Development of Tribological Coatings for Aerospace and Missile Applications

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ABSTRACT

Tribological coatings play a key role in enhancing the performance and service life of engineering systems and components. Over the years, DMRL has worked extensively in studying various aspects of tribological coatings used in aerospace and missile propulsion systems where harsh and hostile environments prevail. The coatings have been deposited mostly by thermal spray processes for these applications. The studies have been done to understand the relationship between microstructure, mechanical properties and tribological behaviour of coatings. In this work, a cermet coating based on carbide to resist wear and an abradable coating based on cobalt alloy to control clearance between the blade tip and shroud for gas turbine aeroengine application and a low friction coating developed for a missile application will be discussed.

Keywords: Tribology; Carbide coatings; Abradable; Carbon based composite; Thermal spray

1. INTRODUCTION

Coatings for tribological applications have been used widely in several engineering systems and components. In many of the applications in aerospace and defence systems, the components have to operate under harsh conditions involving high mechanical and thermal stresses in oxidising or corrosive environment. As the component materials are optimised to possess good bulk properties (strength, fatigue, creep) to withstand static and dynamic loads, they are vulnerable to surface degradation processes like wear, erosion, oxidation and corrosion. It is imperative to modify the surface to improve tribological properties to control wear and friction. The Tribology Group in DMRL has worked extensively on several tribological coatings for different protective and functional requirements. The focus of research and development activities has been to provide tribological solutions for applications in aero-engine, missiles and armaments. In the present article, a brief account of the research activities carried out at DMRL is discussed.

2. CARBIDE COATING FOR WEAR RESISTANCE

Thermally sprayed chromium carbide-nichrome based coatings are widely applied on different engineering components operating in ambient and corrosive atmospheres. They are used for protection against wear by sliding, abrasion and erosion over a wide range of temperatures up to $850 \,^{\circ}C^{1-2}$. The coatings are deposited usually by high velocity coating processes like High Velocity Oxygen-Fuel (HVOF), High Velocity Air-Fuel (HVAF) and Pulsed Detonation Gun Spray (DS) processes to

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minimize decarburization and provide a dense, well-adherent and a hard coating with good wear resistance properties³⁻⁹. These coatings are used most commonly that is next only to tungsten carbide based coatings that are preferred for service temperatures up to 500 °C due to the better thermal stability of chromium carbide. The high carbon containing Cr₃C₂ carbides possessing high hardness are usually used in combination with NiCr alloy binder in the pre-alloyed or composite powder form for thermal spray coating deposition. The coating composition containing hard Cr₃C₂ carbide phase with 20 to 25 wt% ductile metallic NiCr binder is commonly used for many wear resistance applications. The microstructure in thermally sprayed Cr₂C₂-NiCr coating is largely determined by the spray technique, spray parameters and the characteristics of the feedstock powder. During high velocity thermal spraying, the small spray particles (conventionally 5-50 µm) experience rapid heating by the combustion gases and simultaneously accelerate to high velocities (> 600 m/s) towards the substrate. The binder melts partially or completely that causes some amount of carbide dissolution in the molten binder in-flight. The extent of carbide dissolution depends on particle size and temperature. Upon impingement on the substrate, the particles are rapidly quenched resulting in a complex coating microstructure consisting of primary carbides within a supersaturated binder in amorphous or nanocrystalline phase that is metastable^{8,10-11}.

As the Cr_3C_2 -NiCr coating is used over a wide temperature range, the as-sprayed coating microstructure transforms to a more stable microstructure when exposed to elevated temperature. As part of this research work, systematic studies were undertaken to understand the effect of heat treatment on: (i) the carbide volume fraction in the coating and (ii) the binder microstructure and correlation with its mechanical properties.

2.1 Effect of Heat Treatment on Carbide Volume Fraction in the Coating

The carbide volume fraction was measured in the detonation gun (D-gun) sprayed Cr_3C_2 -20 (NiCr) coating in as-sprayed and annealed conditions to trial the extent of secondary carbide formation. The annealing was done at different temperatures up to 800 °C for 90 mins. The volume fraction of carbides in



Figure 1. Variation of carbide volume fraction in Cr_3C_2 -20(NiCr) coating deposited by D-Gun spray process as a function of annealing temperature.



the coatings was measured using image analysis technique. For this, about 8 back scattered electron (BSE) images obtained from different locations in the transverse section of the polished coating specimen were first taken. Then, the carbide volume fraction was determined by selecting the appropriate range of grey scale values in the Biovis image analyzer using the BSE image.

