

Cubic Titanium Trialuminide Thermal Spray Coatings—A Review

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The recently discovered Cr-stabilized cubic titanium trialuminides of the form $(Al,Cr)_3Ti$ exhibit excellent oxidation resistance up to 1200 °C and have formed the basis for development of a new family of protective coatings. These intermetallic compounds can be fabricated into powders and thermal spray coatings much the same as traditional metal alloys. Cubic trialuminide coatings have physical properties that are compatible with a variety of common engineering materials, including alloys based on Ti, TiAl, Fe, Ni, and Al. Typically, the coatings will impart sufficient protection to permit an increase in the service temperature of a substrate alloy by 150 °C, or more. The purpose here is to summarize the development of these new thermal spray coatings, including properties and microstructures, as well as performance of the coating on various substrates. A brief comparison is made between the deposition processes used to date, which include low-pressure plasma spray (LPPS), air plasma spray (APS), and high-velocity oxy-fuel (HVOF) deposition. Recent successes in modifying the coatings to a composite form by incorporating a very fine dispersion of nanoscale carbide particles are also discussed.

Keywords abrasion, fatigue, oxidation, sliding wear, titanium aluminides, titanium trialuminides

1. Introduction

Binary Al_3Ti , from which the cubic trialuminides are derived, is an extremely brittle line compound and a strong alumina former, but offers little resistance to high-temperature oxidation because of the inevitable presence of some Al or lesser aluminides in the microstructure.^[1] Ternary alloying can change the tetragonal crystal structure to a cubic $L1_2$ phase that exists over a range of compositions. Among the possible alloying element choices, Cr and Mn additions produce alloys with the best properties.^[2-4] This is reflected in the development of considerable compressive ductility along with significant tensile ductility, a decrease in shear modulus, and a greater resistance to fracture. Despite the large amount of Al in the alloys, a significant fraction of the room-temperature yield strength (which is ~250 MPa) persists to unexpectedly high temperatures, giving density-compensated strengths in the range of the nickel-base superalloys at 1000 °C.^[5] Much of the work to date has focused on alloys near the composition $Al_{66}Cr_9Ti_{25}$, but recent studies have shown that quaternary alloying to replace some of the Ti can increase the tensile ductility.^[6,7]

The properties of the ternary Cr-Ti trialuminide that make it attractive as a coating alloy include outstanding oxidation resistance up to 1200 °C,^[8] low density, and microstructural stability at elevated temperatures derived from its intermetallic nature. Also, the coefficient of expansion is in the range of 12 to 17×10^{-6} cm/cm/°C, from room temperature to 1200 °C,^[4] providing a good match with several important engineering material substrates. Further, excellent quality powders of the alloy suitable for ther-

mal spray feedstock have been produced by gas atomization and are now available commercially. It should also be emphasized that the low density of the alloy provides an average coating coverage per unit mass, for a given coating thickness and deposition efficiency, that is twice that of competing coating alloys.

Despite the attractiveness of the good specific mechanical properties of low density Ti alloys for use in rotating and reciprocating engine parts, their poor oxidation resistance poses serious problems, and there are few effective protective coatings for these alloys.^[9-11] Therefore, initial work with the new trialuminide alloys focused on development of coatings for protecting γ -TiAl alloys slated for use in various aeroengines. This led to effective protection of those alloys, such as Ti-47Al-2Cr-2Nb, up to 900 °C in long-term cyclic oxidation tests with coatings deposited by means of both low-pressure plasma spray (LPPS) and air plasma spray (APS). Further, as will be described below, high-temperature, high-cycle fatigue testing has shown no significant effects of the presence of the coatings on the fatigue properties of these alloys. Subsequent work also produced successful coatings on Ti-6Al-4V, Ti-6242, as well as on several steels, nickel-base alloys, and Al alloys. In the latter case, the coating provides a surface with considerably enhanced resistance to wear and abrasion, with the coatings proven to be stable during long time heating at 450 °C, which is a very aggressive temperature for the Al alloys.

The intent here is to review the development of these new trialuminide coatings with examples of both the as-deposited and oxidation-tested microstructures as well as the coating properties. In addition, recent successes in modifying the coatings with an ultrafine dispersion of nanoscale carbide particles are discussed.

