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Multi-scale tribological and nanomechanical behavior of cold sprayed $\rm Ti_2AlC$ MAX phase coating



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ABSTRACT

Ti₂AlC based MAX phase coatings were successfully deposited on Inconel 625 substrate by a cold spraying technique. A dense coating of 70 μ m thickness was deposited. Ball-on-disk wear behavior of Ti₂AlC coating at room temperature (25 °C), and high temperature (600 °C) were studied. The coefficient of friction (COF) and wear volume loss at 600 °C reduced by ~ 21% and ~ 40% respectively, due to the lubricious nature of oxide layer formed at a higher temperature. Mechanical properties of the Ti₂AlC coating were also studied by carrying out nanoindentation and nano-scratch tests at room temperature and 300 °C and varying loads. For a low load of 7000 μ N at room temperature, Ti₂AlC coating exhibited a higher elastic modulus of 273 GPa compared to the elastic modulus of 191 GPa at high temperature (300 °C). The room temperature nano-scratch at 7000 μ N displayed brittle behavior with fracture, chipping and wear debris formation along the scratch path. However, high temperature (300 °C) scratch path exhibited ductile nature with plowing, cutting and no wear debris formation. The wear volume loss was several orders of magnitude higher at 8 N load scratch. The overall wear behavior in MAX phase Ti₂AlC coating at multiple load scales is elucidated in terms of the interaction volume varying from a single to several splats in the cold sprayed structure.

1. Introduction

Since the discovery of MAX phase based ternary carbides, Ti₂AlC has emerged as one of the potential materials with superior oxidation resistance for high-temperature applications. Ti₂AlC has low density (4.11 g/cm³), low coefficient of friction, good mechanical, and electrical properties [1-5]. MAX phases are layered, hexagonal carbides and nitrides with the general formula: $M_{n+1}AX_n$, exhibiting both ceramic and metallic properties where (M) stands for a transition metal, (A) is an element from groups 13–16, and (X) is carbon or nitrogen [6]. This group of material exhibit various metallic properties such as thermal and electrical conductivity, resistance to thermal shock, plasticity at elevated temperatures, and good machinability. The ceramic properties include high-temperature stability, excellent oxidation resistance, low density and corrosion resistance [2,7-10]. Synthesis of MAX phase has been tried by various methods to form bulk samples, thin films, and coatings [11-25]. Bulk consolidation has been performed using sintering methods such as hot pressing [11], hot isostatic pressing [12], microwave sintering [13] and spark plasma sintering [14]. Thin film coatings of 500-800 nm thickness have been deposited

by physical vapor deposition (PVD) techniques, primarily magnetron sputtering [16] and pulsed cathodic arc [17]. Thin film coatings of MAX phase are promising for small area applications such as sliding electrical contacts, sensors, anode materials for batteries/fuel cells and thin damping coatings in MEMS [18]. Most recent application discovered for MAX phase materials was the ohmic contacts for high temperature power electronics [26].

Currently, the superalloys used in most jet engines can operate at a temperature up to 1000 °C. The MAX phase coating's thermal barrier properties have the potential to increase this operating temperature. However, the deposition of large scale MAX phases coatings has proven challenging. A few attempts have been made to deposit thicker MAX coatings > 50 µm using High-Velocity Oxy-Fuel spraying (HVOF) [19], plasma spraying [20–22] and cold spraying [23–25]. When producing thicker coatings, high deposition temperatures are usually required. This can result in the occurrence of oxidation and phase transformations. The high deposition temperatures of thermally sprayed processes also introduce residual stresses in the coatings that limit the thickness that can be obtained. Frodelius et al., deposited a Ti₂AlC coating of 50–125 µm thickness by HVOF [19]. However the high deposition

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Table 1

Experimental parameters of quasi-static nanoindentation and nano-scratch tests for the cold sprayed Ti₂AlC coating.

