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Structural and Phase Stability in the 2TiC-Al-Ti System During Milling and Subsequent Annealing

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ABSTRACT

The aim of the present research was to examine the structural and phase transformations, as well as the phase stability, in the 2TiC-Al-Ti system. A specific ratio of TiC, Al, and Ti powder mixture, based on the stoichiometric reaction for the formation of the Ti3AlC2 compound, was prepared and subjected to milling and annealing processes. The prepared samples were analyzed using scanning electron microscopy (SEM), X-ray diffraction (XRD), and differential scanning calorimetry (DSC). The results showed that milling the 2TiC-Al-Ti powder mixture did not result in the formation of a single-phase Ti3AlC2. Instead, the final structure consisted of a combination of TiC and Ti3AlC2 phases. The Ti3AlC2 phase formed during the milling process was unstable and transformed into a single-phase TiCx structure upon further milling or annealing. Additionally, the effect of the partial addition of Sn and Si on the structural and phase changes in the 2TiC-Al-Ti system during the milling and annealing processes was investigated. It was found that the addition of these elements had little effect on the formation and stability of the Ti3AlC2 compound.

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1. INTRODUCTION

Titanium carbide (TiC) forms very stable bonds and does not degrade at any temperature. This carbide is widely used in high-efficiency electrical systems due to its high melting point (3373 °C), as well as its electrical (61 $cm^{-1}\Omega\mu$) and thermal conductivity (33.2 $Wm^{-1}C^{-1}$) (Baviera, et al., 2001). However, the extensive use of TiC has been limited due to its inherent brittleness and tendency for sudden failure. To address this issue, the MAX phase structure has been developed in the Ti-Al-C system. MAX phases are defined by the general formula $M_{n+1}AX_n$, where M represents a transition metal, A is an element from group A (mostly from groups 3, 4, or 5 of the periodic table), and X denotes nitrogen or carbon. These materials exhibit a layered structure and possess unique ceramic and metallic properties (Zhang, et al., 2021) (Kumar, et al., 2022).

The formation of the MAX phase in the Ti-Al-C system has been studied by various researchers, who have used a range of raw materials such as Ti, Al, C, TiAl, TiCx, TiH, Al_2O_3, and Al_4C_3. Different synthesis methods have also been employed, including hot pressing (HP) (Li, et al., 2020) (Yoshida, et al., 2019), hot isostatic pressing (HIP) (Yu, et al., 2020), spark plasma sintering (SPS) (Eryomina, et al., 2021) (Gao, et al., 2020) and self-propagating high-temperature synthesis (SHS)

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(Averichev, et al., 2019) (Pazniak, et al., 2019). For example, Tzenov et al. (Tzenov, et al., 2000) investigated the phase transformation of a Ti, Al_4C_3, and C powder mixture using hot isostatic pressing, applying a pressure of 70 MPa and a temperature of 1400 °C to form the MAX phase. Wang et al. (Wang, et al., 2003) successfully synthesized the Ti_3AlC_2 intermetallic compound via hot pressing (25 MPa at 1500 °C), followed by annealing at 1200 °C. Hongxiang et al. (Hongxiang, et al., 2005) synthesized the Ti_2AlC compound using the SPS method at 1100 °C. They observed that increasing the sintering temperature to 1100 °C caused the Ti_2AlC phase to transform into Ti_3AlC_2, indicating the weaker thermal stability of Ti_2AlC. The work of Łopaciński (Łopaciński, et al., 2001) and Ge et al. (Ge, et al., 2003) on the formation of MAX phases using TiC and Al_4C_3 precursors and the SHS process, and the effect of TiC addition on the formation of the Ti_3AlC_2 compound in Ti/Al/C powder mixtures, represent other significant research efforts in this field. Furthermore, several studies have explored the effect of adding Si and Sn elements on the likelihood of reactions and the prevention of unwanted intermetallic compounds such as TiC and Ti_xAl_y (Cai, et al., 2018) (Cai, et al., 2021) (Guo, et al., 2021).

Despite extensive research on the formation of the Ti_3AlC_2 phase, the formation mechanisms and structural changes of this phase during annealing have not been thoroughly studied. In this regard, the present research aims to explore the structural and phase transformations in the 2TiC-Al-Ti system during milling and annealing processes. In addition, the effect of Si and Sn elements on the formation and thermal stability of Ti_3AlC_2 is investigated.

