Synthesis of $(Ti_x,W_{1-x})_3SiC_2$ MAX phase by mechanical milling

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ABSTRACT

This study has investigated the synthesis of $(Ti_{1-x}W_x)_3SiC_2$ MAX phase via high energy ball milling, and the effect of heat treatment and excess Si on the purity of synthesized powder were explored. In this regards, different mixtures of Ti, Si, and C were ball milled by a planetary ball mill for various milling times up to 15h. The phase evolution of products was studied by X-ray diffraction (XRD), and the morphological changes monitored by a field emission scanning electron microscopy (FESEM) equipped with energy-dispersive spectroscopy (EDS). The results showed that after 15 hours of ball milling the reaction started, and resulted in Ti_3SiC_2 and TiC formation. The as-synthesized powders were then compacted and heat treated at 600, 1000, 1250 and 1400˚C. Heat treatment caused to proceed in the reaction between the intermetallic compounds in Si-Ti system and TiC, and led to increasing the purity of Ti_3SiC_2. In a separate run, a non-stoichiometric composition of Ti: Si:C= 3:1.2:2 was ball milled for 15 h, and heat treated at 1250˚C. The XRD results showed that the purity of the product is higher than the stoichiometric composition. The addition of W to Ti_3SiC_2 was also explored. In this regard, the synthesis of $(Ti_{1-x}W_x)_3SiC_2$ component (x= 0.8, 0.5) was investigated, and the results showed that some W incorporated in Ti_3SiC_2 structure, and the WC and Ti_xW_{1-x} formed during the process.

1-Introduction

Until now, a large number of ternary compounds named $M_{n+1}AX_n$ phases (MAX for short, where n = 1, 2 or 3, M is an early transition metal, A is an A-group element (mostly groups 13 and 14), and X is C or N has been synthesized. This class of materials has been considered as a new and potential candidate for high-temperature applications and severe conditions because of their unique properties [1,2]. The main characteristics of MAX phase materials are the lamellar structure, which gives them a combination of metallic and ceramic properties. Metallic properties include thermal and electrical conductivities, easy machinability, and excellent thermal shock resistance whereas ceramic properties of MAX phases include high fracture toughness, high Young's moduli, high strength, high melting point, and thermal stability [3].

A member of this family, Ti_3SiC_2, is among the most characterized MAX phases to date, and their compressive and flexural strength, the hardness, oxidation resistance, fracture toughness, R-curve behavior, and tribological properties are known [2,4,5]. These properties make Ti_3SiC_2 useful in many fields; for example, it can be used as a high-temperature structural material as an alternative for expensive high-temperature alloys [6].

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There is a considerable amount of literature on the synthesis of Ti_3SiC_2 MAX phases by different methods such as hot isostatic pressing (HIP) [7], self-propagation high-temperature synthesis (SHS) [8,9], hot pressing (HP) [10–12], combustion synthesis (CS) [13], spark plasma sintering (SPS) [11,14] etc.

In addition to the above-mentioned methods for synthesis of Ti_3SiC_2 MAX phase, ball milling as a potential route in the synthesis of advanced materials has been used to fabricate this group of materials. As a powder metallurgy technique, the ball milling, also called mechanical alloying, is one of the most promising technologies for obtaining compounds at room temperature. It developed because of some advantages, including low fabrication cost, simple synthesis method, and easily industrialization [15–22].

This process recently has been successfully used to synthesize some of the MAX phases [23–25]. Several researchers have been used mechanical alloying to synthesize Ti_3SiC_2, and its solid solutions as well as its composites [26–28]. Mechanical alloying for synthesizing MAX phases has a lot of advantages as mentioned above, but a low purity of product caused to limit the application of this process. Researchers try some ways to solve this problem, i.e., use of some additives during ball milling, heat treating of the ball milled powder, etc. [29].

One of the disadvantages of MAX phases is the deficiencies in the mechanical properties, and in order to overcome this problem, some researchers developed solid solution treatment theoretically and experimentally [30–32]. Cui et al. [33] synthesized Ti_3AlC_2/W composites by SPS method and found that the hardness of composite increased with W addition, and the hardening effect is likely achieved by the Ti_xW_{1-x} interfacial layer providing strong bonding between Ti_3AlC_2 and W, and the presence of hard W. Wan et al. [34] doped $TiSi(Al)C_2$ by transition metal elements like Zr, Hf, or Nb to form $(Ti,X)_3Si(Al)C_2$ (X = Zr, Hf, Nb) solid solutions, and demonstrated that these solid solutions had significant high-temperature mechanical properties.

