

# Design and Synthesis of Novel Nano-laminated Particulates for Fabricating Novel Multifunctional Composites

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**ABSTRACT:** In this paper, we report the manufacturing and design of novel nano-laminated ternary carbides. It was demonstrated that the particle size of  $Ti_3SiC_2$  particulates can be engineered by ball milling. In addition, we also report the synthesis of  $Cr_2AlC$ ,  $Cr_2GaC$ , and  $V_2AlC$  particulates after sintering at  $1350^\circ C$  for 4 h,  $1100^\circ C$  for 200 min, and  $1550^\circ C$  for 120 minutes, respectively.

**KEYWORDS:** MAX Phases, particulates,  $Cr_2AlC$ ,  $Cr_2GaC$ , and  $V_2AlC$

## I. INTRODUCTION

$M_{n+1}AX_n$  (MAX) phases (over 60+ phases), where  $n=1,2,3$ ; M is an Early Transitional Metal, A is a Group A element (mostly groups 13 and 14); and X is C and/or N, are novel ternary carbides or nitride which have attractive properties like damage tolerance, thermal shock resistance and machinability [1-5]. Recently, composites of MAX phases with different polymers and metals have attracted a lot of attention [6-13]. In order to further exploit these materials, it is critical to design and engineer novel MAX Phases particulates. The aim of this paper is to report the synthesis and characterization of novel particulates of  $Ti_3SiC_2$ ,  $Cr_2AlC$ ,  $Cr_2GaC$ , and  $V_2AlC$ .

## II. EXPERIMENTAL DETAILS

$Ti_3SiC_2$  powder (-325 mesh, Kanthal, Hallstahammar, Sweden) was ball milled (8000 M mixer Mill, SPEX SamplePrep, Metuchen, NJ) for 30 min in a 8001B hardened steel vial with 8001B hardened steel balls (Composition A). Table 1 shows the specifications of the powder used during this study. For fabricating, (a)  $Cr_2AlC$  (Composition B), a ratio of Cr:Al:C was 2:1.1:1, (b)  $Cr_2GaC$  (Composition C), a ratio of Cr:Ga:C was 2:1.4:1, and (c)  $V_2AlC$  (Composition D), a ratio of V:Al:C was 2:1.2:1, was used. The required ratio of powders was mixed in the ball milled in a methacrylate vial with methacrylate balls for 5 min except  $Cr_2GaC$  (Composition C).

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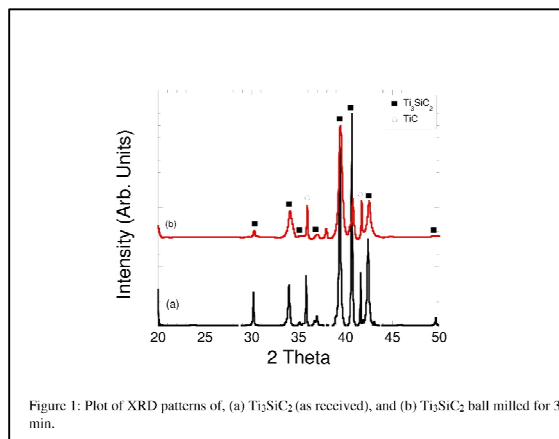
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Table 1: Specification of the raw materials used during this study

Powders	Source	Specifications
Chromium	Part 266299, Sigma-Aldrich, St. Louis, MO	powder, $\geq 99\%$ trace metal basis, -325 mesh
Vanadium	Part 262935, Sigma-Aldrich, St. Louis, MO	powder, 99.5% trace metal basis, -325 mesh
Aluminum	Lot KO5Z034, Alfa Aesar, Haverhill, MA	powder, 99.5% trace metal basis, -325 mesh
Carbon	Lot Y19B065, Alfa Aesar, Haverhill, MA	Graphite powder crystalline, 99%, -325 mesh
Gallium	100 g, Rotometals Inc., San Leandro, CA	99.99% Ingot

For synthesizing  $\text{Cr}_2\text{GaC}$  particulates, the required ratio of Cr and C were dry milled for 5 min, thereafter required amount of Ga was added to the mixed powders, and thereafter it was mixed in a mortar and pestle for at least 15 minutes.