2. MATERIALS AND METHODS

In this research, raw materials with 99.8% purity and particle sizes of about 30 µm, including Ti, Al, TiC, Sn, and Si, were used to synthesize 2TiC-Al-Ti, 2TiC-Al-Ti-xSn, and 2TiC-Al-Ti-xSi (x = 0.1, 0.2, and 0.3) compounds. The powder samples were prepared by milling using an XQM-2A (Tencan company) at a rotation speed of 400 rpm with a ball-to-powder ratio of 10:1. The milling process was carried out in a tungsten carbide chamber using zirconia balls. The annealing process was performed within a temperature range of 350–1400 °C under an argon atmosphere, with a heating rate of 20 °C/min, using a Nabertherm furnace model HTCT 03/16.

The structural changes in the specimens were monitored using XRD (X-ray diffraction) analysis with a diffractometer (Philips PW3710) equipped with Cu Kα radiation (λ = 0.15406 nm, 40 kV). The measurements were taken with a step size of 0.05° and a time per step of 1 s. Morphological analyses of the powder samples were performed using scanning electron microscopy (SEM) with a VEGA-TESCAN-XMU. ImageJ software was used to determine the average particle sizes of the samples. Additionally, differential scanning calorimetry (DSC) was conducted to evaluate the thermal stability of the produced samples, using a Netzsch STA 409 PC/PG differential thermal analyzer. The samples were placed in Al2O3 pans and heated under a dynamic argon atmosphere (99% purity) up to 1000 °C, with a heating rate of 20 °C/min.

3. RESULTS AND DISCUSSION

The XRD patterns of the 2TiC-Al-Ti powder mixture after various milling times, up to 50 hours, are presented in Fig. 1. As shown, significant changes occur in the diffraction patterns as milling time increases. The XRD patterns of the powder samples at the initial stages of milling contain only the peaks corresponding to the raw materials, with no indication of a reaction. As milling progresses to 25 hours, the peaks related to Ti and Al completely disappear, and new peaks corresponding to the Ti_3AlC_2 phase appear in the XRD pattern.

Temperature measurements within the milling chamber, using a wireless thermocouple monitoring system, indicate that the self-propagating reaction responsible for the formation of the Ti_3AlC_2 phase occurs after approximately 20 hours of milling. The temperature increase during this reaction is estimated to be around 35 $^{\circ}$ C. Fig. 2 shows the DSC heating trace of the 2TiC-Al-Ti powder mixture after 25 hours of milling. Notably, there are no signs of endothermic or exothermic reactions in this trace. The absence of an Al melting peak near 660°C confirms the reaction between the initial materials and the formation of the intended phase during milling. This can be explained by the negative Gibbs free energy change for the formation of the Ti_3AlC_2 phase (ΔG^o = -540 kJ/mol (Shahin, et al., 2016)), as well as the adiabatic reaction temperature of 3643 K, which exceeds the threshold value of T_{ad} > 1800 K. In line with this, Yu et al. (Ye, et al., 1997) reported that exothermic reactions in the Ti-Al-C system result in local temperatures exceeding 1900°C.

It is important to note that the reduction in crystalline size and the increase in crystalline defect density (such as dislocations, voids, and grain boundaries) during the milling process are the primary factors contributing to the instability of the milled samples and the formation of the Ti_3AlC_2 phase (Mossino, et al., 2004). Fig. 3 shows SEM images of the powder particles after 25 hours of milling. As seen, the particles exhibit a pseudo-spherical and uniform morphology, with a mean particle size of approximately 400 nm. There is no evidence of sharp or crushed particles in this image, further supporting that the reaction occurs as a self-propagating process during milling (El-Eskandarany., 2015) (Whang., 2011).

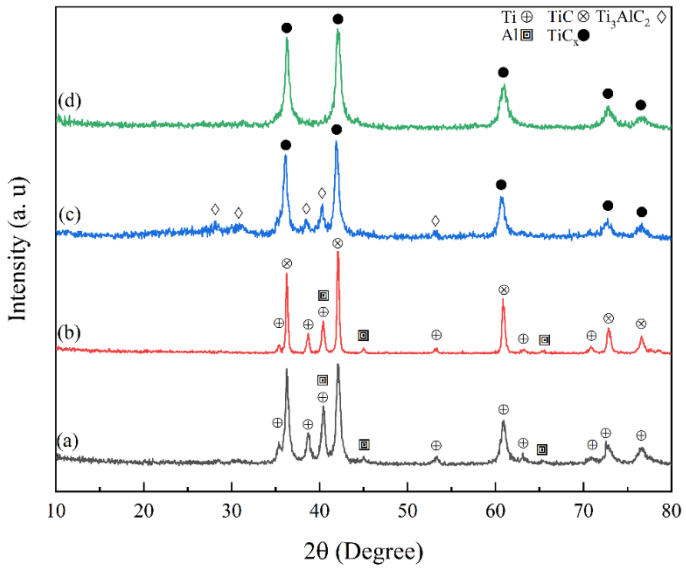


Figure 1. The XRD patterns of 2TiC-Al-Ti powder mixture after different milling periods: a) 0, b) 5, c) 25, and d) 50 h.