2. Production of Thermal Spray Powders

A general requisite for producing acceptable quality thermal spray coatings is the availability of powders with adequate

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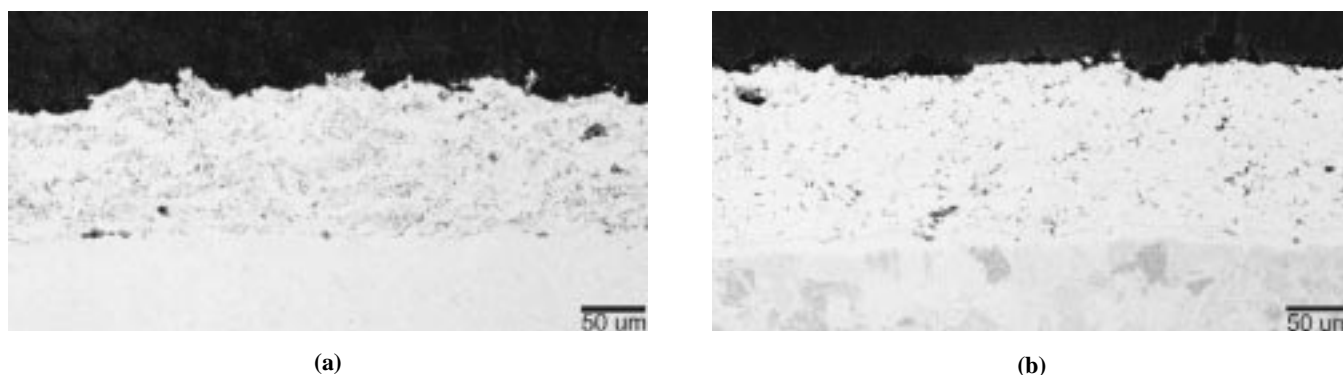


Fig. 1 Ternary trialuminide coating deposited by LPPS on Ti-47Al-2Cr-2Nb substrate. Samples were etched with 10 mL HNO₃, 5 mL HF, and 85 mL H₂O solution. (a) As-deposited coating and (b) after cyclic oxidation testing for 211 h at 815 °C

Table 1 VPS parameters for deposition of cubic trialuminide (type AL 153 powders)

Lab	Plasma spray system/gun	Spray distance	Gun current	Primary Ar flow	Secondary gas flow	Chamber pressure	Powder carrier (Ar)	Powder feed
Howmet	EPI/03CA-111-9	20 cm	1500 A	118 L/min	15 L/min (He)	50 torr	7.5 L/min	5 g/min
CPPM	EPI/03CA-93-9	15 cm	1100 A	66 L/min	28 L/min (He)	200 torr	9.5 L/min	20 g/min
CPPM	EPI/03CA-93-9	15 cm	1100 A	90 L/min	6 L/min (H ₂)	200 torr	9.5 L/min	20 g/min
CTSR	Sulzer/F4	25 cm	680 A	80 L/min	40 L/min (He)	115 torr	3.5 L/min	5 g/min

flowability with a closely controlled size distribution. Initial work established that near-ideal spherical powders of the Cr-modified Ti trialuminide with the desired cubic L1₂ structure could be produced by high-pressure gas atomization.^[12] The original batch sizes of the order of 1 kg gave good yields of particles in the range 40 to 100 μm. Subsequent pilot production batches yielded powders in the 20 to 45 μm size range. The continued development of gas-atomized powders has been shifted to the commercial sector, where production of the trialuminide has commenced with batch sizes up to 250 kg. The inherent stability of the trialuminide intermetallic combined with post-atomization surface pacification procedures (proprietary) results in easy to handle powders exhibiting low flammability. The flowability of these powders is excellent and does not change significantly during exposure to laboratory or spray shop atmospheres. Currently, the Cr-modified ternary trialuminide is available in powder form (Praxair Surface Technologies, Indianapolis, IN, (PST), type AL153) in several size ranges. Further development of Cr-modified quaternary trialuminide alloy powders has also been accomplished.

3. Thermal Spray Coatings

In early efforts, good quality LPPS coatings of the ternary alloy Al₆₆Cr₉Ti₂₅ were produced for Waubik at three different laboratories: Howmet Corporation-Operhall Research Center (Whitehall, MI), the Center for Plasma Processing of Materials (CPPM) at Drexel University (Philadelphia, PA), and the Center for Thermal Spray Research (CTSR) at State University of New York-Stony Brook (Stony Brook, NY). Table 1 provides a list of the basic VPS conditions used at each laboratory. This work established clearly that adherent, reasonably dense, coatings could be applied to a variety of substrates with no obvious

sensitivity to minor changes in deposition conditions and parameters.

