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Microstructure evolution of V–Al–C coatings synthesized from a V_2AlC compound target after vacuum annealing treatment

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ABSTRACT

V₂AlC belongs to a family of ternary nano-laminate alloys known as the MAX phases, which exhibit a unique combination of metallic and ceramic properties. In this work V–Al–C coatings with deposited (V,Al)₂C nano-crystallines and amorphous phase were magnetron sputtered from V₂AlC compound target. The subsequent vacuum annealing for 1 h was carried out at 600 °C, 700 °C and 800 °C, respectively. The crystallization of V₂AlC MAX phase was detected by annealing at 600 °C. Meanwhile, a small amount of amorphous carbon phase appeared. Further increasing annealing temperature to 700 °C led to a complete transformation from amorphous V–Al–C phase to V₂AlC phase, as well as a significant increase in the amorphous carbon content. It was noticed that the crystallinity of V₂AlC phase was significantly enhanced and amorphous carbon was almost disappeared after annealing at 800 °C. The coating toughness became better with the increase in the content of V₂AlC MAX phase. The optimized mechanical and tribological properties of the annealed V–Al–C coatings were further discussed in terms of the microstructure evolution.

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1. Introduction

V₂AlC (space group P6₃/*mmc*) belongs to a family of the socalled $M_{n+1}AX_n$ phases, where M is a transition metal, A is an A group element, X is either C or N, and n = 1-3. A distinctive feature of these phases is their layered structure with alternating $M_{n+1}X_n$ and A layer, the carbon atoms occupying the octahedral voids formed by M atoms, without contact with atoms A. MAX phases are well known for their high stiffness, good mechanical stability as well as chemical and thermal resistance, which are typical properties of ceramic materials. At the same time, they exhibit good electrical and thermal conductivity, as well as facile machinability, almost in the range of metallic materials [1]. Due to their unique structure and interesting performances, MAX phases have been fabricated in form of both bulk materials [2] and in coatings [3].

Compared to the bulk forms, recently, MAX phase coating synthesized by physical vapor deposition (PVD), in particular, have drawn much attentions due to its promising feasibility in the used substrates and deposited large area for wide range of applications. Variety of MAX coatings including Ti₂AlC, Cr₂AlC, Ti₂AlN and Ti₃SiC₂, have been achieved by PVD techniques, where the deposition process, mechanical properties and oxidation resistance of coatings are investigated systematically. Nevertheless, few studies about the V-containing 211 phase V₂AlC coating are reported. Vbased coatings exhibit lower friction coefficient compared to that of Ti or Cr-based coatings [4], V₂AlC MAX phase coatings is considered as a strong candidate for supertough wear-resistant application. Until now, the V₂AlC coatings have been synthesized using magnetron sputtering techniques either from elemental targets or from compound targets at relatively high substrate temperature or alternated by the post annealing. Growth of V₂AlC coatings was reported in 2006 by Schneider et al. [5] utilizing sputtering from three elemental targets at 850 °C substrate temperature. Sigumonroung et al. [6] believed that the threshold temperature range for the formation of phase pure V₂AlC using vapor condensation without ion assistance lied between 650 and 750 °C. Jiang et al. [7] studied the effects of post-annealing temperature (550-850 °C) on the thermal stability and the phases formation of the vanadium







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carbide solid solution $(V,Al)_2C_x$ coatings. They found that the formation of V₂AlC MAX phase could be observed at 650 °C and phasepure V₂AlC emerged at 850 °C. The crystallization kinetics of the deposited X-ray amorphous V₂AlC coatings was investigated by Abdulkadhim et al. [8], where the crystalline V₂AlC phase formed between 565 and 675 °C. Obviously, the structure of V–Al–C coating phase are significantly dependent upon the deposition and annealing temperature, it is crucial to clarify the correlation between the microstructure evolution and physichemcial properties of coating during the formation for its expected applications.

Moreover, the advantages of Ti alloys over other metallic materials lie in their good combination of mechanical and chemical properties, such as high stiffness, low density, good corrosion resistance. Ti-6Al-4V (TC4) is being a family member of titanium alloy, which is one of the most promising alternative lightweight heat resistant alloys. In addition, TC4 can be easily welded, forged and machined [9,10], which makes it more wide applications. Here, the V-Al-C coatings were deposited on TC4 substrate by DC magnetron sputtering using a powder sintered composite target with a pure V₂AlC phase in an industrial deposition chamber, which was followed by a thermal annealing in vacuum from 600 to 800 °C. The aim of this work is to examine the role of deposition and postannealing process on the structure, mechanical and tribological properties of V-Al-C coating. The results showed that the higher content of the formed V₂AlC MAX phase, the better toughness of coating was. The special layered structure of MAX phase emerged in the coating after thermal annealing was considered as the key role for the controllable mechanical and tribological properties.