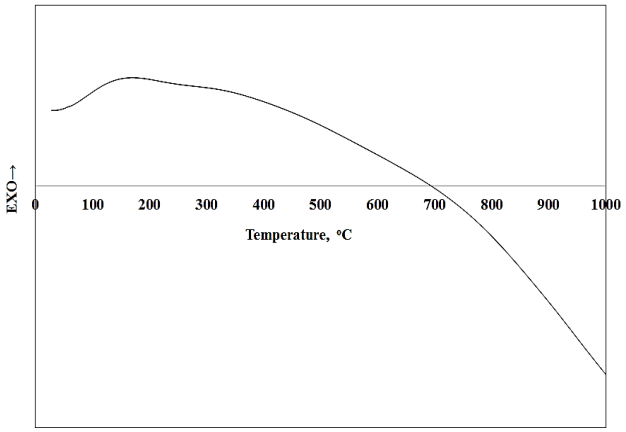
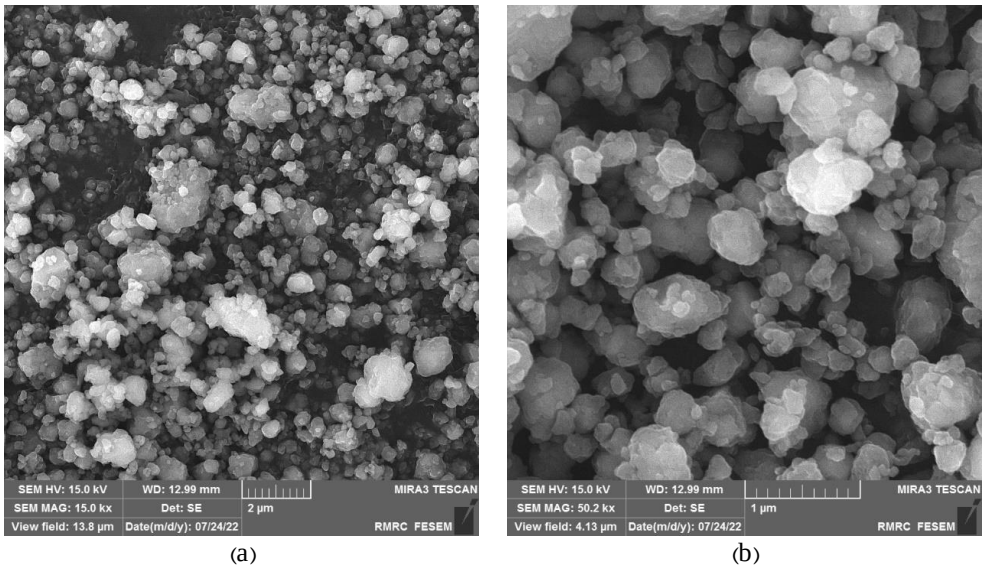


Figure 2. The DSC heating trace of 2TiC-Al-Ti powder mixture after 25 h of milling.



(a) (b)

Figure 3. The SEM micrographs of 2TiC-Al-Ti powder mixture after 25 h of milling (at two different magnifications).

Moreover, as shown in Fig. 1(d), the formed Ti_3AlC_2 compound is unstable and transforms into the TiC_x phase as the milling process continues up to 50 hours. According to the Ti-C equilibrium diagram, the TiC compound is a non-stoichiometric ceramic and remains stable across a wide compositional range ($32 \leq C > 50$ at. %). The formation of a single-phase TiC_x compound in the Ti-Al-C system during milling has also been confirmed by other researchers, such as Li et al. (Li, et al., 2006). However, the sequence of phase transformations in the 2TiC-Al-Ti system during the milling process can be described as follows:

$$2TiC\text{-}Al\text{-}Ti \rightarrow TiC + Ti_3AlC_2 \rightarrow TiC_x \quad (1)$$