In this research work, the mechanical alloying of elemental powders was used to synthesize Ti_3SiC_2 MAX phase and the effects of excess Si during ball milling, and heat treatment of the powder mixtures after ball milling was examined to increase the purity of Ti_3SiC_2 MAX phase. Also, the possibility of $(Ti,W)_3SiC_2$ synthesis was investigated.

2. Materials and methods

Commercially available powders of Ti (particle size < 100µm, 99.7% purity), Si (particle size < 200µm, 99.7% purity), W (particle size <200µm, 99.7% purity), and C (particle size <200µm, 99.7% purity) were selected as raw materials for the synthesis of Ti_3SiC_2 and $(Ti,W)_3SiC_2$ MAX phase. These powders were weighed in a mole ratio according to the composition of Ti_3SiC_2 with Ti:Si:C = 3:X:2 (X=1, and 1.2). The excess Si for X= 1.2 is related to 20%. In addition, $(Ti_{1-x},W_x)_3SiC_2$ compositions with x=0.1, 0.2 and 0.3 was synthesized.

The powder according to the above-mentioned formulations were first put in a cylindrical steel vial with grinding balls having a 10 mm diameter. Retsch planetary-type high-energy ball mill PM 100 was used. Ball milling was performed by rotation speed set at 450 rpm, and the weight ratio of balls to powders was 10:1. The ball milled powders were compacted uniaxially under 200MPa in a K100 alloy steel mold. Then the samples were annealed at the different temperatures of 600, 1000, 1250 and 1400˚C for 3 hours under Ar atmosphere in a tube furnace, and followed cooling in the furnace.

The phase changes during ball milling and annealing of the powders was carried out by X-Ray diffraction (Philips PW-1800) by means of a Cu target, Kα radiation, (40 KV and 30 mA). Also, a scanning step of 0.05˚ and time per step of 1sec was used. The evaluation of the particle size and morphological changes of the samples after milling and annealing was performed by field emission scanning electron microscope (FESEM, Mira 3, TESCAN, Czech) equipped with energy dispersive spectroscopy (EDS). Moreover, the vial temperature was measured every 30 minutes by using a thermocouple during ball milling process to investigate the possible exothermic reactions during the milling process, because of the impossibility of the direct temperature measuring of the powders during the ball milling.

Mostly, the mechanical alloying process causes the formation of more than one product. Hence,

determining the content of each product after milling is important. The following formula was used to calculate the content of products [27]:

$$W_{TC} = \frac{(\frac{I_{TC}}{I_{TSC}})}{1.8+(\frac{I_{TC}}{I_{TSC}})} \text{ X } 100 \quad (1)$$

$$W_{TSC} = (1\text{-}W_{TC}) \text{ X } 100 \quad (2)$$

Where, W_{TC} and W_{TSC} are the weight percent of TiC and Ti_3SiC_2, respectively, and I_{TC} and I_{TSC} are the intensity of all peaks of TiC and Ti_3SiC_2 respectively.

3. Results and discussion

3.1. Stoichiometric ratio of Ti_3SiC_2

3.1.1. Milling process

Fig. 1 depicts the XRD pattern of Ti, Si, and C powder mixture with a stoichiometric ratio after different milling periods up to 10h. As it could be seen, the diffraction peak of 0h ball milling sample or as-received powder includes the peaks of initial powder elements. After 5 hours of ball milling, there is no significant reaction between the reactant elements, and no new phase appears, and just the intensity of Ti, Si, and C peaks was decreased. After 10 hours of ball milling, the peaks of C disappeared from the pattern because of extensive mixing with other elements. Finally, after 15 hours of ball milling, the peaks of initial powders disappeared, and the Ti_3SiC_2 and TiC peaks were observed.