All the powders were then cold pressed and subsequently sintered at different temperatures in a tube furnace with Ar gas flowing through the furnace. Compositions A and B were heated at  $10\text{ }^\circ\text{C}/\text{min}$  to the desired temperature, whereas composition C was heated at  $5\text{ }^\circ\text{C}/\text{min}$  to the desired temperature. The sintered powders were then crushed in a ball mill, and then sieved until -325 mesh in a sieve shaker.



XRD measurements were performed by a Rigaku Diffractometer (SmartLab, Rigaku, Japan) at a scan rate of  $0.05\text{ }^\circ/\text{min}$  from  $20^\circ$  to  $50^\circ$ . For all samples, Secondary electron (SE) images were obtained by using a JEOL JSM-6490LV Scanning Electron Microscope (JEOL USA, Inc., Peabody, Massachusetts).

### III. RESULTS AND DISCUSSION

Figure 1 compares the XRD pattern of  $\text{Ti}_3\text{SiC}_2$  after milling with as-received  $\text{Ti}_3\text{SiC}_2$  (Composition A). By analyzing the XRD patterns of both the samples, we can conclude that there is no sign of oxidation in the milled samples. Figure 2 compares the SE SEM micrographs of  $\text{Ti}_3\text{SiC}_2$  particulates before and after milling. By comparing the micrographs, we can conclude that ball milled powders have lower particle size as compared to as-received  $\text{Ti}_3\text{SiC}_2$ .

Figure 3 shows the XRD patterns of Composition B sintered at different temperatures. Significant amount of binary carbide ( $\text{Cr}_7\text{C}_3$ ) was observed after sintering at

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1300°C for 1 h. The concentration of Cr<sub>7</sub>C<sub>3</sub> decreased on heat treatment at 1300°C for 4 h, and the peaks of Cr<sub>2</sub>AlC increased. On treatment at 1350°C for 4 h, only peaks of Cr<sub>2</sub>AlC were observed. Thus, 1350°C for 4 h can be used as an optimum temperature for synthesizing Cr<sub>2</sub>AlC powders.

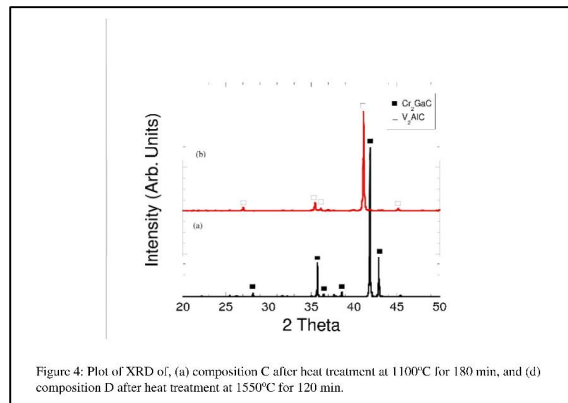


Figure 4: Plot of XRD of, (a) composition C after heat treatment at 1100°C for 180 min, and (d) composition D after heat treatment at 1550°C for 120 min.

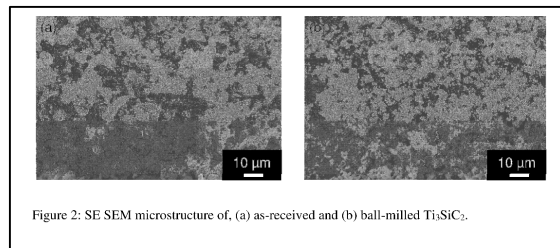


Figure 2: SE SEM microstructure of, (a) as-received and (b) ball-milled Ti<sub>3</sub>SiC<sub>2</sub>.