To explore the thermal stability of the Ti_3AlC_2 phase produced during the milling process, as well as the potential phase changes during annealing, the 25-hour milled sample underwent heat treatment at temperatures ranging from 350°C to 1400°C. The XRD patterns of the annealed samples are shown in Fig. 4. As observed, with increasing annealing temperature, the intensity of the peaks corresponding to the Ti_3AlC_2 phase gradually decreases and is completely eliminated from the diffraction patterns upon reaching 1000°C. At this point, several peaks corresponding to the TiAl3 phase appear in the diffraction patterns. According to the literature and the Ti-Al-C equilibrium diagram, the stability region of the Ti_3AlC_2 compound is narrow (Bandyopadhyay, et al., 2000), with this phase forming only within the 1350-1450°C range. At other temperatures, this phase gradually decomposes into TiC_x, Ti_2AlC, and Ti_xAl_y intermetallic compounds (Pang, et al., 2010) (Yao, et al., 2015), which is consistent with the results obtained in this study.

As the annealing temperature increases up to 1200°C, the intensity of the peaks corresponding to the $TiAl_3$ phase also increases. These results are in line with the findings of Yoshida et al. (Yoshida., 2012), who reported the formation of $TiAl_3$ and TiAl intermetallic compounds within the 1100-1200°C range in the Ti-Al-C system. Based on Fig. 4, the $TiAl_3$ phase is unstable and gradually disappears from the XRD patterns during annealing up to 1400°C. At this temperature, the XRD pattern of the annealed sample includes only peaks related to the TiC_x compound. The formation of a TiC_x single-phase structure during annealing is similar to the structure produced in the 50-hour milled sample (Fig. 1(d)). This confirms that the stable phase in the 2TiC-Al-Ti system is the TiC_x phase. In other words, the excess Al and Ti dissolve into the carbide network without forming a new phase.

However, this result contrasts with Yoshida's research (Yoshida., 2012), which reported the stability of ternary Ti_3AlC_2 and Ti_2AlC phases between 1200°C and 1400°C. The difference in raw materials and production methods likely explains this discrepancy. The SEM morphological images of the annealed samples at 1400°C are shown in Fig. 5. As seen in this figure, the primary morphological characteristics of the resulting powder particles include a relatively uniform distribution of pseudo-spherical particles with a mean particle size of 300 nm.

The sequence of phase changes in this system during the annealing process can be described as follows:

$$TiC + Ti_3AlC_2 \rightarrow TiC + TiAl_3 \rightarrow TiC_x \quad (2)$$

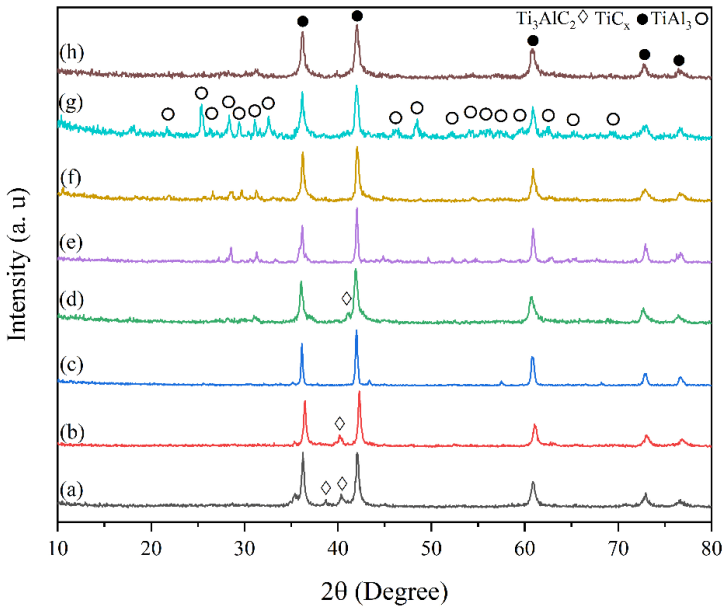
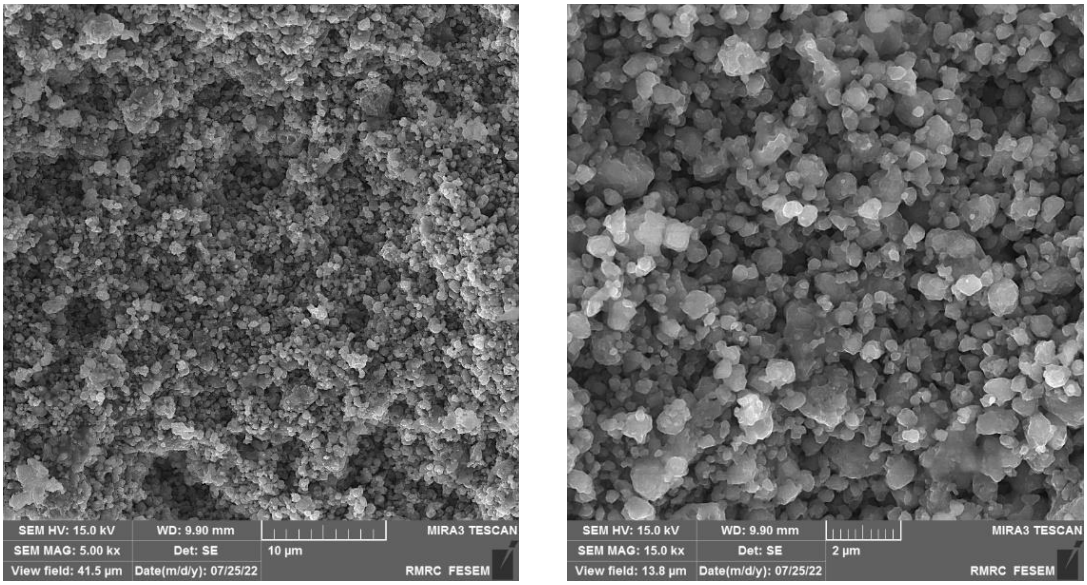