Fig. 2 shows the variation of vial temperature as a function of ball milling time for the 3Ti/Si/2C powder mixture. It should be noticed that the temperature of the vial increased slowly as the milling time extended, which is mainly because of converting mechanical energy to the thermal energy. However, the vial temperature increased sharply to 95°C after 12.67 hours (760 min) milling, suggesting some exothermic reaction(s) occurred between Ti, Si, and graphite. It suggested a high exothermic reaction in the combustion mode occurred during ball milling. The dashed line is the temperature variation of the empty cup and is included in this figure for comparison. Several factors promoted this combustion reaction such as high Ti/Si/C interface areas, the short-circuit diffusion path provided by the increasing number of defects like dislocations and grain boundaries induced during ball milling [35]. The similar results were reported by other researchers for the synthesis of Ti_3SiC_2 and Ti_3AlC_2 MAX phases by mechanical alloying [23,36,37]. This is an important character that Ti_3SiC_2 is synthesized by mechanically induced self-propagation reaction mode (MSR). Also, several works showed that some TiC phase formed during the reactive synthesis of Ti_3SiC_2 and Ti_3AlC_2 in ball milling route. It may due to the short reaction time and large amounts of reaction heat released from the thermal explosion reaction between the Ti and C [24,31,38–40].

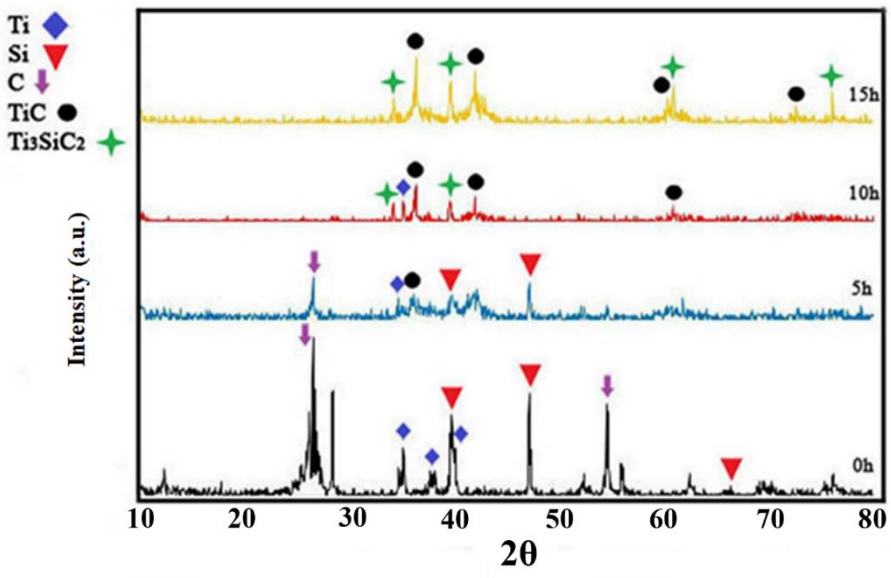


Fig. 1. XRD patterns of powder mixtures with a stoichiometric ratio of Ti_3SiC_2 after different milling times.

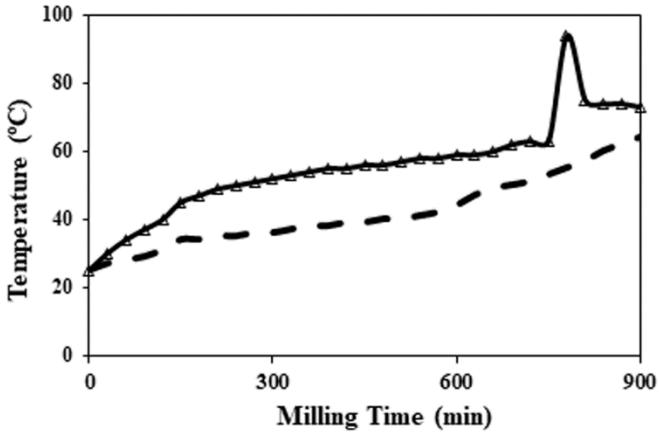


Fig. 2. Variation of vial temperature as a function of milling time for the 3Ti/Si/2C powder mixture.