The initial coatings produced at Howmet^[4] were applied to flat plate substrates of Ti-6Al-4V and Ti-47Al-2Cr-2Nb, evaluated by inspection of metallographic sections, and then subjected to aggressive cyclic oxidation tests up to 700 and 900 °C, respectively. The test procedure was based on a 1 h cycle, consisting of 55 min immersion in a tube furnace at temperature, followed by removal for a 5 min cooldown to room temperature. This cycle was continuously repeated 200 or more times using an automated apparatus. All of these LPPS coatings, which were at least 200 μm thick, generally protected the substrates while suffering no visible changes other than the formation of a protective layer of alumina at the surface. An example of these initial coatings is shown in Fig. 1, which compares the microstructure of as-sprayed and thermal-cycled AL153 coatings on Ti-47Al-2Cr-2Nb. A light etch revealed the residual dendritic structures of unmelted and partially melted thermal spray powders that were retained in the as-sprayed coating. During cyclic oxidation tests, these structures are homogenized and Al diffuses into the substrate, converting the α₂ + γ phase of the TiAl alloy into Al-rich γ at the coating interface.

The protection provided by these early coatings was not absolute, because a network of stress relief cracks formed in the LPPS coatings applied to Ti and TiAl alloys either during deposition or during the first several thermal test cycles. Interestingly, these coefficient of thermal expansion (CTE)-induced cracks were unaffected by further cyclic oxidation testing. No activity occurred at the crack tips other than the formation of an alumina-rich mixture of oxides. In all cases, there was no cracking parallel to, or phase formation at, the coating/substrate interface, either as-deposited or after the oxidation testing. Thus, a general level of protection was maintained. This is shown in Fig. 2, which is a photograph of a pair of Ti-47Al-Cr-2Nb test coupons

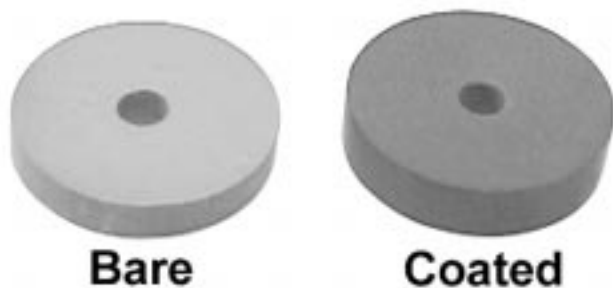


Fig. 2 Ti-47Al-2Cr-2Nb alloy disk specimens, 16 mm diameter \times 4 mm thick, after cyclic oxidation testing for 250 h at 815 °C. Specimen on right was air plasma sprayed with a 25/75% Al_2O_3 /AL153 blended composite coating prior to thermal testing

after cyclic testing. The coupon on the right has been protected by the cubic-trialuminide coating, which remains medium gray in appearance, whereas the bare surface of the left coupon has formed a yellow oxide layer. Additional work at Drexel and the CTSR-Stony Brook concentrated on reducing the as-deposited residual stress in the coatings and minimizing the effects of CTE-induced stresses. Unfortunately, the incidence of the craze cracking with the LPPS coatings could be reduced, but not entirely eliminated.

Subsequent efforts by Waubik working with the CTSR-Stony Brook demonstrated that excellent quality coatings could be deposited with APS and that $<1\%$ oxide formation, all in the form of Al_2O_3 , occurred during spraying. These coatings were found to be more resistant to CTE-induced cracking on Ti and TiAl substrates, but still developed cracks after 25 or more thermal cycles. The APS coatings applied to steels, however, exhibited excellent compatibility and offered impressive levels of oxidation protection. An example of an APS AL153 coating applied to plain carbon structural steel is shown in Fig. 3 after being subjected to cyclic oxidation testing at 800 °C for 237 h. In this case, the APS coating was applied to one surface of the coupon and the remaining surfaces were left bare. The AL153 trialuminide coating provided complete protection of the underlying steel, while more than 1.0 mm of the uncoated surfaces was consumed by oxide growth. Figure 3 also shows that trialuminide coatings applied to Fe (or Ni) alloys are completely free of stress relief or CTE-induced cracks both before and after cyclic thermal testing. Typical parameters for depositing these APS coatings are listed in Table 2.