Type of test	Conditions	Load/type of loading	Temperature (°C)	Scratch length (µm)
Quasi-static nanoindentation	Low load	7000 μN	25	Not applicable
		7000 µN	300	Not applicable
	High Load	5 N	25	Not applicable
		8 N	25	Not applicable
Scratch	Low load	Ramp (7000 μN)	25	10
	(across one or two splats)	Constant (7000 µN)	25	10
		Ramp (7000 μN)	300	10
		Constant (7000 µN)	300	10
	High load	Ramp (8 N)	25	200
	(Across several splats)	Constant (8 N)	25	200

temperature resulted in the formation of TiC and Ti_xAl_y throughout the coating [19]. The presence of these phases hindered the formation of the protective layer of aluminum oxide. Therefore oxidation was observed throughout the coating. Similarly, using plasma spray, Ti₃SiC₂ MAX phase coating of $\sim 200 \,\mu m$ thickness was formed by reaction synthesis of Ti + SiC + C powder mixture [20]. However, intermediate phases (TiC_X, Ti₅Si₃) and TiO were formed along with the major MAX phase. The cold spraying has emerged as a potential technique to alleviate some of these challenges associated with oxidation of MAX phases [27]. Very little investigation has been performed on cold spray deposition of Ti₂AlC. Preliminary studies have shown that thick MAX phase coating can be cold sprayed while retaining the feedstock compositions of the powder [23-25]. Gutzmann et al. successfully cold sprayed MAX phase Ti₂AlC particles without any phase transformation and obtained a coating thickness of 110 to 155 µm. The deformation of the Ti₂AlC particles and splats were found to be enhanced by thermal softening under higher impact and substrate temperature [23]. Rech et al., also fabricated the MAX phase Ti₂AlC coating by cold spray deposition on both soft AA6060 aluminum alloy and hard steel substrates [24]. Dense coatings were achieved by this low-temperature process without any structural change or oxidation of the coating. Maier et al., using cold spray process, deposited Ti₂AlC coating on Zircaloy-4 fuel cladding to improve its oxidation resistance [25]. The Ti₂AlC coated substrate displayed about ~340% increase in microhardness compared to Zircaloy-4 substrate. The wear track depth decreased from $12 \,\mu m$ to 1 µm after Ti₂AlC coating deposition, suggested enhanced wear resistance. Excellent oxidation resistance of Ti₂AlC coating in air at 700 °C and steam at 1005 °C was also observed [25]. These studies indicate cold spray process as a promising technique to obtain a uniform, thick and dense MAX phase Ti₂AlC coating without any phase transformation or oxidation.

In the present study, multiscale tribological and nanomechanical properties of cold sprayed Ti₂AlC coatings on Inconel substrate have been investigated. Inconel, which is a nickel-chromium based superalloy, was chosen as the substrate material to closely resemble the superalloys used in the making of jet engine turbine blades. The purpose of this study is to understand the tribological properties of the cold sprayed Ti₂AlC coating at multiscale loading conditions and room temperature and high temperature conditions, which has never been reported earlier.

2. Experimental details

MAX phase Ti₂AlC coating was deposited on grit blasted Inconel 625 substrate by cold spray deposition technique at a commercial cold spray facility. The cold spray parameters were similar to deposition of Titanium based alloys using high pressure cold spray technique. The exact cold spray processing parameters were not provided by the commercial vendor due to proprietary reason. Maxthal 211 (Ti₂AlC) powder feedstock was procured from Sandvik Materials Technology, Sweden. No bond layer was used between the substrate and Ti₂AlC

coating. The structural characterization of the as-received powders and the cold sprayed coating was carried out using an X-ray diffractometer (XRD, Siemens D5000) with Cu Ka ($\lambda = 1.542$ Å) radiation and lattice parameters were calculated using non-linear least-squares fitting with pseudo-Voigt function. Microstructural characterization of the as-received powders, coating, and fractured surface was examined using JEOL JSM-6330F field emission scanning electron microscope (SEM).

Tribological properties of the polished coating surface, both at room temperature (25 °C) and high temperature (600 °C), were evaluated using a ball-on-disk tribometer (Nanovea, Irvine, CA, USA) in dry air, using alumina ball of 3 mm diameter as the counter material. The wear tests were carried out at a normal load of 5 N and a speed of 100 RPM for 30 min making a wear track of 4 mm diameter. The wear tracks



Fig. 1. XRD patterns of the (a) as-received $\mathrm{Ti}_2\mathrm{AlC}$ powder and (b) cold sprayed $\mathrm{Ti}_2\mathrm{AlC}$ coating.

The large amorphous hump in the background was due to the sample holder.

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