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Facile fabrication of high-purity Cr_2AlC MAX-phase coating via pulsed laser annealing

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ABSTRACT

MAX phase coatings exhibit considerable potential for applications in nuclear systems and electrochemical energy fields. However, synthesis of these coatings strongly relies on magnetron sputtering deposition at high temperatures, which limit the usage of temperature-sensitive substrates. Here, we report a facile strategy with pulsed laser annealing to replace conventional vacuum annealing for fabrication of high-purity Cr_2AlC coatings in air. Specifically, using a nanosecond pulsed laser, amorphous Cr-Al-C coatings were transferred into high-purity crystalline Cr_2AlC without inducing cracks simultaneously. The ultrafast heating and cooling features enabled the precise modulation of grain size by adjusting scanning passes. This non-vacuum, easy technique overcomes the temperature constraints of traditional synthesis methods, significantly extending the applicability of MAX phase coatings to low-melting-point materials.

1. Introduction

MAX phase coatings with a layered hexagonal close-packed structure, also known as $M_{n+1}AX_n$ (n = 1–3), have attracted increasing interests for applications in extremely harsh conditions owing to their unique combination of metallic and ceramic properties [1,2]. Generally, M stands for an early transition metal, A is mainly from group A, and X is assigned to C, N, or B. Benefiting from the unique nanolaminate structure with strongly controlled covalent, ionic and metallic bonds, Cr_2AlC, a particular MAX phase, exhibits superior physiochemical properties, such as high-temperature stability, excellent thermal conductivity, good anti-corrosion and irradiation resistance. These merits make Cr_2AlC coating highly promising for applications in harsh nuclear systems and electrochemical related energy materials [3,4].

Magnetron sputtering is one of the most common methods for fabricating Cr_2AlC coating, which is typically realized via either a one-step deposition [5,6] or a two-step deposition-annealing process [7,8]. In one-step approach, in-situ substrate heating during sputtering is required to directly obtain crystalline Cr_2AlC coating, usually resulting in a columnar grain structure. Alternatively, the two-step route involves the deposition of an amorphous Cr-Al-C precursor at low temperature, followed by a vacuum post-annealing to produce crystallization with an equiaxed grain. Despite the continuous progress, both approaches remain mainly constrained by high thermal inputs. Even with advanced sputtering configurations with high impulse power supply, the lowest reported substrate temperature for one-step synthesis of crystalline Cr_2AlC is close to 480 °C [5], while two-step route identified post-annealing temperature at least ~500 °C to achieve crystalline high-purity Cr_2AlC coating [9,10]. Although the grain size can be effectively refined by lower annealing temperature from 700 °C to 500 °C [11], the synthesis temperatures required for Cr_2AlC formation still exceed ~450 °C, thereby inducing difficulties for thermal sensitive substrates, such as aluminum and copper.

Lasers offer exceptionally high energy density and ultrafast heating rates, favoring them attractive for localized thermal processing [12,13]. According to the Beer-Lambert law [14], optical energy absorption decays with depth, creating a steep thermal gradient, enabling rapid surface heating while maintaining a comparatively low substrate temperature. This suggests that laser annealing of amorphous Cr-Al-C coating could promote crystallization into Cr_2AlC while keeping the substrate cool, avoiding thermal damage. Furthermore, laser annealing is highly efficient and can be performed under ambient conditions, eliminating necessary vacuum environments. By tailoring laser wavelength, power and pulse width, the crystallization depth can be precisely controlled [15,16], offering opportunities to achieve gradient grain structures in MAX phase coatings through multiple laser treatments.

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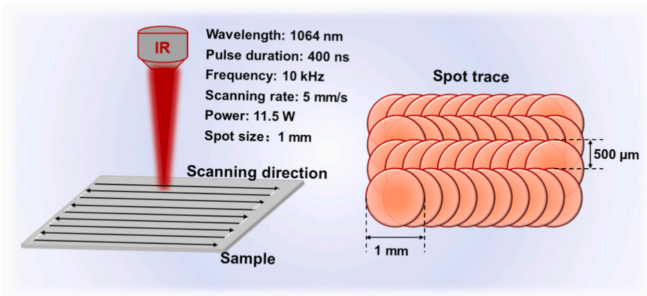

Fig. 1. Schematic diagram of laser annealing process.