Further development of trialuminide coatings for Ti and TiAl substrates has shown that stress relief cracking can be eliminated with the application of gradient coatings formed by incorporating varying amounts of substrate powder in the first spray layers. A simple solution was also obtained by admixing ~ 25 vol.% alumina with the alloy feedstock to make a co-deposited composite coating. These coatings are highly adherent and show no microstructural instability during cyclic high-temperature testing.

Recently, high-velocity oxy-fuel (HVOF) spraying has been used to fabricate $(\text{Al,Cr})_3\text{Ti}$ coatings from type AL153 powder. Both stoichiometric and fuel-rich combustion conditions listed in Table 3 were used with a Praxair HV2000 spray system. The stoichiometric conditions were found to produce the best coat-

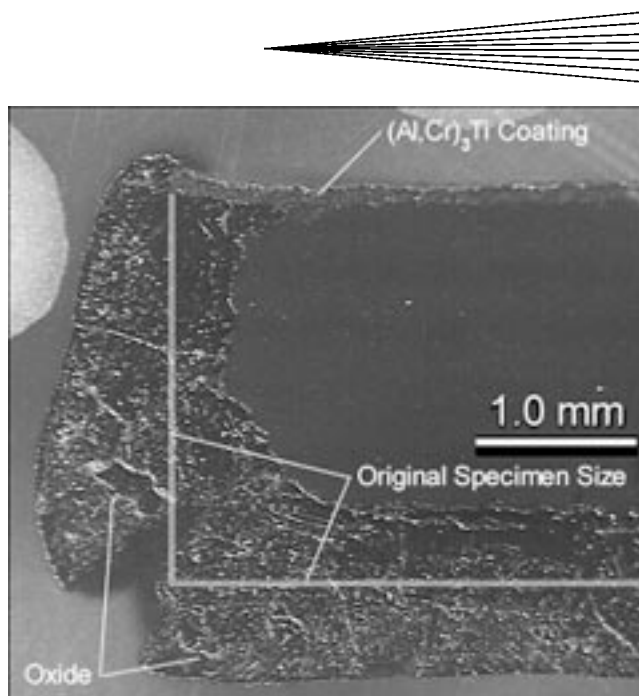


Fig. 3 Effects of cyclic oxidation testing at 800 °C for 237 h on AL153 APS coating applied to one surface of a 1008 steel coupon

ings. Figure 4 is a section micrograph of an as-deposited AL153 HVOF spray coating. The formation of oxides, such as Al_2O_3 , during HVOF spraying was evaluated by measuring x-ray diffraction peak intensities and was found to be equal or less than that of APS coatings.

The results of cyclic oxidation weight gain measurements for surface-finished specimens of APS-coated and uncoated Ti-47Al-2Cr-2Nb are shown in Fig. 5. These measurements were made on special disk-shaped specimens that were finish ground with 600 grit SiC paper, thereby removing approximately one-half of the ~ 200 μm thick coatings and leaving comparably smooth surfaces on uncoated specimens. During the oxidation testing, a significant amount of alumina was rapidly formed on the surfaces and interconnected void area of the coated specimens, causing an appreciable initial weight gain compared to the uncoated specimen. However, beyond ~ 200 h, the coated specimens were quite stable, showing a very small rate of weight gain compared to that of the uncoated specimen, which formed nonprotective TiO_2 along with Al_2O_3 at these temperatures.^[13]

High-temperature high-cycle fatigue tests on coated Ti-47Al-2Cr-2Nb and Ti-6242 cylindrical bars have shown no significant effects of the coatings on fatigue properties; in fact, the properties of the Ti-6242 were actually improved^[14] Figure 6 and 7 show results for axial loaded fatigue tests with $R = 0.1$ on coated and uncoated Ti-47Al-2Cr-2Nb at both 760 °C and 815 °C.

Finally, the cubic-trialuminide coatings have been considered for use as protective coatings on Al alloys, where they provide wear and corrosion resistance. Figure 8 shows the appearance of an AL153 coating applied by APS to an aluminum alloy (type 319) after heating the coated specimen at 450 °C for 1320 h in air. As can be seen in the inset enlargement, the coating/substrate interface has remained stable and unchanged by the thermal treatment.



