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Process Optimization for the Synthesis of V_2AlC MAX Phase to Enhance Sustainable Production

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In this study, we present a comprehensive optimization study for the synthesis of the V_2AlC MAX phase by systematically investigating the effects of key parameters, including precursor ratios, reaction temperature, reaction time, and heating rate. The resulting V_2AlC MAX phase samples are characterized using X-ray diffraction, scanning electron microscopy, and energy dispersive X-ray spectroscopy analysis to evaluate their phase purity, structure, and morphological features. The optimum temperature for the synthesis of V_2AlC MAX phase is 1500°C with a holding time of 4 h at a heating rate of 5°C to achieve MAX phase with a high quality. The optimized synthesis conditions outlined in this study serve as a fundamental basis for scaling up the yield of the V_2AlC MAX phase in a more sustainable and efficient approach and subsequently enable the wider adoption of V_2AlC MAX phase in various applications, promoting a sustainable future.

Keywords: 2D Materials; MAX phase; V_2AlC MAX phase; ball milling; sustainability

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Nanostructured materials are highly demanded for a variety of applications due to their extraordinary properties [1-5]. Among the various new generation materials, MAX phases have emerged as a distinctive class of materials with a layered structure, combining both metallic and ceramic properties [6-9]. Their exceptional combination of mechanical, electrical, and thermal characteristics makes them highly desirable for various applications across industries [10-13]. Among these MAX phases, V_2AlC has garnered significant attention due to its exceptional mechanical properties, including high hardness, excellent wear resistance, and thermal stability [14]. Additionally, V_2AlC exhibits unique electronic properties, rendering it a promising candidate for advanced electronic devices and energy storage systems. The synthesis of the V_2AlC MAX phase involves a complex reaction between vanadium, aluminum (Al), and carbon precursor elements. Traditional synthesis methods for the V_2AlC MAX phase necessitate high temperatures exceeding 1500°C and extended reaction times, resulting in considerable energy consumption, elevated production costs, and environmental impact [15]. While previous studies have explored various synthesis methods for MAX phases, including powder metallurgy [16], solid-state reactions [17], and chemical vapor deposition [18], there remains a gap in knowledge regarding the fine-tuning and optimization of the synthesis process for V_2AlC MAX phase to achieve sustainable production without compromising material quality [19]. To address these challenges and facilitate the sustainable production of the V_2AlC MAX phase, optimization of the synthesis process becomes imperative [20]. Systematic investigation and optimization of crucial parameters, such as precursor ratios [21], reaction temperature [22], reaction time [23], and heating rate [24], can significantly reduce energy consumption, minimize waste generation, and enhance overall process efficiency.

Therefore, this research work aims to contribute to the scientific understanding of V_2AlC MAX phase synthesis by presenting a comprehensive optimization study. By elucidating the effects of key parameters such as reaction time, reaction temperature, etc. and employing experimental design, this study seeks to achieve optimal synthesizing conditions for production sustainability and efficiency, ensuring the high-quality yield of the V_2AlC MAX phase. We perform the analysis by using scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDX), X-ray diffraction (XRD), and field-emission SEM (FESEM) for the different reaction conditions and show the optimized variables for the synthesis of the quality product for further application. The findings of this research help in the facile synthesis of MAX phases that enables sustainable manufacturing practices thereby facilitating the wider adoption of V_2AlC MAX-phase in diverse applications.

EXPERIMENTAL SECTION

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Chemicals and Materials

Vanadium powder (99.5%, 325 mesh) and Al powder (99.5%, 325 mesh) were procured from Alfa Aesar, USA. Carbon (>99% purity, 200 mesh) was obtained from R&M chemical, UK. All reagents were of analytical grade and used without further purification.

Synthesis Methods of V_2AlC MAX Phase

V_2AlC powders were thermally treated to create the V_2AlC that was employed in this investigation. The synthesis begins by selecting high-purity powders of vanadium, Al, and carbon with uniform particle sizes and composition to ensure the desired MAX phase is formed. In **Figure 1** we present the entire synthesis process. The powders are weighed and thoroughly mixed in the desired stoichiometric ratio. powders were combined in a 2:1.1:1 molar ratio to create V_2AlC. This mixing process ensures uniform distribution of the elements and promotes homogeneity in the final MAX phase [25]. The mixed powders are subjected to a high-energy milling process, such as mechanical milling or ball milling. This step helps to promote the intimate mixing of the elements and reduce particle sizes, facilitating subsequent reaction steps [26].