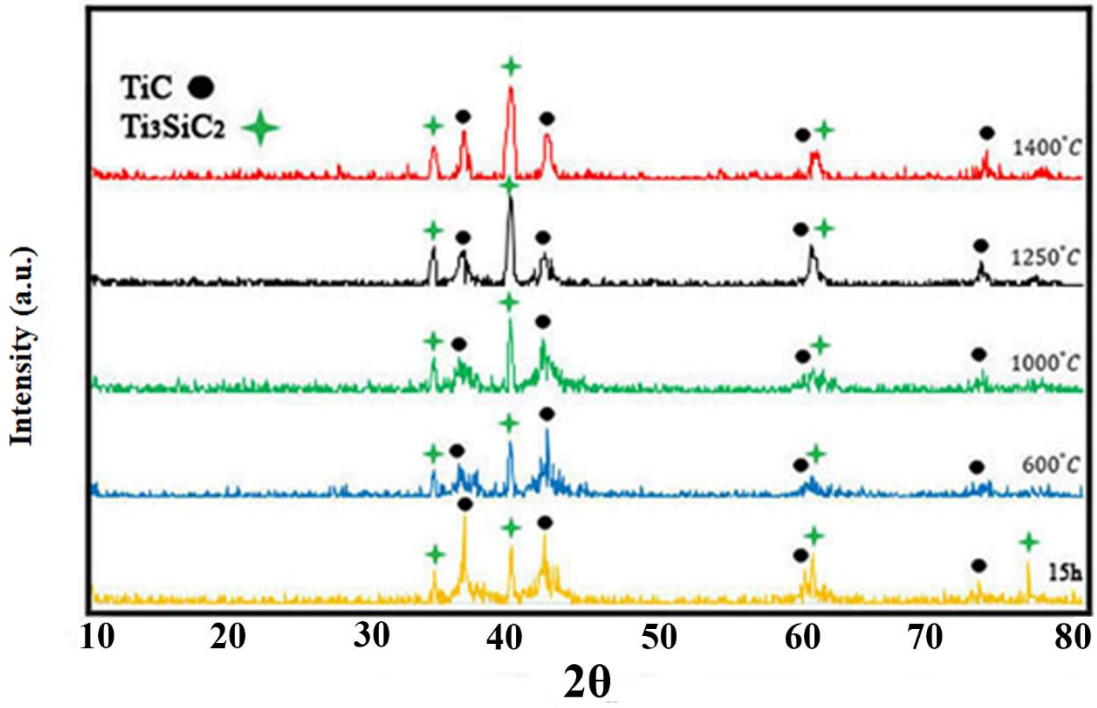


Fig.3. XRD patterns of powder mixture with the stoichiometric ratio: as-received, 15 hours ball milled and heat treated at after annealing at 600, 1000, 1250, and 1400˚C.

3.1.2. Heat treatment

Fig. 3 shows XRD patterns of 15 hours ball milled stoichiometric samples before and after annealing at 600, 1000, 1250, and 1400˚C. As can be seen, the intensity of the Ti_3SiC_2 peaks increases by increasing the heat treatment temperature, suggesting that the amount of this phase increases. The content of each product after milling was calculated by using Eq.1 and 2. The results showed that the 15 hours ball milled powder consist of 44 wt% Ti_3SiC_2 MAX phase, and after annealing at 600˚C the content of Ti_3SiC_2 remains almost constant, while by increasing the annealing temperature to 1000, 1250 and 1400˚C this amount increases to 56, 64 and 65 wt %, respectively. It is clear from these results that Ti_3SiC_2 was not obtained fully during ball milling, and a heat treatment is required for progressing the synthesis of Ti_3SiC_2 MAX phase. This is because that the TiC is a stable phase, and needs a considerable time at high temperature to complete the reactions to convert to Ti_3SiC_2. Moreover, the Ti_3SiC_2 content doesn't change considerably between 1250 and 1400˚C, and thus, 1250˚C can be

chosen as an optimum annealing temperature.

3.2. Nonstoichiometric ratios

In order to increase the content of Ti_3SiC_2 after ball milling method, the Si content in the initial powder mixture was used 20% more than the stoichiometric ratio. So, the Si content in the initial powder mixture was chosen as $Ti_3Si_{1.2}C_2$, i.e., a powder mixture of 3Ti/1.2Si/2C. Fig. 4 shows the XRD pattern of 15 hours ball milled sample, and after annealing at 1250˚C. The pattern depicted TiC and Ti_3SiC_2 phases, and the intensity of Ti_3SiC_2 peaks in this pattern is higher than TiC peaks. By adding Si more than stoichiometric composition, the purity of Ti_3SiC_2 MAX phase increased, and the content of Ti_3SiC_2 MAX phase has reached to 82 wt% and increased, even more, to 86 wt% after annealing at 1250˚C. So, the combination of using excess Si and heat treatment at 1250˚C significantly affects the purity of Ti_3SiC_2 phase and increased it up to 86 wt%.