Fig. 4 AL153 coating applied to 1008 steel with the HVOF method

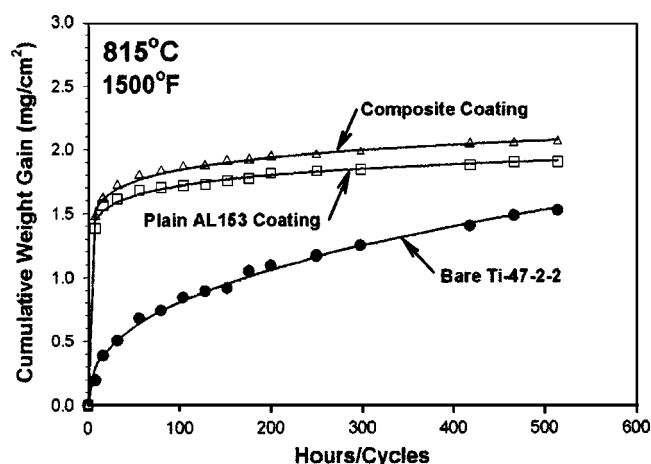


Fig. 5 Weight gain during cyclic oxidation at 815 °C for Ti-47Al-2Cr-2Nb coated with the ternary trialuminide (AL153) and a blended composite formed by co-deposition of 25/75% $\text{Al}_2\text{O}_3/\text{AL153}$, as compared to the uncoated substrate

4. New Composite Forms of the Trialuminide Coatings

Early attempts to synthesize the ternary trialuminides with a refined microstructure by mechanical alloying established a means for introducing large volume fractions of finely dispersed nanoscale (10 to 40 nm) carbides in the alloy. While the ternary alloy itself has reasonable wear resistance, similar to that of γ -TiAl, this finding has permitted the development of new composite forms of the coatings with excellent wear/abrasion resistance, to augment the oxidation resistance.

Specifically, milling the ternary trialuminide in the presence of a carbonaceous processing agent leads to the formation of composite powder particles having microstructures with extremely fine titanium-based carbides in the intermetallic matrix. The powders formed by the milling are excellent feedstock for producing thermally sprayed coatings with the same composite microstructure. Figure 9 and 10 show a composite coating with 12 vol.% carbide applied with APS to Ti-47Al-2Cr-2Nb and mild steel. No special procedures were required for depositing these composite coatings; the thermal spray parameters were the same as those listed in Table 2 for coatings produced with the AL153 powder.

In order to evaluate the wear/abrasion properties of the composite coatings, dry sand rubber wheel (DSRW) tests were carried out on composite coatings with 12 and 25 vol.% carbides applied to several substrates. For comparison purposes, a

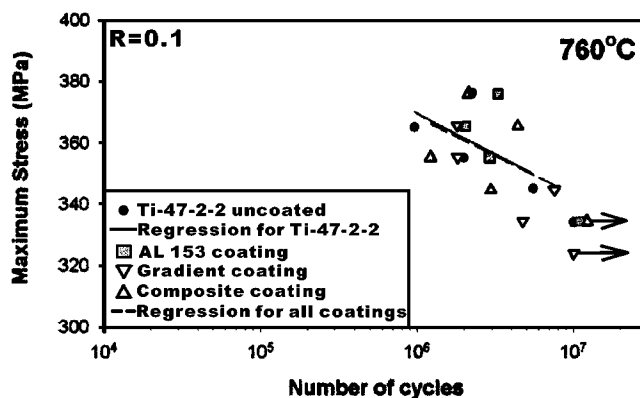


Fig. 6 High-cycle fatigue S-N response for coated and uncoated Ti-47Al-2Cr-2Nb at 760 °C

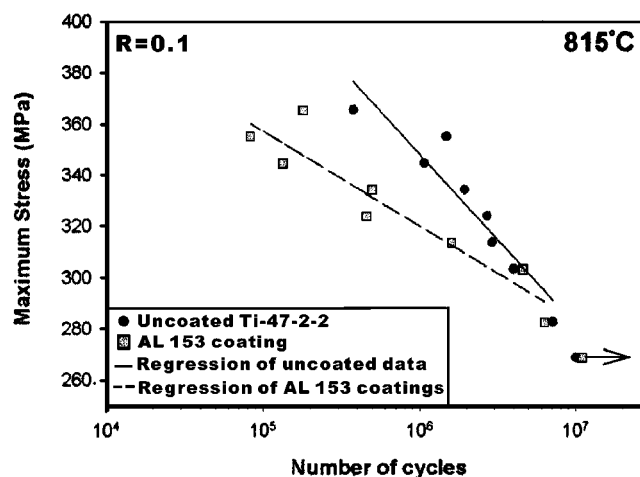


Fig. 7 High-cycle fatigue S-N response for coated and uncoated Ti-47Al-2Cr-2Nb at 815 °C

