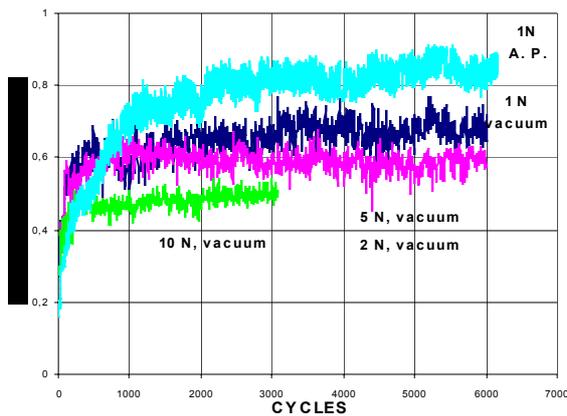


Figure 2. HVOF TH103 friction coefficient employing X750 pins at 1.5 cm/s linear speed.

SEM-EDS mapping of the tests pins indicate that no coating transfer has taken place to the atmospheric pressure pin¹¹, whereas under vacuum, for all three loads some coating material has been transferred to the pin forming a self lubricant transfer film on its surface (figure 3). This could very well explain the lower friction coefficients obtained in vacuum. Other authors have indicated that oxidation of the coating may prevent film transfer at atmospheric pressure explaining also the absence of wear on the coated disc¹³. However, debris from the pin was found on the disk track.

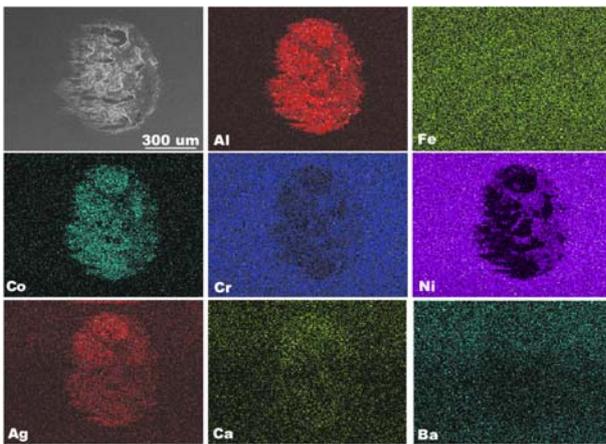


Figure 3. Mapping of the X750 pin scar, test carried out at 1 N normal applied load. Transfer film with debris on the pin scar.

Also as is shown in figure 2, the friction coefficient decreases as the applied load increases under vacuum. This effect, already observed for metal-MoS₂ composite coatings, is not fully understood¹⁴ but could be due to a higher amount of self-lubricant transfer from the coated disc to the pin at higher applied loads or to a higher lubricated contact surface. There is no evidence of transfer of material from the pin to the disc for the three

applied loads (figure 4) and the discs suffer moderate/high wear, (10^{-4} mm³/N.m), but only related to coating loss and never reaching the substrate. The pins showed moderate wear (10^{-5} mm³/N.m.).

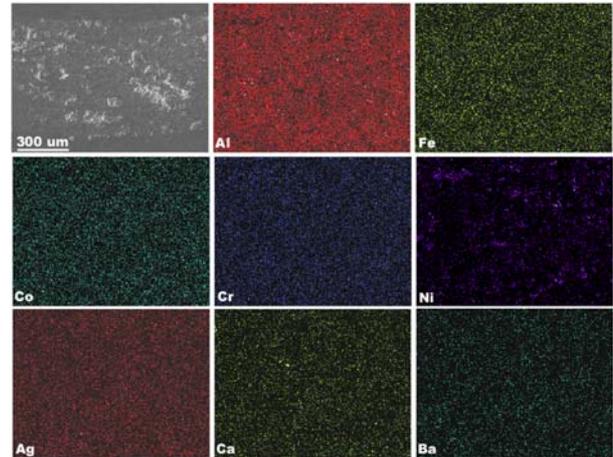


Figure 4. Mapping of the coating wear track disc after pin-on-disc test in vacuum showing no evidence of pin material on it.

3.2.2. Influence of pin materials

Figure 6 shows the friction coefficients of three different pin materials (Al₂O₃, X750 and 100Cr6) rubbing against coated discs.

Both X750 and 100Cr6 pins exhibited the same friction coefficient although 100Cr6 is harder than X750 (700 HV vs. 400 HV), probably indicating that the lubrication provided by the coating overcomes the expected higher wear from a harder material. The pin scars and the wear track discs of both pins are very similar (figures 3 and 4 respectively).

On the other hand, the Al₂O₃ pin, with a much higher hardness (1100 HV), shows a higher friction coefficient.