Figure 4. The XRD patterns of 2TiC-Al-Ti milled powder mixture for 25 h after annealing for 1 h: a) 350, b) 700, c) 900, d) 1000, e) 1100, f) 1200, g) 1300 and h) 1400 °C.



(a) (b)

Figure 5. The SEM micrographs of 2TiC-Al-Ti milled powder mixture for 25 h after annealing at 1400 ºC for 1 h.

According to the literature (Cai, et al., 2018) (Guo, et al., 2021), adding small amounts of Sn and Si elements to the Ti-Al-C system prevents the formation of binary intermetallic compounds such as TiC, Ti_xAl_y, and Al_2O_3, and also prevents thermal explosions. These two elements are incorporated into the intermetallic compound Ti_3AlC_2 as replacement elements for layer A (Al), increasing the density of dislocations. Since Sn and Si are more electronegative than Al, they strengthen the bond between the M-A and A-X layers. In this regard, increasing bond strength may contribute to the stability of the MAX phase. To investigate the effect of Sn and Si on the formation and stability of the Ti_3AlC_2 compound in the Ti-Al-C system, the XRD patterns of the 2TiC-Al-Ti-xSn and 2TiC-Al-Ti-xSi powder mixtures (x = 0.1, 0.2, 0.3) after 25 hours of milling are presented in Figs. 6 and 7, respectively. As observed, the XRD patterns of the examined samples containing these additives consist only of peaks corresponding to TiC_x and Ti_3AlC_2 phases. There is no evidence of Sn and Si in the XRD patterns. This is expected, as the percentage of these two elements is less than 5 vol.%, making them undetectable by XRD.

The absence of the Al melt peak in the DSC curves (Fig. 8) confirms the occurrence of reactions between the components during the milling process. The XRD patterns indicate that the addition of Sn and Si has a detrimental effect on the formation of the mentioned phases in the 2TiC-Al-Ti system. As shown in Figs. 6 and 7, the intensity of the Ti_3AlC_2 peaks decreases as the percentage of Sn and Si in the composition increases. These results contradict the findings of Li et al. (Li, et al., 2008), Mingxing et al. (Mingxing, et al., 2006), and Zhu et al. (Zhu, et al., 2004), who reported that these elements stabilize the Ti_3AlC_2 phase. However, the formation of the Ti_3AlC_2 compound in the 2TiC-Al-Ti system occurs through the SHS reaction during milling. The addition of Sn and Si reduces the local temperature and significantly diminishes the intensity of SHS reactions due to heat absorption, thereby affecting the formation of the resulting phases. In fact, Sn has a more pronounced effect in reducing the intensity of the Ti_3AlC_2 peaks than Si. This is likely due to the lower melting and evaporation temperatures of Sn compared to Si, enabling Sn to absorb more reaction heat and hinder the formation of the Ti_3AlC_2 compound.

The SEM morphological characteristics of the powder mixtures containing 0.1 atomic percent of Si and Sn are compared in Fig. 9. As observed, the morphology of the resulting powders in the presence of these additives is similar to the SEM images presented in Fig. 2, consisting of pseudo-spherical particles with an average size of about 400 nm.