blended coating of TiC particles admixed with AL153 and a commercial wear-resistant coating (CRC415 from PST, which has Cr_3C_2 in a Ni-Cr matrix) were applied to the same substrates.^[15] The DSRW results from a series of standard tests on these coating systems are summarized in Fig. 11. The effectiveness of the new composite coating, which has 25% carbide content, in resisting abrasion is near that of the CRC415, which contains 35 vol.% carbides. The advantage of the ultrafine microstructures of the new composite coatings is also illustrated in Fig. 12, which shows the volume loss rate derived from extended DSRW tests as a function of the amount of carbide. It is clear that the new nanoscale composite coating outperforms the blended composite AL153/TiC and the CRC415 coatings, both of which have a much coarser carbide particle structure.

The sliding wear performance of ternary trialuminide and carbide composite coatings was evaluated through ball on disk testing. A variety of coatings were applied to TiAl, Ti-6242, Al-6061, and 1008 steel substrates using APS. These included the ternary AL153, an AL153/TiC 75/25% co-deposited powder blend, two trialuminide composite coatings with 12 and 25 vol.% carbide, and the commercial CRC415 coating from PST

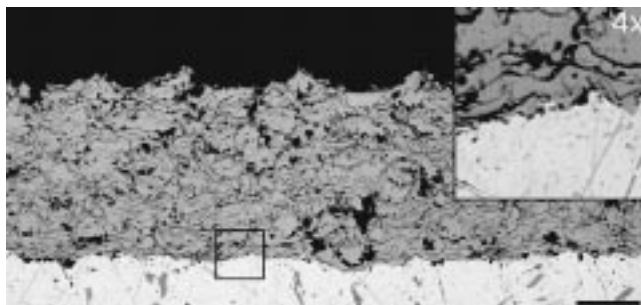


Fig. 8 Ternary trialuminide APS coating on type 319 aluminum alloy after heating at 450 °C for 1320 h

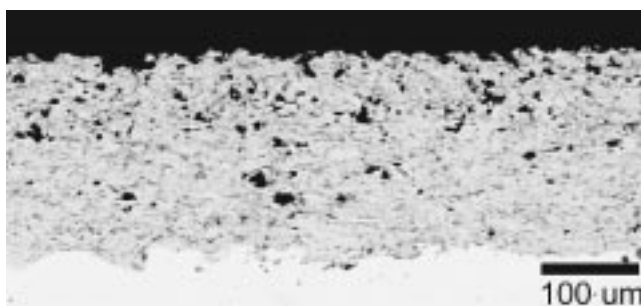


Fig. 9 Composite ternary trialuminide coating with 12 vol.% carbides applied to Ti-47Al-2Cr-2Nb

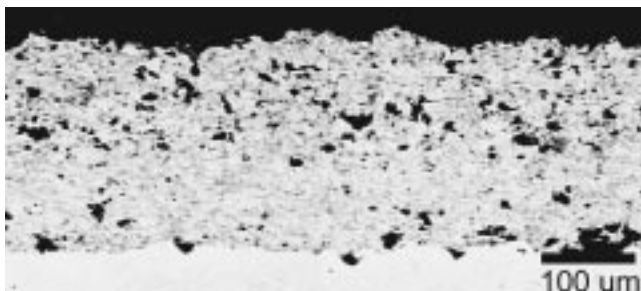


Fig. 10 Composite ternary trialuminide coating with 12 vol.% carbides applied to 1008 steel