The consolidated compact is then subjected to an annealing process in a controlled atmosphere, typically in a vacuum or inert gas environment. In a tube furnace, the mixture was heated at different temperatures such as 1300°C, 1400°C, and 1500°C for 5 h under argon environment and the corresponding samples were denoted as Sample A, Sample B and Sample C, respectively. The annealing temperature and duration are carefully controlled to induce the desired reaction between the elements and promote the formation of the MAX phase [27]. The material was crushed and sieved using a 500-mesh sieve, the resultant material was turned into powders with a particle size of less than 25 µm for further processing.

Characterizations

The microstructure and morphology of the V_2AlC MAX phase sample was examined by SEM (Tescan Vega 3, Czech Republic) for the morphological features. The composition and elemental distribution information in the sample was analyzed by EDX (Oxford Instruments EDX). The V_2AlC MAX phase was synthesized using a solid-state reaction method and XRD was employed to determine the phase purity and crystal structure of the MAX phase. The crystal phases of all samples were examined by the D/teX Ultra2 X-ray diffractometer (Rigaku, Japan) with Cu Kα radiation (λ = 0.154 nm) over the range of 2θ = 0° - 90° at a scan speed of 2°/minute under texture mode.

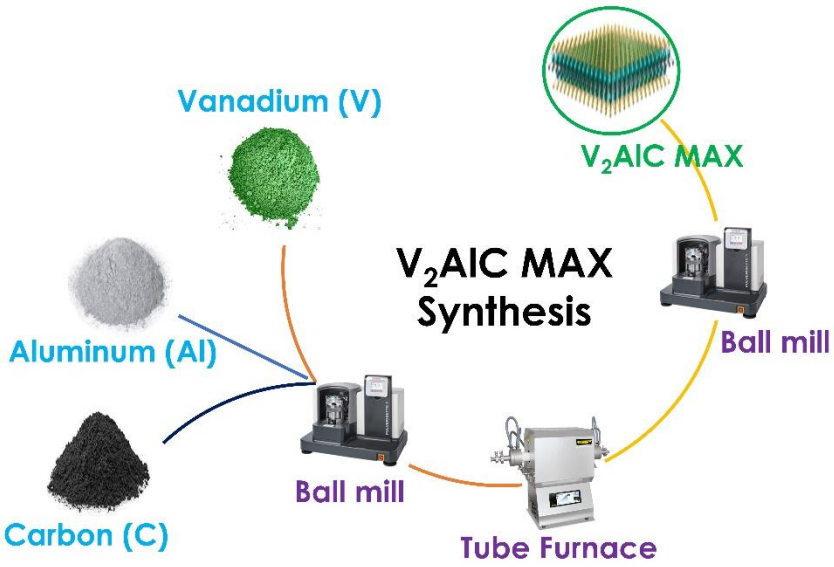

Figure 1. Schematic diagram of synthesis of V_2AlC MAX phase.

RESULTS AND DISCUSSION

SEM is an imaging technique that provides information on the microstructure and morphology of materials [28-34]. **Figure 2a** represents the V_2AlC MAX phase precursors mixture, which includes V, Al, and C for the synthesis V_2AlC MAX Phase. The V_2AlC MAX phase synthesized at different temperatures (1300°C,

1400°C and 1500°C) were examined using SEM and the corresponding images are shown in **Figure 2(b-d)**, respectively. Upon examining the shape of the V_2AlC MAX phase in these samples, it is evident that sample C exhibits a significantly greater quantity of impurities compared to the other samples. This higher impurity content suggests a deviation from the desired V_2AlC MAX phase composition. Conversely, Sample A demonstrates a relatively lower impurity level. However, when comparing the alignment and formation of the MAX phase layers, it is clear that Sample C exhibits the best formation. From a comprehensive perspective, considering both impurity content and the quality of MAX phase layer alignment, Sample C emerges as the superior choice. Therefore, the experimental conditions selected for Sample C is opted as optimal conditions for synthesizing V_2AlC MAX phase. This selection is based on a visual evaluation of the results, indicating that Sample C offers the most favorable characteristics for the desired V_2AlC MAX phase synthesis compared to the other samples.

