



Kerosene-fuelled high velocity oxy-fuel (HVOF) spray of Ti₂AlC MAX phase powders



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ABSTRACT

Kerosene-fueled high velocity oxy-fuel (HVOF) spray was employed to deposit commercial Ti₂AlC MAX phase powders onto Inconel 600 substrates. After optimizing three process parameters including flow rates of kerosene and oxygen as well as spray distance, a dense Ti₂AlC coating with a very low decomposition was obtained. Using Rietveld phase analysis of the X-ray diffraction pattern, the weight percentage of Ti₂AlC in the coating was determined to be 53.0%, which is not only far better than the coating deposited by plasma spray, but also is comparable with the optimum Ti₂AlC coatings sprayed by H₂-fueled HVOF and cold spray. This optimized Ti₂AlC coating was further analyzed by a variety of techniques to probe its surface chemistry, morphology, cross-sectional microstructure, mechanical properties and salt fog corrosion resistance.

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1. Introduction

Ti₂AlC belongs to a family of ternary carbides and/or nitrides called M_{n+1}AX_n (n = 1, 2 or 3) or MAX phase materials, where M represents a transition metal (Sc, Ti, Zr, Hf, V, Nb, Ta, Cr, Mo, etc), A corresponds to a group 12–16 element (Cd, Al, Ga, In, Tl, Si, Ge, Sn, Pb, P, As, etc), and X is carbon and/or nitrogen, respectively. New MAX phase materials have been synthesized from time to time and they now consist of 70 plus members. MAX phase materials possess a common layered hexagonal structure (space group P6₃/mmc), which is constructed by vertically repeated two M_{n+1}X_n layers intercalated by one atomic A layer in between [1,2].

Ti₂AlC is one of the first few MAX phase materials which have been synthesized with high purity. Bulk polycrystalline Ti₂AlC was first fabricated in 1997 using reactive hot isostatic pressing of Ti, Al₄C₃ and graphite powders at 1600 °C for 4 h under a pressure of 40 MPa [3]. Through detailed investigation of Ti₂AlC and other structurally related MAX phase materials (i.e., Ti₂AlN, Ti₃AlC₂,

Ti₃SiC₂, Ti₄AlN₃, etc), their uniqueness in possessing both metallic (good electrical and thermal conductivity, high ductility, easy machinability, superior thermal shock resistance and damage tolerance) and ceramic (high melting point and high temperature oxidation resistance) properties has been unveiled to the public [1,2,4,5]. As a result, interests on MAX phase materials have surged since mid-1990s. The distinctive combination of these properties is attributed to the co-existence of the weak metallic M-A bonds (which account for the metallic properties) and the strong covalent-ionic M-X bonds (which account for the ceramic properties) [1,2]. More recently, it has been discovered that the weak metallic M-A bonds can be exploited to etch A layers selectively away by acid (e.g., HF), leaving behind layered M_{n+1}X_n structures (such as Ti₃C₂, Ti₂C, Ta₄C₃, Ti₃CN_x, etc [6]) with -O, -OH, and -F terminations to replace the A layers (such as Al, Si and Ga). This new family of 2D materials are called MXene in order to emphasize their relationship with the MAX phases and to draw parallel with graphene [7]. MXene has been exploited in energy storage devices such as batteries and electrochemical capacitors due to their intrinsically high specific surface areas [7], which in turn promotes another application of MAX phase materials as the source materials

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for synthesizing MXene.

As a representative MAX phase material, Ti_2AlC has been explored for its application in the fields including high temperature protective coatings [8] and radiation-tolerant cladding material for the nuclear power plants [9,10]. In such applications, coatings thicker than 100 μm are usually required. Although the magnetron sputtering [11–14] or pulsed cathodic arc technique [15] are able to produce polycrystalline [13,14] or even single crystalline Ti_2AlC thin films [11,12,15], they would not be the feasible or cost-effective techniques to fabricate coatings thicker than 30 μm up to several millimeters, which are usually required to restore the original dimensions of worn parts, or to impart the underlying parts the coatings' superior properties (e.g. thermal barrier coatings). Thick films can be deposited using chemical/electrochemical plating, brazing, weld overlays and thermal spray. Recently, the availability of Ti_2AlC MAX powders, which also contains Ti_3AlC_2 with minor amount of TiC [10,16–23], enables thermal spray as a feasible option to deposit such thick coatings [10,16–21,24,25]. Thermal spraying techniques are frequently used for coating process, in which coating materials in a powder or wire form are heated or even melted by the thermal energy through electrical (plasma or arc) or chemical (combustion flame) means and are subsequently accelerated and sprayed onto a surface at high velocity [26]. There have been attempts using three different thermal spray techniques including cold spray [10,21,27], high velocity oxy-fuel (HVOF) spray [16–20] and plasma spray [25,28] to deposit Ti_2AlC MAX powders.

Among the three thermal spray techniques, cold spray technique utilizes the least amount of thermal energy (100–1000 $^{\circ}C$) to soften the Ti_2AlC powders but the highest kinetic energy to accelerate the powders beyond a critical velocity in a supersonic gas jet (500–1000 m/s) towards the substrates. It has produced quite dense and adherent Ti_2AlC coatings with very minimum phase decomposition and oxidation [10,21,27]. However, occurrence of cracks in such coatings is detrimental for reliable coatings [21,27], while the coating thickness was limited to 50–80 μm when the process gas (N_2) temperature was 800 $^{\circ}C$ or lower [27]. Ti_2AlC coatings with a thickness of 110–155 μm can only be achieved when the N_2 temperature was raised to 1000 $^{\circ}C$ [21]. On the other hand, plasma spray utilizes the highest amount of thermal energy (12000–16000 $^{\circ}C$ [29]) to melt the Ti_2AlC powders but the lowest kinetic energy to accelerate the powders towards the substrates. As a result, the Ti_2AlC powders can be heavily decomposed using the inappropriate process parameters [25]. Similarly, Ti_3SiC_2 MAX phase powders were nearly decomposed during plasma spray [28]. However, careful optimization of the process parameters can significantly reduce the degree of decomposition and enable deposition of any desired thickness of the Ti_2AlC coating using plasma spray [25].