Figure 1 shows the variation of carbide volume fraction with increase in annealing temperature. The carbide volume fraction is the total carbide fraction i.e. primary and secondary carbides present in the coating. The plot shows the average carbide volume fraction with scatter of one standard deviation.

The as-sprayed coating contained an average carbide volume fraction of 31 % that is mainly primary carbides. The low carbide content may be attributed to significant dissolution of carbide in the binder¹². The reduced carbide content may also occur due to the rebounding off of large carbides or unmelted spray particles from the coating surface during spraying¹³. The carbide volume fraction in the 400 °C annealed coating was close to the carbide vol. fraction in as-sprayed coating. For 600 °C annealed sample, the average carbide volume fraction was 37 %. But scatter in data was wide that may be attributed to varying size distribution of the secondary carbides precipitating in the supersaturated binder. As most of the carbide precipitates were too fine (tens to few hundred nanometers),



Figure 2. a) and c) SEM micrographs of HVOF Cr₃C₂-50(NiCr) coating in as-sprayed and 600 °C annealed conditions, b) and d) Bright field TEM images of a portion of the corresponding coatings.

it is possible that some of them may not be accounted in the measurements. At 800 °C annealing, when secondary carbides coarsen significantly, the average carbide volume fraction measured was about 48 %. It is to be mentioned that the coating powder as well as the as-sprayed coating consisted of largely Cr_3C_2 type carbide and some amount of Cr_7C_3 type carbides. However, it was observed that the secondary carbides that precipitate are mainly Cr_3C_2 type carbides. As the temperature increases, nucleation of fine carbides (secondary carbides) in supersaturated NiCr alloy binder regions occur increasing the carbide volume fraction. Due to these microstructural changes the hardness of the coating increases⁸.

2.2 Effect of Heat Treatment on Binder Microstructure and Mechanical Properties

It is well known that a ductile binder improves the wear performance in cermet coatings14-15. But the influence of microstructure of the binder on the behaviour of mechanical properties in thermally sprayed carbide based coating is not clearly understood. This is due to the very limited studies on the micro-mechanical behaviour done exclusively on the binder phase. For this study, HVOF coating based on commercially available Cr₃C₂-50(Ni20Cr) material was used to evaluate the binder properties. The Cr₂C₂-50(NiCr) coating is used for wear resistance application against hard surfaces and abrasive particles at elevated temperatures between 540 - 815 °C in a highly corrosive environment like steam power generation plants. It produces very tough and dense coating with good adhesion with the substrate. The presence of higher binder content in this coating material aids in characterising the intrinsic properties of binder phase. The HVOF deposited Cr₂C₂-50(Ni20Cr) coating in the as-sprayed and annealed conditions were studied to examine the microstructure and mechanical properties of the binder phase using electron microscopy and micro/nano indentation techniques, respectively. The coating was annealed up to 800 °C for 90 mins in this study.

Figure 2(a) shows the SEM micrograph of the as-sprayed Cr_3C_2 -50(Ni20Cr) coating in which primary carbides (dark

regions) are distributed uniformly in the binder. The binder region shows varying grey-scale distribution indicating difference in the extent of carbide dissolution in the metallic NiCr binder during thermal spraying¹⁰. The bright field TEM image given in Fig. 2(b) shows the binder region consists of both amorphous and nanocrystalline phases. The SEM micrograph of 600 °C annealed coating (Fig. 2(c) shows fine precipitates of carbide (i.e., secondary carbides) distributed in the matrix phase in addition to primary carbides. Also, it showed (Fig. 2(d) coarser crystalline NiCr-based binder with grain size lower than 150 nm. Further, Cr_3C_2 type secondary carbides were largely present in the NiCr binder region as XRD detected only this carbide phase in the coating annealed at 600 °C.