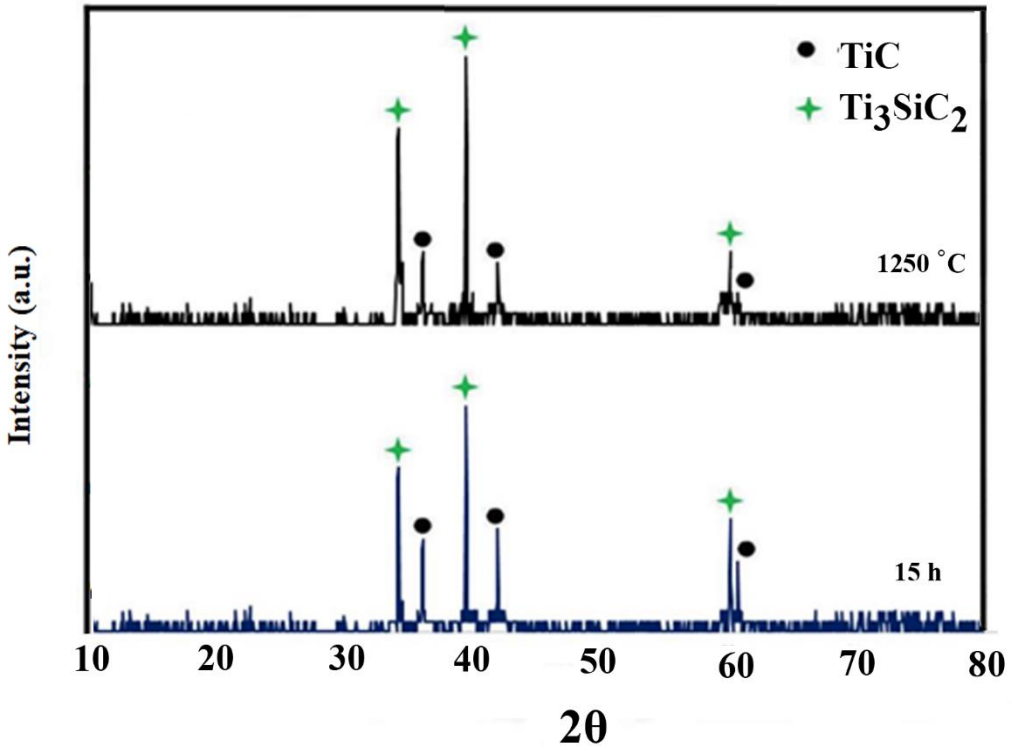


Fig. 4. XRD patterns of powder mixture with 3Ti/1.2Si/2C ratio, 15 hours ball milled, and 15hours ball milled-heat treated at 1250˚C for 3 hours.