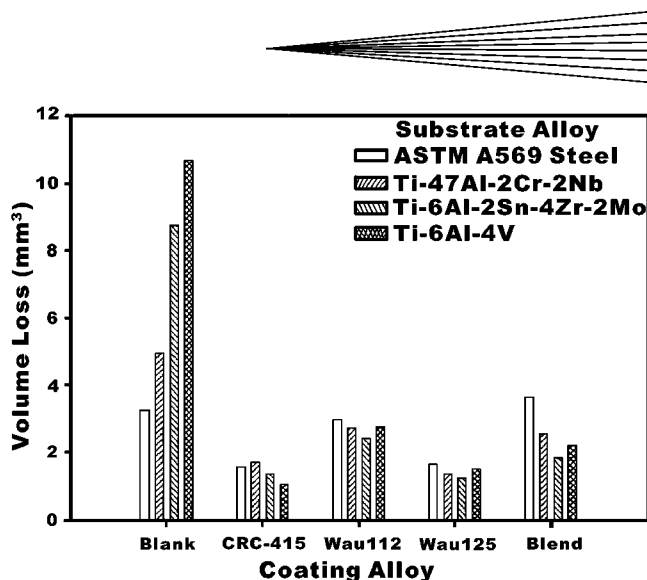


Fig. 11 Volume loss for coatings and substrates tested by DSRW abrasion according to ASTM G65 procedure C. Each bar represents the mean of two tests on a particular material

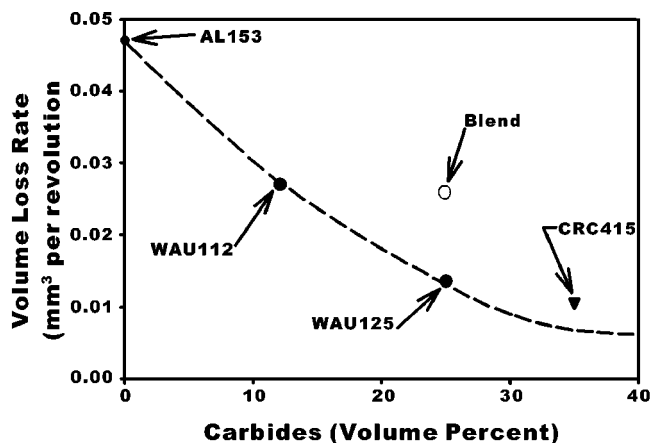


Fig. 12 Abrasive wear rates vs carbide content for the new nanoscale composite coatings (WAU112 and WAU125) compared to a blended AL153/TiC coating and a commercial wear resistant coating (CRC415) with chromium carbides in a NiCr matrix

Table 2 APS parameters for deposition of AL 153 powders and related (Al,Cr)₃ Ti powders and powder blends at CTSR-Stony Brook

Plasma spray system/gun	Spray distance	Gun voltage	Gun current	Primary Ar flow	Secondary gas flow	Powder carrier gas	Powder feed
Sulzer/F4	10 cm	68 V	750 A	40 L/min	40 L/min (He)	6.0 L/min (He)	16 g/min
Sulzer/F4	10 cm	68 V	500 A	50 L/min	11 L/min (H ₂)	3.5 L/min (Ar)	5 g/min

Table 3 HVOF thermal spray conditions used for the deposition of AL 153 powder with the Praxair HV-2000 system

Spray condition	Stoichiometric	Fuel-rich
Fuel gas (C ₃ H ₆)	110 SLPM	125 SLPM
Oxygen	360 SLPM	360 SLPM
Carrier gas (Ar)	70 SLPM	70 SLPM
Powder feed	25 g/min	25 g/min

containing 35 vol.% carbide. All of these coatings were applied to 75 × 75 mm substrate plates to a thickness of 200 μm and then ground to a 600 grit finish using SiC papers. Ten millimeter diameter sapphire and 440C stainless steel balls were used as the wear instruments. The balls were held in contact with the coated plates with a force of 9.8 N (1 kg mass), and the plates were affixed to a turntable in order to inscribe a 50 mm diameter wear groove. The rotation speed corresponded to a contact velocity of

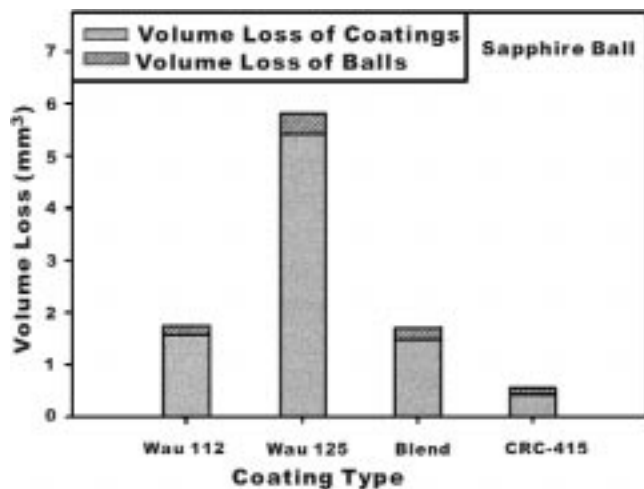


Fig. 13 Volume loss of wear-resistant coatings and sapphire balls during unlubricated sliding wear (ball on disk) tests at room temperature for a 1 km sliding distance

1.23 m·s⁻¹, with the test being carried out for a total lineal distance of 1000 m. All tests were performed in laboratory air with no lubrication. The volume losses from wear surfaces were calculated from start and end weight measurements and confirmed by optical profilometry of the wear groove.