Compared with cold spray and plasma spray, high velocity oxy-fuel (HVOF) spray employs a median heat energy (2100–3000 $^{\circ}C$ [18]) and median kinetic energy (400–800 m/s) to produce the coatings. In HVOF spraying, heat is produced by burning oxygen with either gas-fuel (i.e., hydrogen, ethane, propylene, propane or acetylene) or liquid-fuel (i.e., Jet-A or kerosene). The powders are fed into the path of the combusted fuel exiting through the converging/diverging nozzle of the gun at near supersonic speeds, flatten plastically upon impact with the substrate, cool and solidify to form the coating. During gas-fuelled (i.e., H_2 , ethane, etc) HVOF spray process, the temperature in the center of flame (2100–3000 $^{\circ}C$ [18]) is significantly higher than the decomposition temperature of bulk Ti_2AlC (1400 $^{\circ}C$ [20]). The short dwell time (in a few millisecond) in the flame still leads to the softening and partial melting of Ti_2AlC MAX phase powders by the thermal energy. The resulting coatings were dense and had good adhesion to the stainless steel substrates, but Ti_2AlC in the coatings partially

decomposed into a mixture of TiC , Ti_3AlC_2 and Al_xTi_y phases [16,18]. The efforts to mitigate the impact of thermal energy on the phase decomposition include to increase powder size and to decrease gas ($H_2 + O_2$) flow rate, but thinner coatings with lower cohesion were produced [18]. Annealing such HVOF-sprayed Ti_2AlC coatings around 850 $^{\circ}C$ in vacuum with a base pressure of 10^{-3} Pa helped intercalate Al into the under-stoichiometric TiC and hence facilitated partial restoration of Ti_2AlC wt% in the coating [19].

Liquid-fuelled HVOF spray system is also capable of depositing thick coatings using median thermal energy. However, compared with gas-fuelled HVOF spray, it offers additional advantages of generating more momentum output to powder particles and lowering down the production cost due to usage of low-cost liquid fuel such as kerosene instead of costly H_2 gas. Such benefits render liquid-fuelled HVOF an economic viable technology for depositing thick coatings in an industry (rather than lab) scale. However, there has been no reports so far in employing kerosene-fuelled HVOF to spray the Ti_2AlC MAX phase powders and to compare the coating properties with those by gas-fueled HVOF. Such information would be critical in verifying the feasibility of adopting liquid-fuelled HVOF to spray Ti_2AlC MAX phase material and other temperature sensitive materials. Therefore, in this work, kerosene-fuelled HVOF was explored to spray the Ti_2AlC powders. Three key process parameters including flow rates of kerosene and oxygen, as well as spray distance were optimized through X-ray diffraction (XRD) and Rietveld phase analysis of the coatings. The optimized coating was further evaluated by X-ray photoelectron spectroscopy (XPS), scanning electron microscopy (SEM), optical microscopy, macro-hardness tester, pulling test, three-point bending test, wear test and salt spray test for its surface chemistry, morphology, cross-sectional microstructure, macro-hardness, bond strength, adherence, wear resistance and salt fog resistance, respectively.

2. Material and methods

A customized large powder size (45–100 μm) of Ti_2AlC MAX phase powders (Maxthal 211[®]) was produced in Sandvik AB Sweden by crushing and sifting bulk Ti_2AlC after combustion synthesis. Large powders help minimize the oxidation and decomposition drawbacks the small powders encounter when they were propelled through the hot flame during the spray. As the Ti_2AlC powders are not produced by gas atomization which is commonly used for other commercial powders, Ti_2AlC powders are not spherical but irregular in shape [23]. Substrates are Inconel 600 coupons (AMS-5540N, $0.1 \times 4.45 \times 7.62$ cm³), which were cleaned using acetone and sand blasted using Al_2O_3 grit 60 at a pressure of $1.7\text{--}2.1 \times 10^5$ Pa prior to the spray. The substrates were mounted vertically on a carousel with support from the back to prevent coupon distortion by the high speed flame. There was no coupon rotation during spray. The HVOF spray was performed using a Praxair JP-5000[®] HVOF system. Powders were dried in an oven at 60 $^{\circ}C$ for a minimum of 2 h, and fed at the center of a JP5220 gun with 15.2 cm (or 6") barrel by a GTV PF2/1 powder feeder (Fig. 1(a)). During the spray, the gun was held horizontally and traversed back and forth across sample surface with an index spacing of 3 mm. Coupons were pre-heated by one pass prior to spray. The movement of the spray gun in one pass is illustrated in Fig. 1(b). Parameters including kerosene flow rate, O_2 flow rate and spray distance were systematically tuned.

The crystalline structures of the resulting coatings were analyzed using Bruker D8 Advance XRD operated at a voltage of 40 kV and a current of 40 mA (Cu $K\alpha$ X-ray, $\lambda = 1.54$ Å). The different phases' relative weight percentages were subsequently determined by Rietveld full-pattern phase analysis using TOPAS software after considering the peak positions and their weight percentages. The

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