In this work, we employ a 1064 nm nanosecond pulsed laser to irradiate amorphous Cr-Al-C coatings deposited using a hybrid cathodic arc and magnetron sputtering system. High-purity Cr_2AlC coatings with tunable grain sizes were successfully fabricated, and the microstructural evolutions were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), and transmission electron microscopy (TEM). This laser-based annealing strategy provides a versatile, low-thermal-input route for integrating MAX phase coatings with temperature-sensitive substrates.

2. Materials and methods

Amorphous Cr-Al-C coatings were deposited on TC4 substrates using a hybrid deposition system combining cathodic arc and magnetron sputtering sources. The detailed deposition parameters are reported elsewhere [10]. The as-deposited amorphous coatings, with a thickness of approximately 7.8 μm, were subsequently annealed in ambient air using a pulsed infrared laser (JPT YDFLP-CL-500-50-W). The laser annealing parameters are summarized in Fig. 1. To tailor the grain size of the fabricated Cr_2AlC coating, multiple laser scanning passes with an overlap ratio of 50% were introduced. Specifically, samples treated with one scanning pass and five passes are denoted as S1 and S2, respectively.

Phase composition of the coatings was identified by XRD (Bruker). Surface morphology and elemental distribution were characterized by SEM equipped with energy-dispersive X-ray spectroscopy (EDS). Cross-sectional microstructures were examined by TEM, with lamellae prepared using focused ion beam milling. The mechanical properties were measured using an MTS-G200 nano-indenter in continuous stiffness measurement mode.

3. Results and discussion

Fig. 2a presents the XRD patterns of the coatings before and after laser annealing. The as-deposited Cr-Al-C coating exhibits a typical broad amorphous diffraction peak, indicative of a disordered structure. However, after laser irradiation, distinct diffraction peaks corresponding to the Cr_2AlC phase emerge. Compared with S1, sample S2 exhibits sharper and more intense peaks, demonstrating the enhanced crystallinity and grain growth with increasing laser scanning passes. Fig. 2b~2d displays the surface morphologies of the as-deposited and laser-annealed coatings. No significant differences in surface topography are observed after laser treatment. Notably, the absence of solid ripple features on the irradiated surfaces suggests that laser annealing did not induce melting, indicating a solid-state crystallization process from the amorphous precursor. Furthermore, despite the ultrafast heating and cooling cycles, no surface cracks are detected. The inset EDS analysis reveals an increased oxygen content from 1.39% to 5.94%, accompanied by a slight increase in the Al/Cr atomic ratio after laser annealing, illustrating the preferential surface oxidation of Al. The oxide layer is confined to the surface, with a thickness of approximately 30 nm (Fig. 3). The formation of such Al-rich surface oxides has been reported to be beneficial for enhancing the corrosion resistance of Cr_2AlC coating [17]. No obvious changes in coating thickness were observed after laser irradiation.

In order to clearly identify the crystallinity in the coating induced by laser annealing, Fig. 3 shows the cross-sectional bright-field TEM