The XRD patterns and SEM micrographs of the samples containing 0.1% Sn and Si after annealing at 1400°C are presented in Figs. 10 and 11. As seen, the phase and morphological structures of the examined samples with additives are very similar to those of the sample without additives, showing no significant differences. In this case, the phase structure consists solely of the TiC_x compound, the particles are spherical, and the average particle size is estimated to be around 300 nm. These results indicate that Sn and Si additives do not significantly influence the formation or stability of the Ti_3AlC_2 phase.

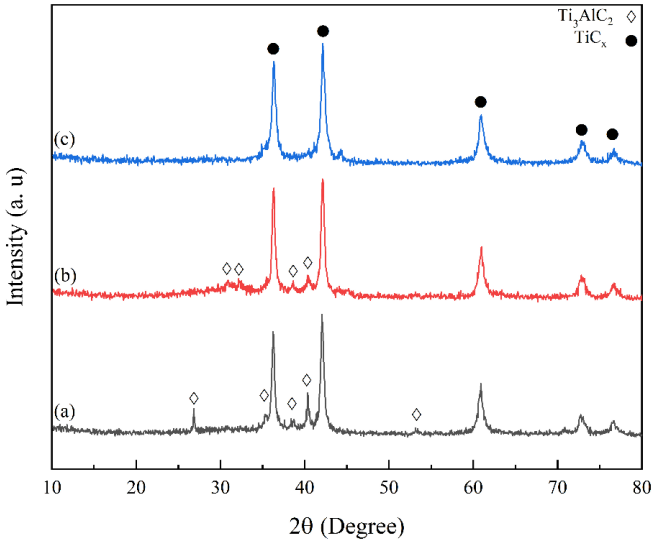


Figure 6. The XRD patterns of 2TiC-Al-Ti-xSn powder mixture after 25 h of milling: a) x=0.1, b) x=0.2 and c) x=0.3.

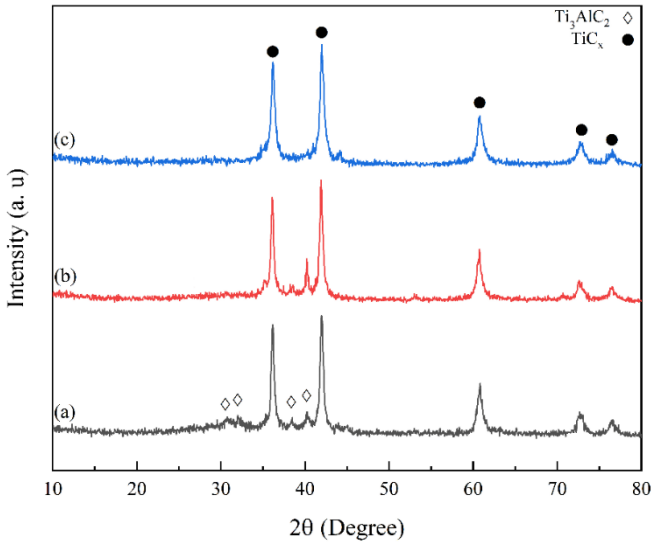


Figure 7. The XRD patterns of 2TiC-Al-Ti-xSi powder mixture after 25 h of milling: a) x=0.1, b) x=0.2 and c) x=0.3.

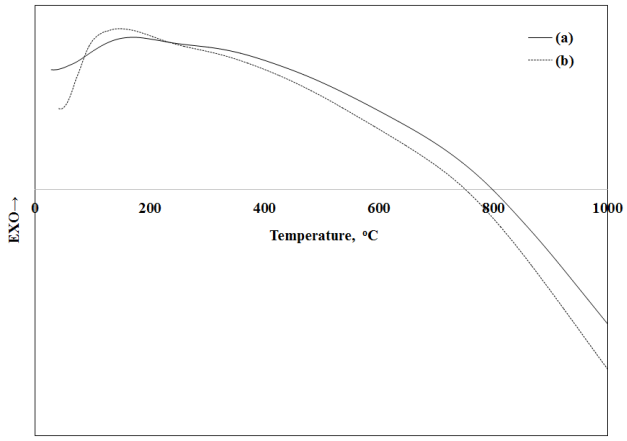


Figure 8. The DSC heating traces of a) 2TiC-Al-Ti-0.1Sn and b) 2TiC-Al-Ti-0.1Si powder mixtures after 25 h of milling.