Typical L–D (load–displacement) curves obtained by nanoindentation tests on coatings performed within the binder region in different annealed conditions are shown in Fig. 3. The difference in these curves reflects the changes in the response of the indentation behaviour of the binder owing to changes







Figure 4. The nanohardness and ductility in the NiCr binder and microhardness of HVOF Cr₃C₂-50(Ni20Cr) coating in As-sprayed and annealed conditions.

in the microstructure due to annealing. For comparison, the L–D curve for primary carbide obtained under similar test conditions is also shown.

The nanohardness (in HV scale) of the binder for different annealing temperatures of the coating determined from the L–D curves based on the method proposed by Oliver and Pharr¹⁶ are shown in Fig. 4. The hardness values of the carbide particles were in the range 1800 HV to 2000 HV. In case of as-sprayed coating, the average nanohardness value in the binder region was 978 HV that increased to 1241 HV (average value) for 400 °C annealed coating. On further annealing, it decreases. This trend in hardness variation is similar for both micro- as well as nanoindentation tests.

However, absolute microhardness values are lower up to 600 °C annealing temperature. This is due to the fact that the volume of indentation is quite large covering several microstructural features that may include defects like porosity / voids and inter-splat regions and it encompasses many splat structures. On the other hand, in nanoindentation, the probability of determining the intrinsic properties of the binder is higher due to very small sampling volume involved in nanoindentation measurements. The results suggest that the NiCr based binder consisting of a mixture of amorphous and nanocrystalline phases in as-sprayed coating has a lower hardness value compared to the binder consisting of ultra-fine chromium carbide precipitates in the nanocrystalline binder (as observed in TEM image of coating annealed at 400 °C). This shows that the presence of nanocrystalline grains with ultrafine carbide precipitates in a splat structure may contribute to an increase in coating hardness in addition to sintering that may occur at inter-splat boundaries. Further, annealing at 600 °C and above, the hardness of NiCr-based binder reduces possibly due to coarsening of both the metallic grains and the chromium carbides in the binder. In 800 °C annealed coating,



the nanohardness value of the binder is much lower than its microhardness value due to essentially the metallic Ni binder.

A similar trend in microhardness was also observed in case of D-gun sprayed Cr₃C₂-20(NiCr) coating where peak in hardness occurred at 600 °C. The ductility of the binder phase in Cr₃C₂-50(NiCr) coating at different annealing temperatures was also obtained from the L-D curves. In case of an elastoplastic indentation response of a material, the ratio of plastic work, W_p to the total work, W_t indicates the degree of plastic flow which is taken as a measure of ductility in a material¹⁷. The plastic work (W_n) is calculated from the area enclosed by the loading and the unloading curve and the area under the loading curve gives the total work (W_t) . The W_p/W_t ratio in binder region for the present coating system (Cr_2C_2 -50(NiCr)) for different annealing temperatures is also shown in Fig. 4. The W₂/W₂ ratio for the binder in as-sprayed coating (consisting of a mixture of amorphous and nanocrystalline phases) is about 0.58. At 400 °C annealing, the W_p/W_t ratio increases slightly to 0.62. It shows that presence of partially amorphous phase in the as-sprayed coating may reduce some ductility. In coating annealed at 600 °C and 800 °C, the values of W,/W, ratios in the binder were 0.73 and 0.81, respectively. The W_{μ}/W_{μ} ratios, measured under identical indentation test conditions, for Cr₂C₂ carbide particles and pure Ni (electrolytic) were 0.5 and 0.92, respectively. Thus, it suggests that with increase in annealing temperature the binder becomes increasingly ductile. This change in the mechanical properties occurs mainly due to compositional variation in the binder that leads to enrichment of Ni as well as coarsening of the crystalline binder phase.