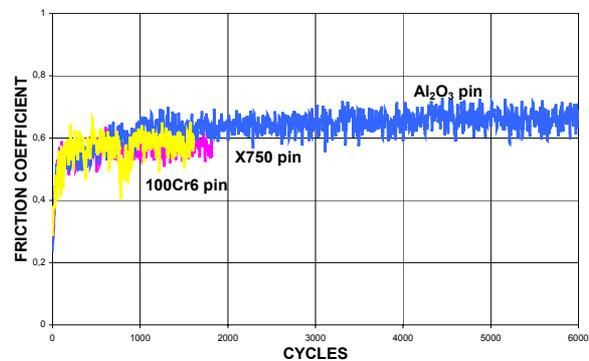


Figure 5. Influence of the pin material on the friction coefficient of HVOF TH103 coating under 2 N load and 1.5 cm/s linear speed.

The three disc wear coefficients (table 1) are moderate/high (10^{-4} mm³/N.m), while the pins exhibit low wear, 10^{-6} mm³/N.m. X750 and 100Cr6 pins are protected by the lubrication provided by the coating whereas the Al₂O₃ pin has low wear due to its high hardness and therefore better wear resistance behaviour.

3.2.3. Influence of the linear speed

The influence of the linear speed employing X750 pins at two different normal loads (5 and 10 N) is shown in figure 6.

Under 5 N, increasing the linear speed results in a reduction of the friction coefficient, but the wear coefficients of both discs and pins remain very similar (moderate/high wear for the discs at 10^{-4} mm³/N.m, and moderate wear for the pins, 10^{-5} mm³/N.m). Under a 10 N applied load, incrementing the linear speed also causes a lower friction coefficient, which tends to decrease as a function of the number of cycles, reaching a value of 0.32 after 40,000 cycles. Moreover, the wear coefficient of both disc and pin are significantly reduced to moderate (10^{-5} mm³/N.m) and low (10^{-6} mm³/N.m) respectively.

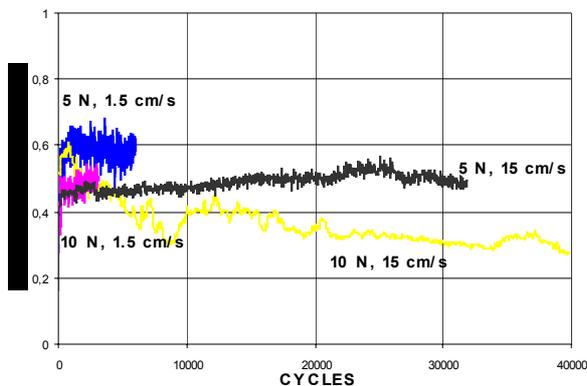


Figure 6. Influence of the linear speed on the friction coefficient of HVOF TH103 coating at different loads

A larger amount of lubricant material transferred to the pin due to the higher pressure can account for the lower coefficients of friction and wear as can be seen in figure 7 as compared to figure 3. Moreover, no pin material debris was found on the disc scar. In addition, the higher speed will likely cause a local increase in temperature, and previous results with this type of coating have shown significant reduction of the friction coefficient at high temperatures¹¹.

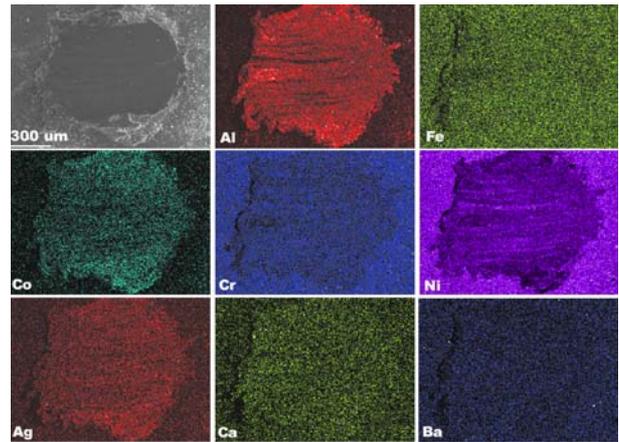


Figure 7. Mapping of the X750 pin scar from test under 10 N load at 15 cm/s linear speed. Complete transfer film without debris on the pin scar.

These results can be interpreted by a mechanism implying an initial transfer of lubricant material from the coating to the pin until proper efficient lubrication is obtained, explaining the reduction on the friction coefficient as a function of the cycles. Once the amount of transferred material is enough to cause good lubrication between the pin and the disc, the disc wear stops or becomes insignificant. Table 2 shows the disc worn track cross section areas for the experiments carried out under 10 N and 15 cm/s as a function of the number of cycles. Initially, the worn track cross-section grows in size while coating material is being transferred from the disk to the pin, then from 12,000 to 40,000 cycles, there is no significant difference between the disc track cross-section size, indicating insignificant or undetectable wear of the disc.