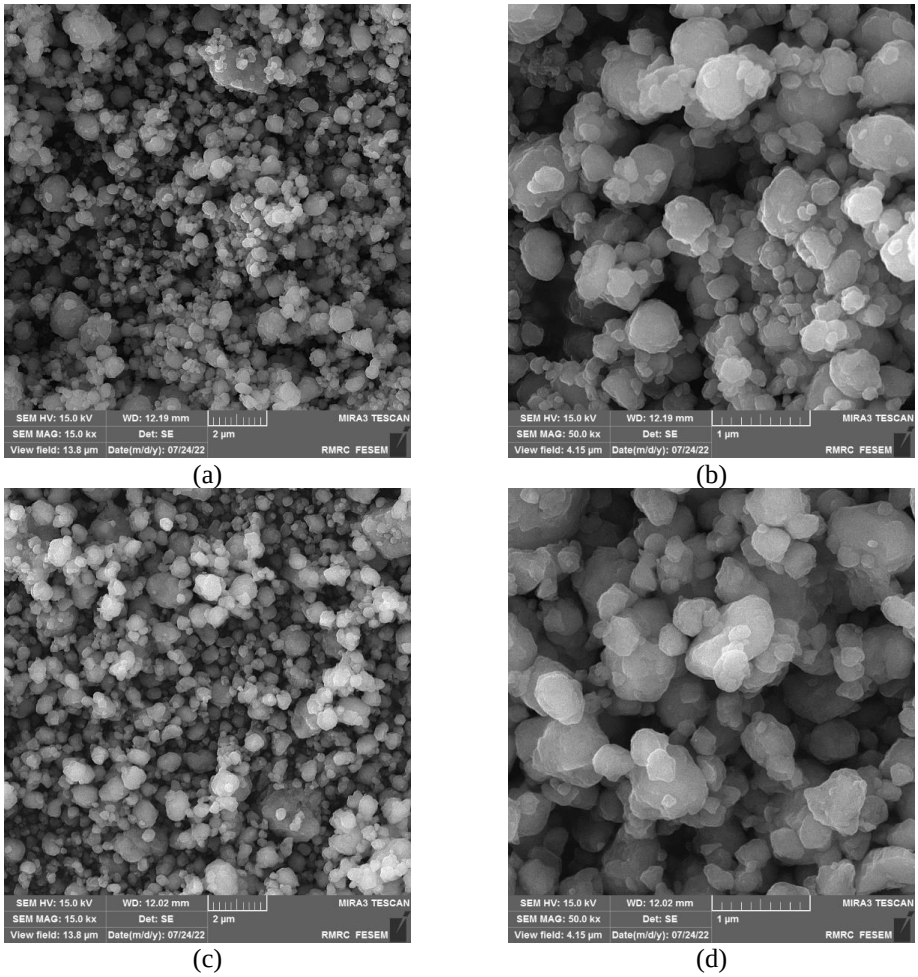


Figure 9. The SEM micrographs of a & b) 2TiC-Al-Ti-0.1Sn and c & d) 2TiC-Al-Ti-0.1Si powder mixtures after 25 h of milling.

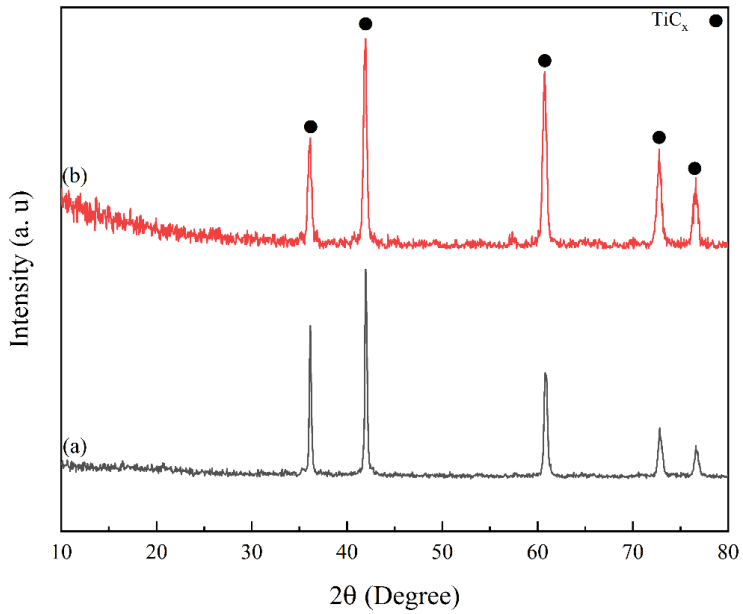


Figure 10. The XRD patterns of milled a) 2TiC-Al-Ti-0.1Sn and b) 2TiC-Al-Ti-0.1Si powder mixtures after heat treated at 1400 ºC for 1 h.