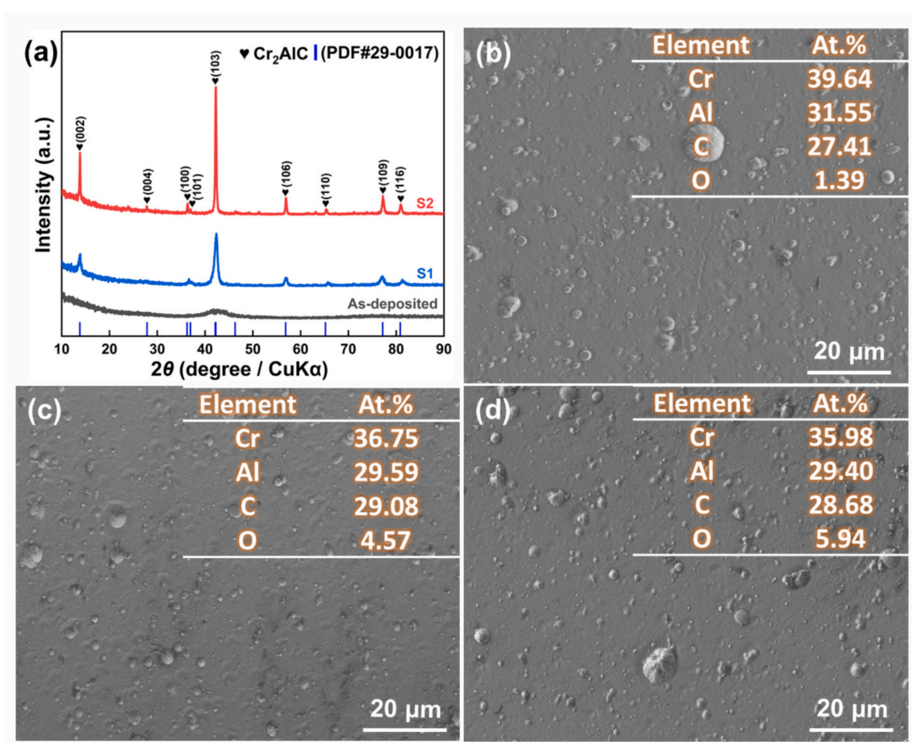

Fig. 2. Phase composition surface SEM micrographs of the Cr-Al-C coatings before and after laser annealing. (a) XRD patterns, SEM micrographs of as-deposited coating (b), S1 sample (c), and S2 sample(d).

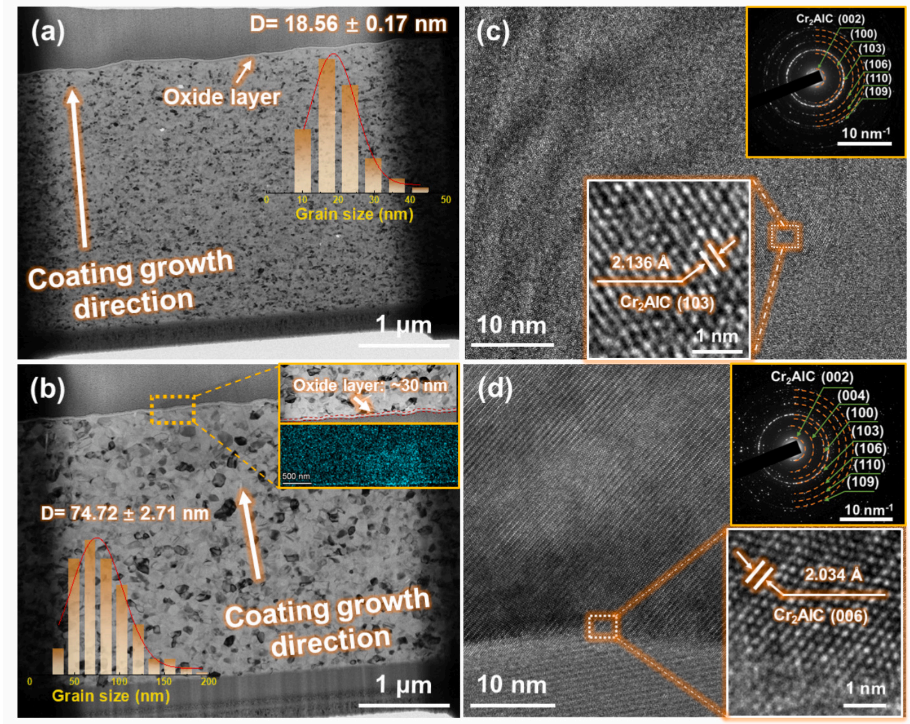

Fig. 3. Cross-sectional TEM microstructures of laser-annealed Cr-Al-C coatings. Bright-field TEM microstructures of S1(a) and S2 (b), HRTEM microstructures with inserted SAED patterns of S1 (c) and S2 (d).