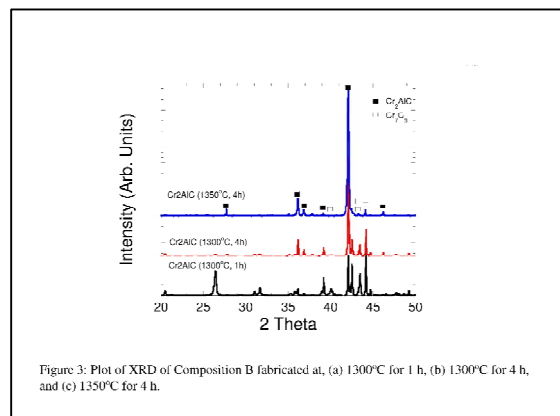
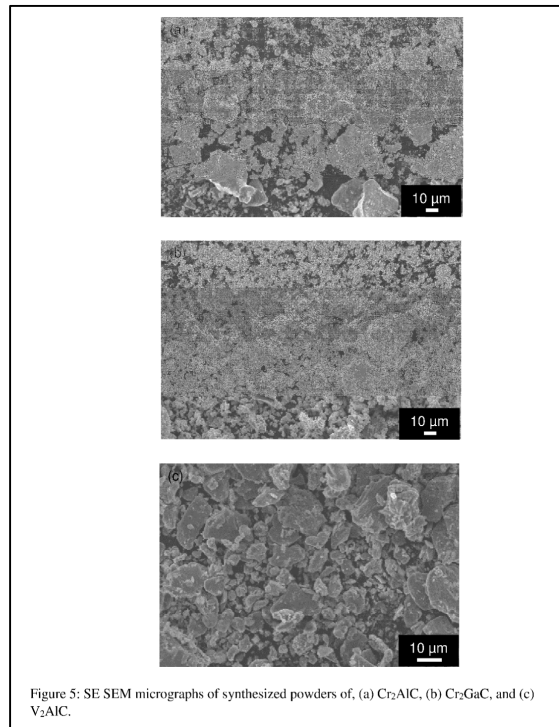


Figure 3: Plot of XRD of Composition B fabricated at, (a) 1300°C for 1 h, (b) 1300°C for 4 h, and (c) 1350°C for 4 h.



Xue et al. [14] and Su et al. [15] had also reported the synthesis of Cr<sub>2</sub>AlC particulates within a similar temperature range. Similarly, Figure 4 shows the XRD patterns of Cr<sub>2</sub>GaC (Composition C) and V<sub>2</sub>AlC (Composition D) powders after heat treatment at 1100°C for 200 min and 1550°C for 120 min, respectively. Both the compositions showed the formation of single phase of Cr<sub>2</sub>GaC and V<sub>2</sub>AlC, respectively. Earlier, Cuskelly et al. [16] have reported the synthesis of Cr<sub>2</sub>GaC by carbothermal process. As far as authors are aware, this is the first report to synthesize Cr<sub>2</sub>GaC particulates by sintering. Earlier, Neguib et al. [17] have reported the synthesis of V<sub>2</sub>AlC particulates by sintering at 1500°C for 4h. Figure 5 shows the typical distribution of the ball milled and sieved particles of different compositions. In future, these particulates can be used for designing composites.

#### IV. CONCLUSIONS

The important conclusions of this paper are: (a) the particle size of Ti<sub>3</sub>SiC<sub>2</sub> particulates can be engineered by ball milling, (b) Cr<sub>2</sub>AlC particulates can be synthesized after sintering at 1350°C for 4 h, (c) Cr<sub>2</sub>GaC particulates can be synthesized by sintering at 1100°C for 200 min, and (d) V<sub>2</sub>AlC particulates can be synthesized after sintering at 1550°C for 120 minutes.

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