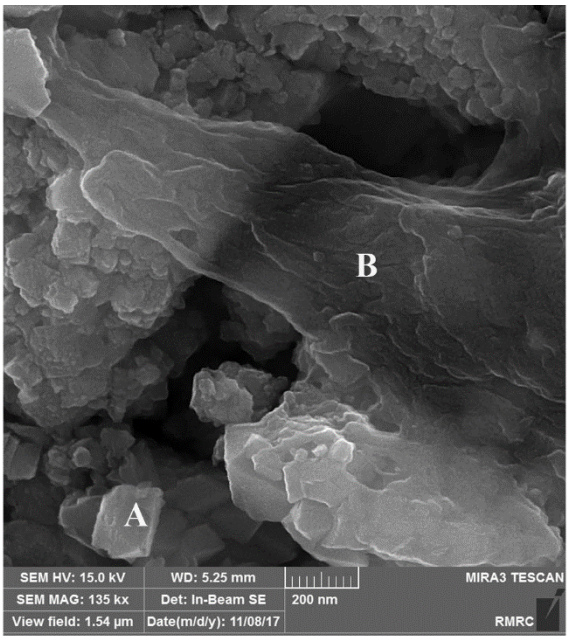
3.3. Microstructure of powder

Fig. 5 shows the SEM micrograph of 15 hours ball milled powder mixture with stoichiometric ratio and after annealing at 1250˚C. According to the XRD results, the two phases of TiC and Ti_3SiC_2 are the main phases, and clearly, the micrographs consist of two different morphology including granular and layered phases. The granular and layered phases are TiC and Ti_3SiC_2, respectively. It should be noted that the TiC grain is relatively fine (less than 500 nm), and possesses cuboid shape which could be formed by the growth of cubic TiC crystal particle. The EDS result from point A shows this point consists of Ti, and C elements. Also, the Ti_3SiC_2 powders have a layered shape, and the EDS results of point B in the Fig. 5 shows Ti, Si, and C elements. In addition, some Fe could be founded comes from ball milling cup.

3.4. $(Ti_{1-x},W_x)_3SiC_2$ system

A solid solution is a useful way to enhance mechanical properties of the MAX phases, and fortunately, both the M- and A-atoms can be substituted with other M or A-atoms [2]. In this regard, the synthesis of $(Ti_xW_{1-x})_3SiC_2$ components (x= 0.8, 0.5) was investigated. The

powder mixtures related to $(Ti_{0.8}W_{0.2})_3SiC_2$ and $(Ti_{0.5}W_{0.5})_3SiC_2$ were ball milled for 15 hours, and then annealed in the furnace at 1250˚C for 2h. The XRD patterns of the ball milled powder mixtures showed in Fig. 6. As it could be seen, some W incorporated in Ti_3SiC_2 structure, and also the WC and Ti_xW_{1-x} phases formed during the process. It was found that the reflection peaks of $(Ti_x,W_{1-x})_3AlC_2$ shift to larger angles with the increment of W content (Fig. 7), which is attributed to the decrease of lattice parameters. It should be noted that the peak shift for x=0.8 and 0.5 are identical because a specific amount of W could be dissolved in the Ti_3SiC_2 phase and by increasing the W content, some other W containing phases such as WC or Ti_xW_{1-x} were formed.



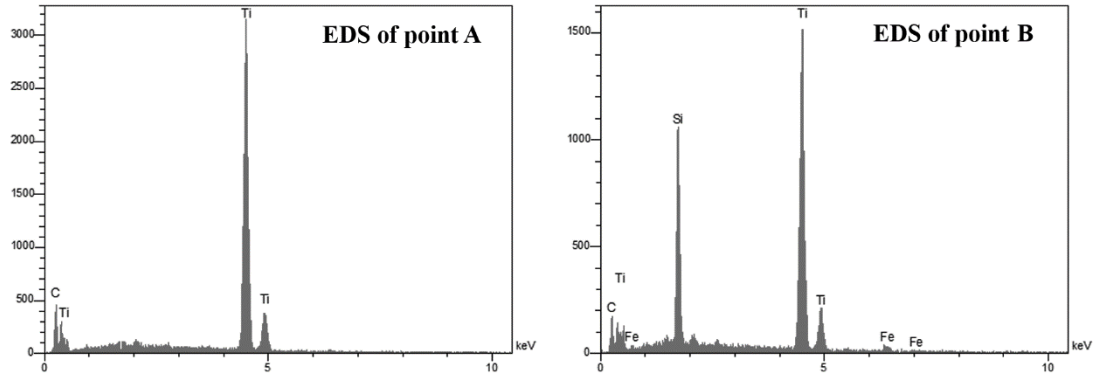


Fig. 5. SEM micrograph and EDS microanalysis of 15 hours ball milled powder mixture with 3Ti/1.2Si/2C ratio after annealing at 1250˚C.