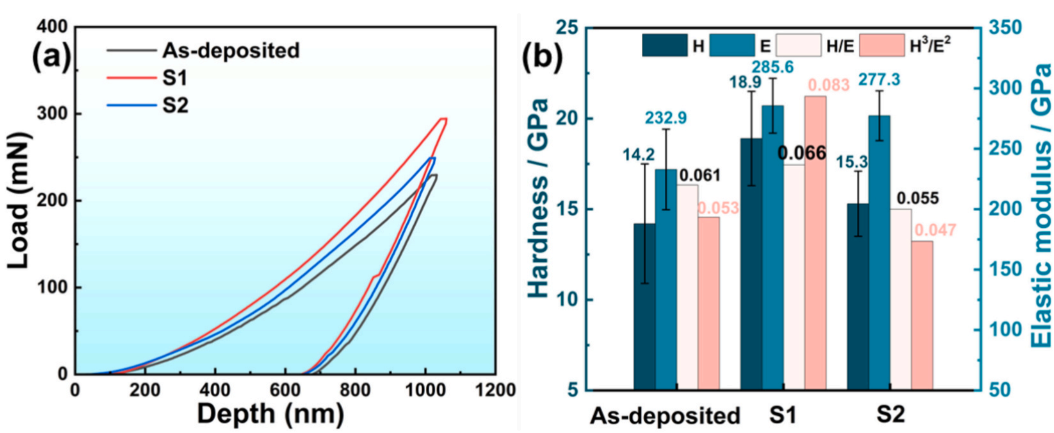

Fig. 4. Nano-indentation tests of Cr–Al–C coatings. (a) Load-depth curves, (b) hardness, elastic modulus, *H/E* and H^3/E^2.

microstructures and corresponding selected-area electron diffraction (SAED) patterns of the laser-annealed coatings. Both S1 and S2 samples exhibit a typical equiaxed grain structure, as shown in Fig. 3a and b, which are consistent well with the typical characteristic of Cr_2AlC coatings produced by conventional vacuum annealing [10]. Notably, no continuous oxide layer was observed within the coatings. Statistical analysis of TEM images yields the average grain sizes 18.56 ± 0.17 nm for samples S1 and 74.72 ± 2.71 nm for S2. High-resolution TEM (HRTEM) images and the inserted SAED patterns (**Fig. 3c** and **d**) display the spotty-discontinuous diffraction rings, evidencing the nanocrystalline feature of the coatings. The measured interplanar spacings of 2.136 Å and 2.034 Å are in good agreement with the (103) and (006) crystallographic planes of Cr_2AlC phase, respectively.

Fig. 4 presents the mechanical properties of the three coatings. It can be observed that the crystalline Cr_2AlC phase formed after laser annealing exhibits significantly higher hardness (*H*) and elastic modulus (*E*) than the as-deposited amorphous Cr-Al-C coating. Benefiting from the fine-grain strengthening effect [18], the S1 sample with the finer grain size demonstrates the best overall mechanical performance (H: 18.92 ± 2.63 GPa, E: 285.6 ± 22.7 GPa, H/E: 0.066, H^3/E^2: 0.083). Notably, the Cr_2AlC coatings fabricated using this method exhibited superior mechanical properties to the annealed coatings [11,19].

Previous studies have shown that laser annealing can markedly modify surface residual stress states [20,21]. For example, Wei et al. [20] demonstrated that CO_2 laser annealing transforms surface tensile residual stress into compressive stress in ZrO_2 coatings. Such laser-induced compressive stresses are effective in suppressing crack initiation and propagation under contact loading, thereby enabling improvement of hardness, wear resistance, scratch resistance, and fatigue performance. Therefore, the development of surface compressive residual stress via laser annealing is expected to be advantageous for the service reliability of the present Cr_2AlC coatings. Beyond stress regulation, laser irradiation offers exceptional processing flexibility. By adjusting key parameters, including wavelength, laser power, and scanning speed, the transient temperature gradient within the coating can be precisely modulated. This enables independent control of crystallization depth and grain structure in MAX phase coatings while minimizing thermal impact on the substrate, representing a critical advantage for the fabrication of functionally graded coatings on temperature-sensitive materials.

4. Conclusion

In summary, nanocrystalline high-purity Cr_2AlC coatings with tunable grain sizes were successfully fabricated by irradiating amorphous Cr-Al-C coatings with a 1064 nm pulsed laser under ambient conditions. After laser annealing, the coatings maintained the dense surface morphology without any distinguished cracks or solidification ripple features. Nevertheless, a slight increase in surface oxygen and aluminum content in the coating is expected to improve corrosion resistance. This ultrafast and non-vacuum laser annealing approach provides a versatile and facile strategy to fabricate MAX phase coatings on temperature-sensitive substrates. Future work will explore the precise regulation of gradient grain structures in these laser-annealed coatings to further tailor their functional properties for harsh conditions.

CRediT authorship contribution statement

Jianghuai Yuan: Writing – original draft, Data curation. **Yingjie Wang:** Data curation. **Zhenyu Wang:** Supervision. **Aiying Wang:** Writing – review & editing, Supervision, Funding acquisition. **Lin Li:** Supervision. **Zhu Liu:** Writing – review & editing, Supervision, Funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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