2. Experimental

V-Al-C coatings were deposited by a hybrid ion beam deposition system [11] consisting of a circular DC magnetron sputtering and a linear anode-layer ion sources (LIS). The samples of TC4 alloy with the size of 15 mm \times 10 mm \times 2 mm were chosen as the substrate in the present work. The nominal composition (wt.%) of the alloy is given as follows: Ti, bal; Al, 6; V, 4; Fe, 0.25; O, 0.15; C, 0.1; N, 0.04; H, 0.015. After being polished to 3000-grit, the substrates were ultrasonically cleaned in acetone and ethanol each for 15 min followed by a dried process in warm air. Then, the substrates were fixed on a rotary sample holder at a distance of 15 cm to the target. The base pressure was pumped less than 3×10^{-3} Pa. Prior to deposition, the substrates were sputter-cleaned for 15 min using Ar ions generated by LIS to remove the contaminants of surface. The magnetron cathode with a diameter of 80 mm was fulfilled by a compound target composing the single-phase V₂AlC, which was homemade by spark plasma sintering of elemental V, Al, and C powders with a molar ratio of 2:1.2:0.9 at 1500 °C for 30 min. The applied power density of sputtering target was 14 W/cm². During deposition the argon pressure was maintained at 0.77 Pa. The negative bias voltage of -150 V, with a frequency and duration of 350 kHz and 1.1 ms, was applied to the substrates without other intentionally heated. After 8 h deposition, the thickness of the coating was about 4 µm. Then, annealing treatment of the asdeposited coating was conducted at 600 °C, 700 °C and 800 °C in vacuum for 1 h. Before the annealing process, the chamber was pump down to 1.0×10^{-2} Pa and heated to the annealing temperature with a heating rate of about 4 °C/min.

The microstructures of the coating were investigated by the combined x-ray diffraction (XRD), scanning electron microscopy (SEM), and transmission electron microscopy (TEM). Structural analysis was carried out using x-ray diffraction (XRD), with a BrukerD8 Advance diffractometer, using Cu K α radiation. The detected phase content was calculated on the basis of XRD pattern refinement [12]. The surface secondary electrons morphology was

examined by a Hitachi S4800 high resolution SEM. The crosssection backscattered electrons morphology was examined by a FEI Quanta FEG 250 SEM, equipped with an energy-dispersive X-ray spectrometry (EDX) using an EDAX Sapphire Si (Li) detector. Quantification was performed using the standard ZAF method, which incorporated individual corrections from the atomic number (Z), absorption (A), and fluorescence (F). Cross-sectional TEM (XTEM) studies were performed on a IEOL 2100-HR system. Raman spectroscopy (InVia-reflex, Renishaw) equipped with a 60 mW He-Ne laser of 532 nm exciting wavelength was used to evaluate the carbon atomic bond structure in the coatings. Mechanical properties were measured by a MTS Nanoindenter G200 tool in a continuous stiffness measurement mode using a Berkovich diamond tip. Hardness and Young's modulus were determined using the Oliver-Pharr analysis [13]. The characteristic hardness was chosen in a depth of around 1/10 of the coating thickness, where the measured value was not affected by the substrate. Six replicate indentations were done for each sample.

Tribological behavior was evaluated by reciprocating ball-onplate dry sliding tests against Al_2O_3 balls with a diameter of 6 mm on a UMT-3 tribometer (CETR, USA). The sliding speed and frequency were set at 0.01 m/s and 1 Hz, respectively. All tests were conducted under a load of 10 N at the ambient temperature (~293 K) and a relative humidity of 50–60%. The total sliding time was 3600 s. After tests, the wear track profiles were measured by the surface profilometer. The wear rates were evaluated as volume per sliding distance per load [14].

3. Results and discussion

Fig. 1 shows the XRD patterns of the pristine V–Al–C coatings and the annealed ones at 600 °C, 700 °C and 800 °C. A broad diffraction peak appeared at about 43° for the pristine coatings and no obvious crystalline peaks can be found within the XRD resolution limits, indicating that the pristine V–Al–C coatings were amorphous or some small nano-crystalline. This result was consistent with that obtained by Li [15] and Abdulkadhim et al. [8], where the amorphous Cr–Al–C and V–Al–C coatings were reported by sputtering the Cr₂AlC and V₂AlC composite target at the room temperature, respectively. A further careful examination was conducted by HRTEM in this study later. However, if one considers the MAX phases belong to the high-order material systems, in





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