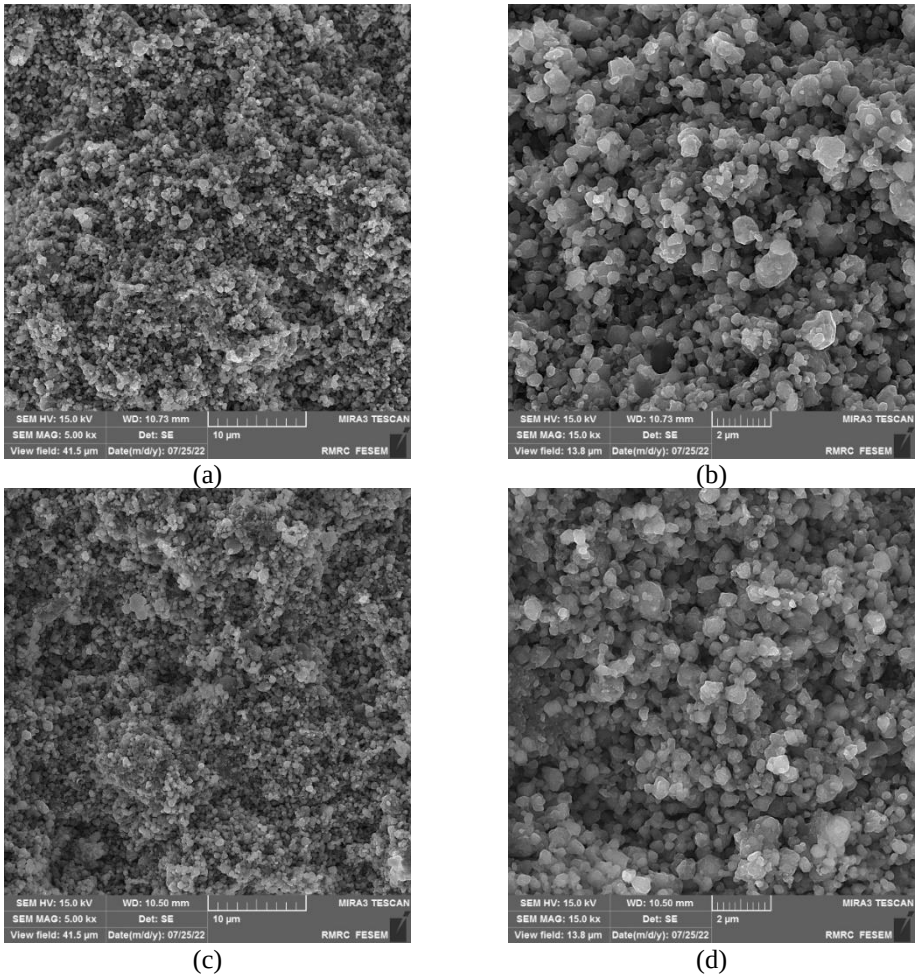


Figure 11. The SEM micrographs of milled a & b) 2TiC-Al-Ti-0.1Sn and c & d) 2TiC-Al-Ti-0.1Si powder mixtures after annealing at 1400 ^{o}C for 1 h.

4. CONCLUSION

This research focused on the structural and phase transformations in the 2TiC-Al-Ti system during the milling and annealing processes, as well as the effect of Sn and Si additions on the formation and stability of the Ti_3AlC_2 phase. The investigation revealed the following key findings:

- It was not possible to create a single-phase Ti_3AlC_2 structure through the milling process in the 2TiC-Al-Ti system. The structure obtained during milling was a combination of TiC_x and Ti_3AlC_2 phases.
- The Ti_3AlC_2 phase formed during milling in the 2TiC-Al-Ti system was unstable and transformed into a single-phase TiCx solid solution as milling continued.
- The TiC/Ti_3AlC_2 composite structure in the 2TiC-Al-Ti system transformed into TiC/Al_3Ti, and eventually into TiC_x, during annealing up to 1400 °C. Ultimately, the stable phase in the TiC-Al-Ti system was the single-phase TiC_x solid solution.
- The addition of Sn and Si had a negative effect on the formation and stability of the Ti_3AlC_2 compound.

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