3. ABRADABLE COATING FOR DYNAMIC SEALING

In aero-engines, the clearance between the blade tips and the shroud has to be as low as possible to reduce or



Figure 5. SEM micrographs of cross-section of air plasma sprayed Co based abradable coating a) As-deposited coating, b) Heat treated coating, c) and d) are high magnification micrographs of as-deposited and heat treated coatings, respectively.

prevent the gas leakage in the clearance. Due to a number of uncontrollable factors like centrifugal and thermal expansion of the components, vibration etc. during the operation of the engine the optimum clearance necessary is difficult to estimate. A sacrificial coating material is usually applied on the shroud which allows the rotor blade tip to rub against the coating without damaging the rotating blade and the shroud and thereby achieving optimum clearance during the service of the engine. This coating referred as abradable or clearance control coating provides dynamic sealing between the rotating blade tips and the shroud and prevents damage of shroud and the blades. The application of abradable coating also improves the thermal efficiency and overall performance of the aeroengine¹⁸⁻²¹. Several abradable coating materials are applied on different sections of the aero-engine depending on the service temperature and rubbing contact materials (blade and shroud).

To evaluate the behaviour and performance of abradable coatings, it is essential to test these coatings under similar contact conditions that exist during the rubbing of blade tip with the shroud in the aero-engine. The conventional tribological tests like pin-on-disc, block-on-ring, disc-on-disc etc. are not suitable for evaluating these coatings. A novel test rig called the Clearance Control Test Rig was indigenously developed and set up at DMRL to evaluate the abradable coatings. This test rig is a scaled down test facility that simulates the intermittent rubbing and incurring of the blade tip with the shroud. The testing is done under ambient conditions and the maximum blade tip speed achievable is 50 ms⁻¹. The detailed description of the test rig and the test procedure is given in Ref²². Several abradable coating materials have been tested using this test facility at DMRL²³. In this article, studies carried out on Co based abradable coating used in high pressure compressor section of an aero-engine will be described briefly.

A commercially available CoNiCrAlY-15wt.% Polyester-4wt.% Boron Nitride (Metco 2043) abradable coating was deposited on Ni base alloy substrate by air plasma spray process with a coating thickness of 1 mm. The clearance control tests were carried out to evaluate the abradable characteristics of the Co based composite coating by allowing a single IN718 blade specimen to incur and rub against the coating. The dimensions of the coated specimen and the blade specimen were 50 mm x 50 mm x 3 mm and 12 mm x 12 mm x 1.6 mm, respectively. The blade tip speed, incursion speed and depth of blade incursion were 50 ms⁻¹, 150 μ ms⁻¹ and 250 μ m, respectively.

The Co based composite coating microstructure is shown in Fig. 5. It shows the bright region consisting of the Co based alloy phase interspersed uniformly with polyester and hexagonal BN in the dark region. The coating material was abraded predominantly by cutting mechanism when the blade tip rubs against the coating during the clearance control tests. Figure 6 shows the as-sprayed coating rubbed by the blade tip. The cutting type of material removal is considered to be beneficial for abradable coatings²³⁻²⁴. The peak normal load and the tangential load during the rubbing interaction between the blade tip and the coating were low resulting in low coefficient of friction indicating smooth rubbing interaction. However, there was predominant blade wear due to slightly higher hardness of the coating (285 HV) compared to the blade



Figure 6. Photograph of Co-based abradable coating after rubbing with Ni base alloy blade tip. (a) As-sprayed coating and (b) Heat treated coating.



Figure 7. Mass loss of the Co-based abradable coating and the Ni-based blade when blade tip rubs against the coating in as-sprayed and heat treated conditions.

(265 HV). The coating and the blade mass loss are shown in Fig. 7. Such blade wear is not desirable. Some samples were heat treated to remove the polyester content by decomposition. The heat treatment was carried out at 450 °C for 4 hrs in ambient air. The microstructures of the heat treated coating are shown in Fig 5b and 5d. The heat treatment increases the volume of porosity and improves friability and abradability of the coating²⁴⁻²⁵. The heat treated samples were tested in the clearance control test rig under similar conditions as followed for as-sprayed coatings. Figure 6 shows the heat treated coating rubbed by the blade tip under similar test conditions. It was observed that the cutting mechanism in the latter coating was much smoother with lower tangential load in comparison to the former coating. There was predominant mass gain of the blade indicating transfer of coating material onto the blade tip. The low mass transfer minimises blade damage. The heat treated coating showed better abradable behaviour.