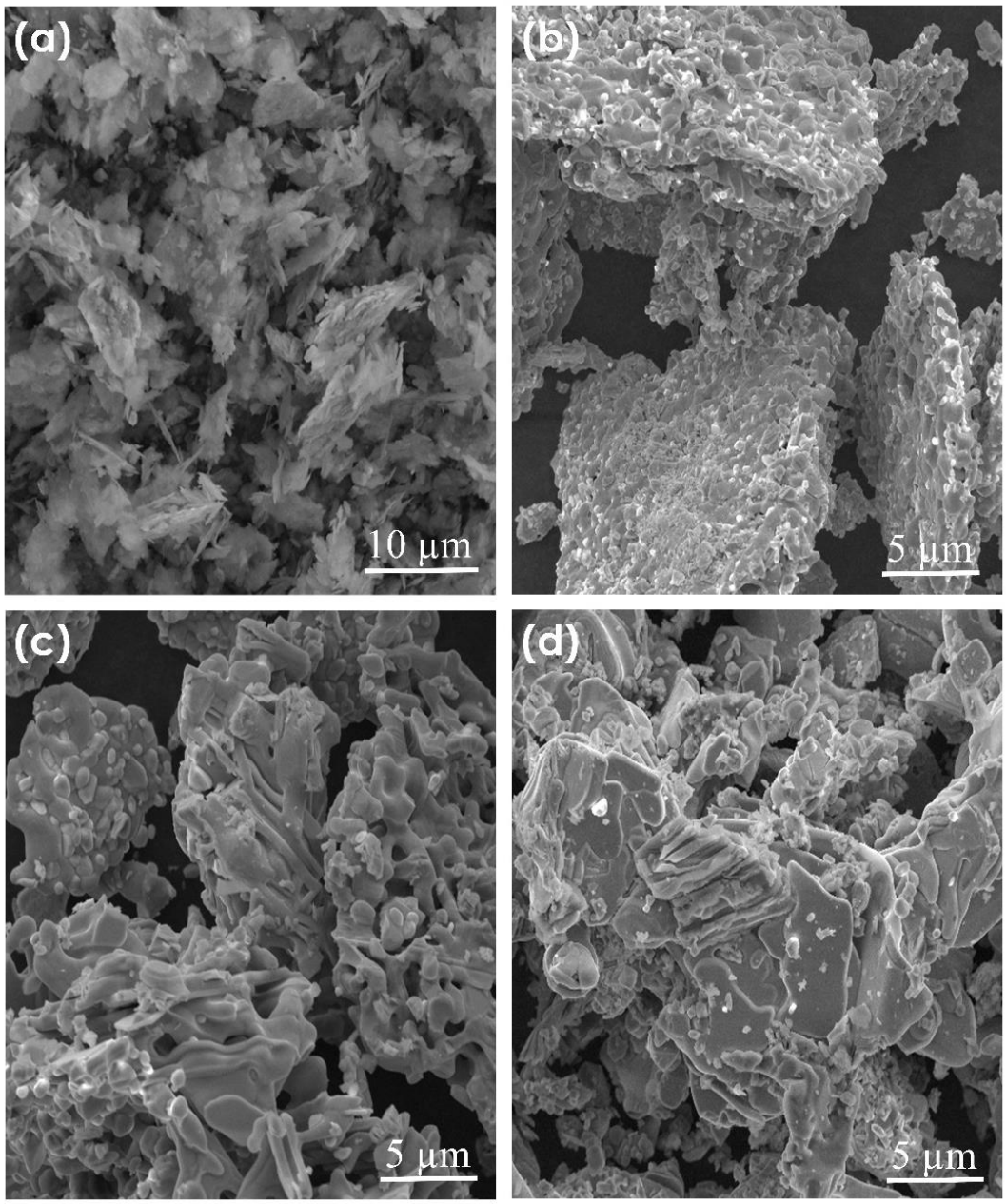

Figure 2. (a) SEM of the V_2AlC MAX phase precursors mixture which includes V, Al, and C and (b-d) SEM of the V_2AlC MAX phase synthesized at different temperatures: 1300°C (b), 1400°C (c) and 1500°C (d).