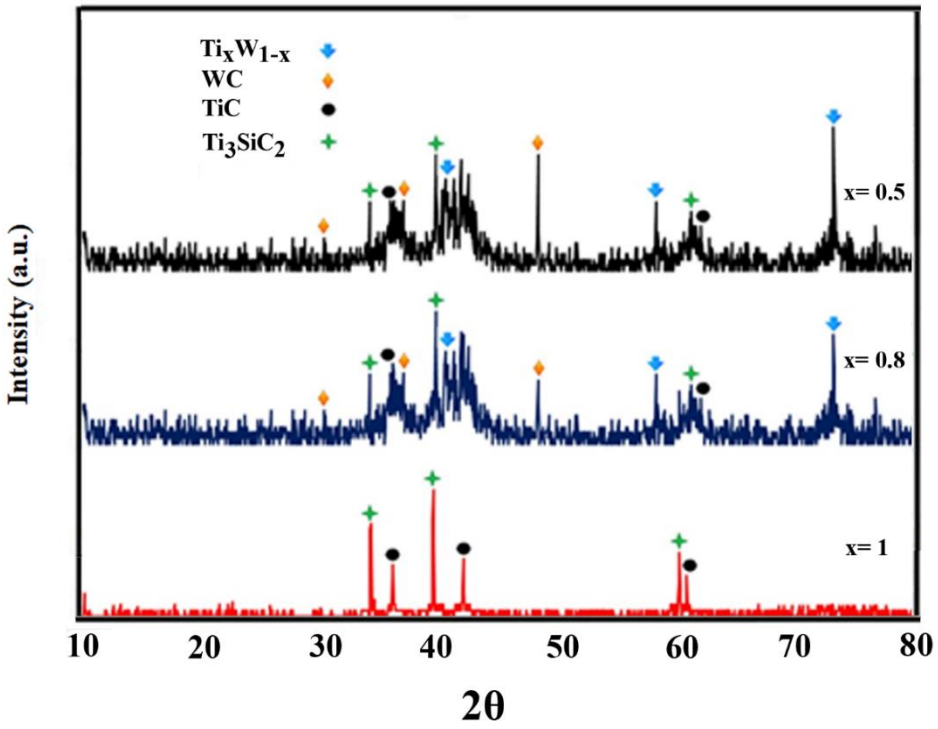


Fig. 6. XRD patterns of $(Ti_xW_{1-x})_3SiC_2$ component (x= 1, 0.8, 0.5) after 15 h ball milling.

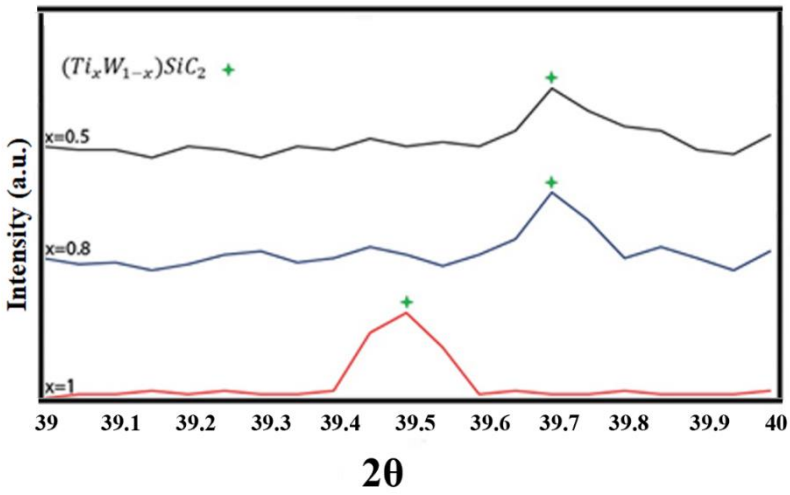


Fig. 7 The shift of the reflection peaks of $(Ti_x,W_{1-x})_3AlC_2$ to larger angles with the increment of W content.

Conclusion

This study has investigated the synthesis of $(Ti_{1-x}W_x)_3SiC_2$ MAX phase by high energy ball milling and the effect of heat treatment at temperatures of 600, 1000, 1250 and 1400˚C on synthesized powder were investigated. To this aim, mixtures of Ti, Si, and C were ball milled by a planetary ball milling for different milling times up to 15h. The results showed that after 15 hours ball milling the raw powders react together, which resulted in Ti_3SiC_2 and TiC formation. Heat treatment caused to react between the intermetallic compounds in Si-Ti system and TiC and led to increasing the purity of Ti_3SiC_2. In a separate run, a non-stoichiometric composition of Ti: Si:C= 3:1.2:2 was ball milled for 15 h, and heat treated at 1250˚C. The XRD results showed that the purity of the product is higher than the stoichiometric composition. The addition of W to Ti_3SiC_2 was

explored. In this regard, the synthesis of $(Ti_{1-x}W_x)_3SiC_2$ component (x= 0.8, 0.5) was investigated, and the results showed that some W incorporated in Ti_3SiC_2 structure and the WC and Ti_xW_{1-x} formed during the process, too.

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