Number of cycles	Disk track worn area (mm ²)
3,000	1.2×10^{-2}
12,000	3.7×10^{-2}
40,000	3.0×10^{-2}

Table 2. Disc worn track cross section areas for the experiments carried out under 10 N and 15 cm/s as a function of the number of cycles.

4. Conclusions

The results of pin-on-disc testing indicate that the HVOF TH103 self lubricant quasicrystalline composite coating improves the tribological behaviour of X750 and is a better self lubricant in vacuum than at atmospheric pressure conditions. This is due to the generation of a transfer film over the pin scar under vacuum, whereas at atmospheric pressure no transfer of material from the coating to the pin takes place and the coating behaves as a wear resistance material rather than as a lubricant.

Increasing in the normal applied load results in a reduction in the friction coefficient probably due a transfer of higher amounts of coating material from the disc to the pin or to a higher lubricated contact surface.

Very hard pin materials such as Al₂O₃ cause an increase in the friction coefficient of TH103.

A friction coefficient of 0,32 is attained under a 10N applied normal load and at 15 cm/s linear speed after 40,000 cycles. Results are indicative of an initial coating transfer from the disc to the pin thereby “coating the pin” until very good lubrication is obtained. No significant disc wear takes place after transfer of a critical amount of coating is attained under these conditions.

In any of the tests carried out with the coated specimens the wear scar reached the substrate material.

Future work will be focused in understanding the transfer mechanism in a vacuum pin-on-disc test and the possible influence of the oxygen content in the starting powders in the coating tribological properties.

The best coatings after on-ground optimisation and qualification will eventually be tested in a tribometer (TriboLAB instrument) that will be integrated onto de EuTEF (European Technology Exposure Facility) at the International Space Station (ISS).

5. Acknowledgments

The authors wish to acknowledge the valuable contribution from the personnel at the Metallic Materials Area of INTA.

The vacuum pin-on-disc tests had been carried out in the Aerospace and Space Materials Technology Test House – Austrian Research Centres (AMTT-ARC) in Seibersdorf (Austria) under MRI contract n. PRI-CT-1999-00024.

This work has being partly financed by Spanish Ministry of Science and Technology of Spain under project PNE-008/2000-C-01.

6. References

¹ E. W. Roberts, M. J. Anderson, S. G. Gould, “Protective Coatings and Thin Films”, p.135, Kluwer Academic Publishers (1997)

² J.I. Oñate, M. Brizuela, A. García-Luis, J.L. Viviente, J. García de Blas, A. Agüero, F. Longo and A. Román, “Development and qualification of new solid lubricant

coatings. A tribology experiment at the TriboLAB onto EuTEF”. Proceedings of 8th International Symposium on Materials in a Space Environment and 5th International Conference on Protection of Materials and Structures from the LEO Space Environment. Arcachon (France). 5-9 June 2000. Ed. CNES

³ M.J. Anderson and E.W. Roberts, “An Assessment of Solid Lubricant Films for Use in High Temperature Space Applications”, ESA SP-334, 379-384, April 1993

⁴ C. DellaCorte, Tribology Transactions, vol. 43 (2000), 2, 257-262

⁵ C. DellaCorte and J. A. Fellenstein, NASA TM-107522, June 1998

⁶ M. K. Stanford, C. DellaCorte, NASA/TM-2003-212125, February 2003

⁷ S. S. Kang, J.M. Dubois, Philosophical Magazine B, 66, 151 (1992)

⁸ J.M. Dubois, S. S. Kang and J. Von Stebut, J. Mater. Sci. Lett. 10, 537 (1991)

⁹ B. R. Lawn and V.R. Howes, J. Mater. Sci. 16, 2745 (1981)

¹⁰ Sánchez, A.; García de Blas, J.; Algaba, J.M.; Álvarez, J.; Vallés, P.; García-Poggio, M.C.; Agüero, A. "Application of quasicrystalline materials as thermal barriers in aeronautics and future perspectives of use for these materials". Materials Research Society, Symposium Proceedings Volume 553, Quasicrystals, 447-458. MRS, Warrendale, Pennsylvania.

¹¹ A. Román, A. Agüero, C. de Miguel, F. J. García de Blas, F. Longo, R. Muelas, A. Sánchez. “Characterisation of tribological quasicrystalline composite coatings” Proceedings of International Thermal Spray Conference 2002, 419-423, Essen (Germany).

¹² F.J. García de Blas, A. Agüero, F. Longo, A. Román, A. Sánchez, “High Temperature Self Lubricant Quasicrystalline Composite Coatings”, poster presented at the International Conference on Metallurgical Coatings and Thin Films, April 30, 2001

¹³ C. Donnet, Surface and Coatings Technology 80 (1996) 151-156.

¹⁴ V.C. Fox, N. Renevier, D.G. Teer, J. Hampshire and V. Rigato, Surface and Coatings Technology 492 (1999) 116-119.