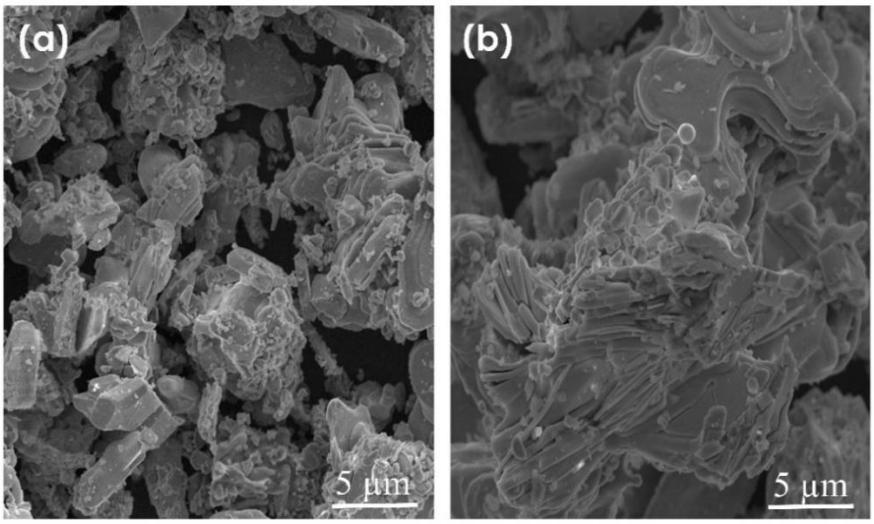

Figure 3. SEM of the V_2AlC MAX phase samples synthesized at different temperature holding time: (a) V_2AlC/1500/2 and (b) V_2AlC/1500/4.

By investigating the SEM images (**Figure 3(a,b)**), it becomes evident that manipulating the holding time at a fixed temperature has a significant impact on the purity of V_2AlC MAX phase. Upon comparing the two images, it is apparent that a holding time of 4 h at 1500°C (as depicted in **Figure 3b**, denoted as V_2AlC/1500/4) results in a more well-aligned layered structure when compared to that of the one kept at a holding time of 2 h at 1500°C (as depicted in **Figure 3a**, denoted as V_2AlC/1500/2). This observed feature signifies an improved crystalline arrangement and overall quality of the material. It demonstrates a reduction in impurities such as VC_x and Al_2O_3 compared to that of the sample kept for 2 h. The decrease in impurities reveals high purity of the as-synthesized V_2AlC MAX phase material. Also, **Figure 3a** exhibits a higher quantity of impurities, and the layers appear more deformed and bowed, indicating a lower degree of structural integrity. Although both **Figure 3a** and **3b** exhibit the presence of V_2AlC MAX phase, the disparities in impurity content and layer morphology confirm the quality of the MAX phase. To gain deeper insights into the elemental composition, EDX analysis is deemed necessary.

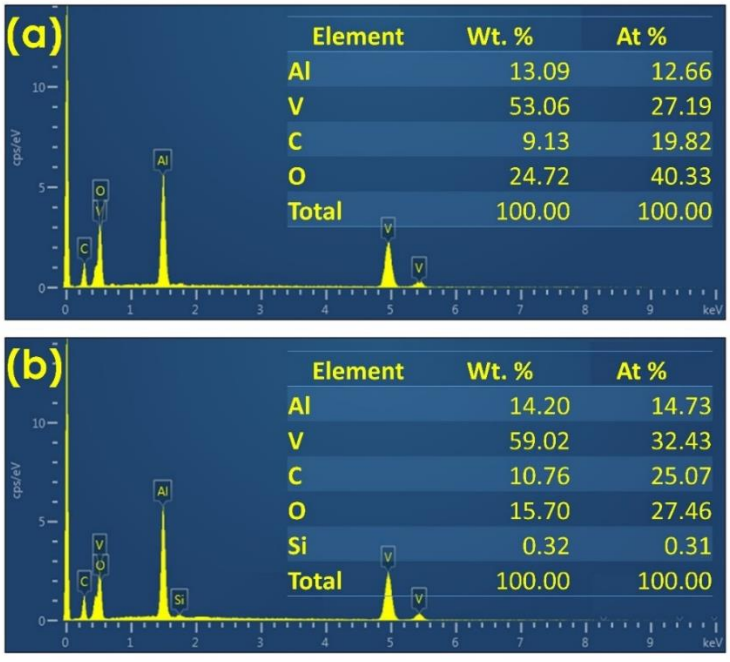

Figure 4. EDX spectra of the V_2AlC MAX phase synthesized at different temperature holding time: (a) V_2AlC/1500/2 and (b) V_2AlC/1500/4.