The test results are summarized in Fig. 13 and 14 for contact with the sapphire and stainless steel balls, respectively. The stacked graphs indicate the overall wear of both the coating and the ball for each test configuration and are averages of volume loss measurements for coatings applied to different substrate materials and for measurements on corresponding balls. In the case of wear against sapphire, the 12 vol.% composite coating gave similar performance to the blended carbide coating, but both were not quite to the level of wear resistance of the CRC415 coating. The 25 vol.% composite coatings, WAU125, suffered greater material loss in these tests, most likely as a result of greater frictional force between the two materials. The results of wear against 440C stainless steel were markedly different. The 25 vol.% carbide coatings gave outstanding performance, with some tests showing almost no wear, and in some instances, the coating gained material at the expense of the ball. In this case, the blended carbide coatings performed similarly to the CRC415, but the 12 vol.% composite coatings exhibited slightly greater overall material loss. It is expected that higher density coatings, such as those deposited by HVOF, will offer even more effective sliding wear performance.

5. Summary

Thermal spray coatings of chromium-modified cubic titanium trialuminides have been shown to provide excellent oxidation protection for a variety of substrates, including alloys based on Ti and Fe. These new ternary trialuminide coating alloys are strong alumina formers, showing outstanding oxidation resistance up to 1200 °C and having coefficients of thermal expansion that are compatible with many commercially important engineering materials substrates. Aggressive cyclic oxidation

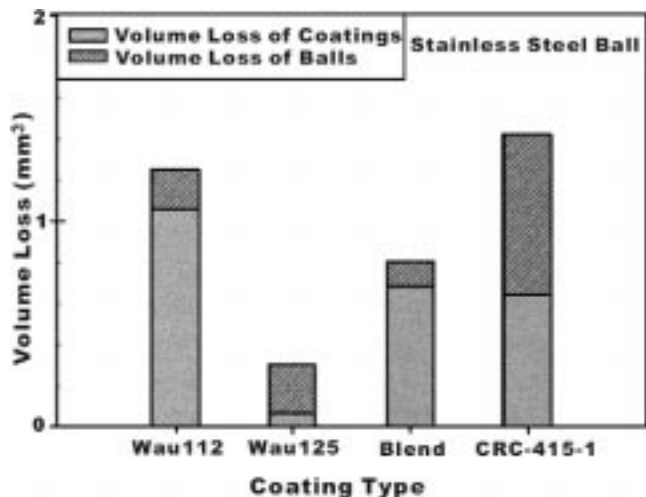


Fig. 14 Volume loss of wear-resistant coatings and stainless steel balls during unlubricated sliding wear (ball on disk) tests at room temperature for a 1 km sliding distance

testing of thermal spray coatings has demonstrated that the trialuminide coatings are highly adherent and microstructurally stable, with no spallation or coating/substrate interface degradation. Currently, it appears that these coatings can provide environmental protection sufficient to raise the service temperatures of the substrates in air by 150 °C, or more.

Thermal spray feedstock powders of the ternary alloy with desired particle sizes and size distributions are produced by conventional gas atomization methods and do not require special handling. A distinct commercial advantage of the coating alloys is their low density, which provides for about twice the coverage per unit mass available with most other coating alloys, such as the MCrAlY types.

Although these ternary trialuminide alloys have good mechanical strength with reasonable resistance to abrasion and wear, the recent development of ultrafine composite intermetallic/carbide microstructures has made it possible to access truly unique coating properties. In particular, a means for introducing any desired volume fraction of extremely fine (10 to 40 nm) carbides into the intermetallic powder particles, a microstructure that can be preserved in as-sprayed coatings, yields a family of wear-resistant coatings that have good oxidation resistance. Currently, the relationship of processing conditions to the nature of the carbides and their formation mechanism is being studied, as are possible enhancements in microstructure and properties achievable through quaternary alloying of the intermetallic matrix.

Acknowledgments

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