It is pertinent to address the relevance of ambient condition study for coating meant to be used at elevated temperature. The abradable coating is used at elevated temperature. The study was done to understand the behaviour of the coating under ambient conditions in as-sprayed and heat treated conditions using the Clearance Control Test (CCT) rig set up in DMRL. The CCT rig is a scaled down test rig in which the maximum blade tip speed attainable is 50 ms⁻¹ and the tests can be done only under ambient conditions. Also, during the engine start up, the blade tips rub against the coated shroud under ambient conditions. Thus, the tests under ambient conditions provide useful information.

4. LOW FRICTION COATING FOR A MISSILE APPLICATION

Carbon based composites are widely used in missile applications owing to their low specific weight, high strength, stiffness and good resistance to ablation²⁶⁻²⁸. A missile application requiring deflection of shaft within a journal bearing was failing by fracture due to the generation of high torque between the mating components during static bed ground tests. The components were both made of carbon based composite materials and experienced temperatures more than 500 °C. Systematic studies were carried out to understand the cause of failure. A high temperature tribometer in DMRL was used to simulate the sliding contact conditions between the mating components. The test coupons of same component materials were used for testing in a pin-on-disc configuration. The tests were done under similar sliding conditions at different test temperatures. The friction traces for self-mating of the carbon based composite at different test temperatures are shown in Fig. 8. The steady-state coefficient of friction (COF) was 0.25 at room temperature and at 400 °C. The steady state COF increased to a high value of 0.6 at 600 °C and reduced slightly to 0.55 at 800 °C.



Figure 8. Coefficient of friction vs time for self mating of bare carbon based composite material at different test remperatures in pin-on-disc configuration. Test condition- Normal load: 60 N, Sliding speed: 0.14 ms⁻¹ and Environment: Ambient.

A suitable coating was developed to provide low friction for this application by DMRL. A composite coating based on cobalt base alloy containing significant amount of high temperature solid lubricant was selected for the application. The composite coating powder was an air plasma sprayable grade material. The coating was initially deposited with optimised spray deposition parameters on a metallic test coupon to characterise the coating. The microstructure of the









coating is shown in Fig. 9. The coating consists of matrix of cobalt based alloy represented by bright regions and the dark regions interspersed within the matrix were solid lubricant.

The coating was then deposited on the discs made of actual component material with optimised spray parameters.

The sliding wear tests were performed on the coated disc with pin of the same material as counterface. The sliding tests were done at room temperature, 600 °C and 800 °C. Figure 10 shows the friction traces for the coated carbon based composite at different test temperatures. The friction traces for the uncoated composite disc sliding against bare composite pin is also shown in Fig. 10a for comparison. In case of coated carbon based composite the steady state COF was 0.23 at room temperature. At higher test temperatures of 600 ° C and 800 °C, the COF was close to 0.3 as shown in the Fig. 10b. The steady state COF in case of uncoated composite the COF is 0.25 while at 600 °C and 800 °C, the COF is 0.55 and 0.5, respectively. The coating was able to provide low friction at the desired operating conditions. The static bed tests conducted on actual components with coating performed satisfactorily even under extended test duration without failure.

5. CONCLUSIONS

The research and development of coatings for tribological applications required for different defence systems have been carried out at DMRL. In this article, research works on a wear resistant carbide coating, a high temperature abradable coating and a low friction coating for high temperature application are presented. The as-sprayed chromium carbide based coating deposited by high velocity spray process produces a dense hard coating but has low primary carbide content due to the dissolution of some carbide in the NiCr binder. With heat treatment the volume fraction of the carbides increases due to precipitation that occurs in the supersaturated metallic binder. A concomitant change in the microstructure and composition of the binder phase also occurs. The microstructural changes that occur with heat treatment of the coating affect its mechanical properties. The abradable coating in the as-sprayed condition resulted in predominant wear of the blade tip during rub interaction test. There was predominant mass transfer from the coating onto the blade tip when the coating is heat treated resulting in better abradable coating behaviour. A low friction Co based coating based on thermal spray process was developed successfully for C based composite component for a missile application.

The work highlights the studies on different tribological applications in defence systems, particularly aero-engine and missile systems. As the tribological performance of a component sub-system significantly affects the performance of a system, the studies have to be done systematically. It is imperative to understand the tribological issues, material system (including coating system) that forms part of the tribosystem, perform testing that closely simulate the tribological interactions and carry out analysis and interpretation of results to provide suitable tribological solution.

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