EDX analysis confirmed the presence of V, Al, and C in the expected stoichiometric ratio. The absence of other elements further confirmed the purity of the synthesized V_2AlC MAX phase. Based on **Figure 4**, it is evident that the purity of the two samples differs significantly. In the case of V_2AlC/1500/4, the composition in terms of wt% of the sample is deemed satisfactory whereas V_2AlC/1500/2 is found to be oxygen rich. However, upon closer inspection of the composition in terms of at%, it becomes apparent that the sample exhibits a decrease in quality due to the presence of oxygen. Furthermore, the carbon content in V_2AlC/1500/2 is slightly lower compared to V_2AlC/1500/4. Also, a small amount of Si is present, which can be considered as an impurity. This suggests that the longer holding time has positively influenced the sample's characteristics. The observations suggest that the holding time and temperature conditions employed in the experiment have significant effects on the purity and composition of the samples. It is evident that the longer holding time in V_2AlC/1500/4 has led to improved results in terms of composition (in terms of both wt% and at%) when compared to V_2AlC/1500/2.

Figure 5 represents the EDX elemental mapping of the V_2AlC MAX phase, V_2AlC/1500/4. From the figure, it is evident that the constituent elements within the material form bonds in a uniform manner, thereby demonstrating a high degree of homogeneity. This homogeneous bonding pattern is indicative of the successful formation of the V_2AlC MAX phase, characterized by the desired chemical composition and crystal structure. The attainment of such a well-defined MAX phase implies that the synthesis process employed was effective in promoting the formation of the desired material.

XRD analysis confirmed the formation of the desired V_2AlC MAX phase, with some diffraction peaks matching the expected crystallographic planes of the MAX phase. In **Figure 6**, the peaks positioned at 2θ = 13.89° (002), 26.09° (004), 41.65° (103), 51.13° (106), 64.79° (110), 74.86° (109), and 79.19° (116) correspond to the crystalline V_2AlC MAX phase, which agrees with JCPDS Card No. 29-0101 [17-21]. With the heating temperature of 1500°C and a holding time of 4 h, the dominant peaks are predominantly related to the crystalline phase of V_2AlC, accompanied by minority peaks of VC_x and V_2C, with weaker intensities. For a holding time of 2 h, other than V_2AlC peaks additional minor peaks like VAl, VAl_3, VC_x, and V_2C, appear as well.

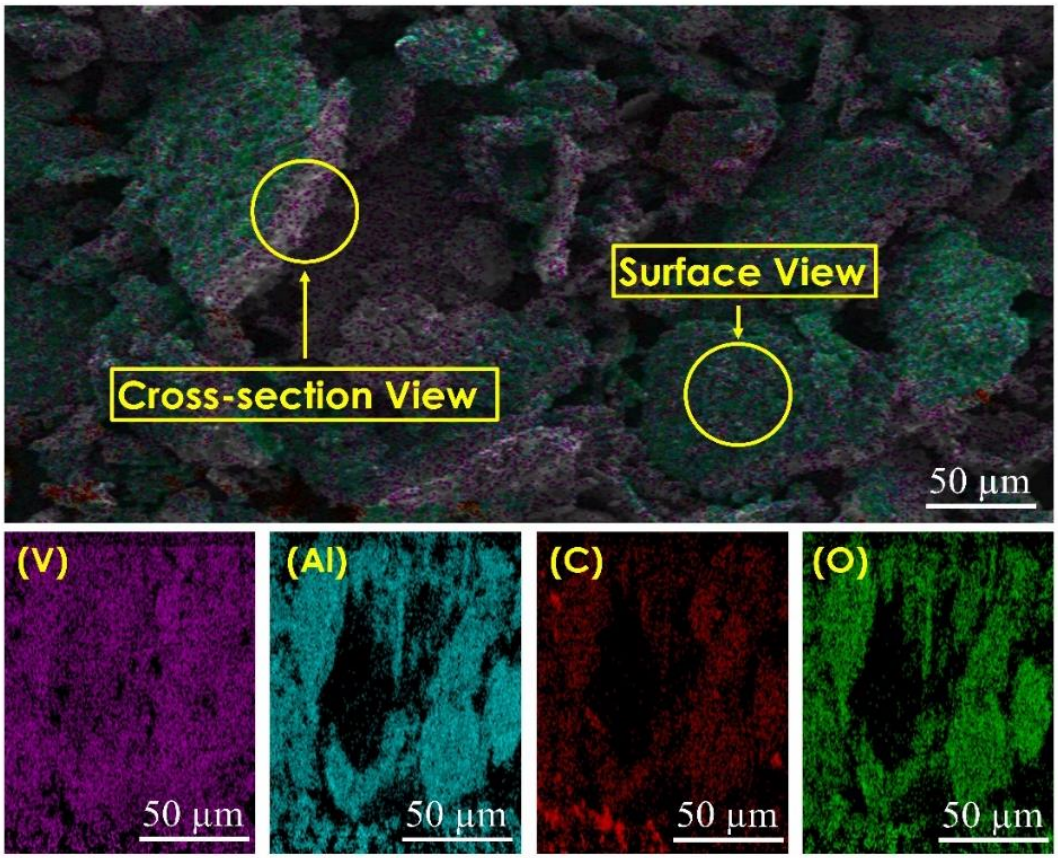

Figure 5. EDX elemental mapping of the V_2AlC MAX phase, V_2AlC/1500/4.

