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 Ti3SiC2 particulates can engineered by ball milling. In addition, we also report the synthesis of Cr2AlC, Cr2GaC, and V2AlC particulates after sintering at 1350°C for 4 h, 1100°C for 200 min, and 1550°C for 120 minutes, respectively.

KEYWORDS: MAX Phases, particulates, Cr2AlC, Cr2GaC, and V2AlC

I. INTRODUCTION

Mn+1AXn (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 synthesize and characterization of novel particulates of Ti3SiC2, Cr2AlC, Cr2GaC, and V2AlC.

II. EXPERIMENTAL DETAILS

Ti3SiC2 powder (-325 mesh, Kanthal, Hallstahamm, 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) Cr2AlC (Composition B), a ratio of Cr:Al:C was 2:1.1:1, (b) Cr2GaC (Composition C), a ratio of Cr:Ga:C was 2:1.4:1, and (c) V2AlC (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 Cr2GaC (Composition C).
Table 1: Specification of the raw materials used during this study

<table>
<thead>
<tr>
<th>Powders</th>
<th>Source</th>
<th>Specifications</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chromium</td>
<td>Part 266299, Sigma-</td>
<td>powder, &gt;99% trace</td>
</tr>
<tr>
<td></td>
<td>Aldrich, St. Louis, MO</td>
<td>metal basis, -325 mesh</td>
</tr>
<tr>
<td>Vanadium</td>
<td>Part 262935, Sigma-</td>
<td>powder, 99.5% trace</td>
</tr>
<tr>
<td></td>
<td>Aldrich, St. Louis, MO</td>
<td>metal basis, -325 mesh</td>
</tr>
<tr>
<td>Aluminum</td>
<td>Lot K032034, Alfa</td>
<td>powder, 99.5% trace</td>
</tr>
<tr>
<td></td>
<td>Aesar, Haverhill, MA</td>
<td>metal basis, -325 mesh</td>
</tr>
<tr>
<td>Carbon</td>
<td>LotY19B065, Alfa</td>
<td>Graphite powder crystalline,</td>
</tr>
<tr>
<td></td>
<td>Aesar, Haverhill, MA</td>
<td>99%, -325 mesh</td>
</tr>
<tr>
<td>Gallium</td>
<td>100 g, Rotometals Inc.,</td>
<td>99.99% Ingot</td>
</tr>
<tr>
<td></td>
<td>San Leandro, CA</td>
<td></td>
</tr>
</tbody>
</table>

For synthesizing Cr2GaC 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 °C/min to the desired temperature, whereas composition C was heated at 5 °C/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 °/min from 20° to 50°. 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 Ti3SiC2 after milling with as-received Ti3SiC2 (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 Ti3SiC2 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 Ti3SiC2.

Figure 3 shows the XRD patterns of Composition B sintered at different temperatures. Significant amount of binary carbide (Cr7C3) was observed after sintering at
1300°C for 1 h. The concentration of Cr7C3 decreased on heat treatment at 1300°C for 4 h, and the peaks of Cr2AlC increased. On treatment at 1350°C for 4 h, only peaks of Cr2AlC were observed. Thus, 1350°C for 4 h can be used as an optimum temperature for synthesizing Cr2AlC powders.
Xue et al. [14] and Su et al. [15] had also reported the synthesis of Cr2AlC partilcat within a similar temperature range. Similarly, Figure 4 shows the XRD patterns of Cr2GaC (Composition C) and V2AlC (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 Cr2GaC and V2AlC, respectively. Earlier, Cuskelley et al. [16] have reported the synthesis of Cr2GaC by carbothermal process. As far as authors are aware, this is the first report to synthesize Cr2GaC partilcat by sintering. Earlier, Neguib et al. [17] have reported the synthesis of V2AlC partilcat by sintering at 1500°C for 4 h. Figure 5 shows the typical distribution of the ball milled and sieved particles of different compositions. In future, these partilcats can be used for designing composites.

IV. CONCLUSIONS

The important conclusions of this paper are: (a) the particle size of Ti3SiC2 partilcat can engineered by ball milling, (b) Cr2AlC partilcat can be synthesized after sintering at 1350°C for 4 h, (c) Cr2GaC partilcat can be synthesized by sintering at 1100°C for 200 min, and (d) V2AlC partilcat can be synthesized after sintering at 1550°C for 120 minutes.

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REFERENCES


BIOGRAPHY

Matt Fuka joined the University of North Dakota in Summer 2016 to pursue an MSc in mechanical engineering.

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