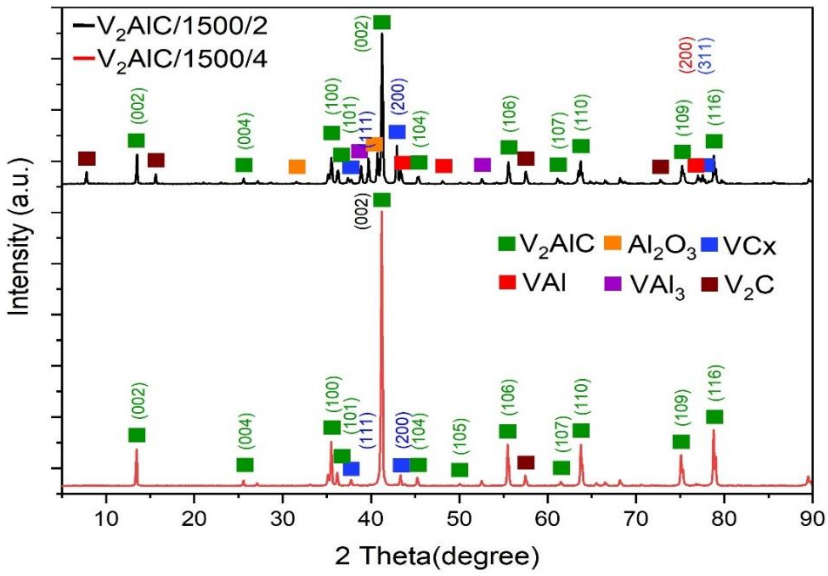

Figure 6. XRD analysis of the V_2AlC MAX phases: V_2AlC/1500/2 and V_2AlC/1500/4.

The formation of Al_2O_3 can be attributed to the initial oxidation at the surface of the starting powders of Al and V [20]. In addition, oxygen can originate from the air atmosphere, despite that the heating has been done under an argon gas environment. However, with a longer holding time with sufficient argon to stabilize the environment, the formation of Al_2O_3 is hindered. The holding time of 4 h produces the MAX phase efficiently, as compared to that of 2 h holding time, having more different compounds. A holding time of 4 h at 1500°C is capable of synthesizing good quality V_2AlC MAX phase, as confirmed by XRD results. With the corresponding EDX elemental mapping images (**Figure 5**) and SEM images (**Figure 3**) reveal fine lamellar morphology, the synthesis of the V_2AlC MAX phase is thus optimized and established.

The quality assessment of the V_2AlC MAX phase reveals several notable features. The synthesized material exhibited high purity, as confirmed by XRD and EDX analyses. The microstructure displayed a fine lamellar morphology, suggesting a well-formed V_2AlC MAX phase.

CONCLUSIONS

In conclusion, this study has presented a process optimization approach for the synthesis of the V_2AlC MAX phase, with a specific focus on enhancing sustainable production practices. By systematically investigating and optimizing key parameters such as reaction temperature, and reaction time, we have successfully achieved significant improvements in energy efficiency and waste reduction. The results say that the optimum temperature of synthesis V_2AlC is 1500°C. This not only contributes to sustainable production practices but also promotes the circular economy by minimizing the dependence on raw materials and reducing waste generation. The characterization of the synthesized V_2AlC MAX phase samples using various analytical techniques has confirmed their phase purity, desirable microstructure, and excellent properties. For purity assurance, the best holding time is 4 h, and the heating grading is 5°C/minute. These results validate the effectiveness of the process optimization approach in achieving sustainable production without sacrificing the desired material quality. The optimized synthesis conditions presented in this study provide a solid foundation for scaling up the sustainable production of the V_2AlC MAX phase. Future research directions may include exploring additional process parameters, investigating the long-term stability of the optimized synthesis conditions, and further analyzing the economic viability of sustainable production methods. The process optimization for the synthesis of the V_2AlC MAX phase presented in this study not only enhances sustainable production practices but also paves the way for the widespread adoption of MAX phases, driving the development of advanced materials and promoting a more sustainable and environmentally conscious manufacturing industry.

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