The International Journal of Advanced Manufacturing Technology (2019) 104:1561–1570
https://doi.org/10.1007/s00170-019-04063-9

ORIGINAL ARTICLE

Designing sintering time for a TiSiC compound: a microwave and conventional comparison

Lucas D. Calado[1] · Giovana S. Padilha[1] · Wislei R. Osório[1,2] · Ausdinir D. Bortolozo[1,2]

Received: 3 April 2019 / Accepted: 20 June 2019 / Published online: 13 July 2019

Abstract
Ti_3SiC_2 has an important role on the resulting mechanical properties, which is based on the ceramic and metallic aspects. The microstructural array of the Ti_3SiC_2 and its corresponding hardness and mechanical behavior are analyzed. X-ray diffractometry (XRD), microstructural analysis using scanning electron microscopy (SEM-EDS), and densification using Archimedes' principle are used. It is found that the microwave (MW) processing provides higher hardness values and compressive strengths than the conventional (CN) sintered samples, i.e., ~ 8 GPa and ~ 240 MPa, respectively. Also, it is found that the compressive strength to density ratio clearly favors the sample produced using MW processing. This induces that a lightweight effect associated with an environmentally friendly aspect is also attained. It is induced that a Ti_3SiC_2 compound can be successfully produced by using a microwave treating when a conventional process is compared.

Keywords MAX phase · Microwave heating · Mechanical properties · Refine microstructural array

1 Introduction

Ti_3SiC_2 was firstly synthesized and characterized by Jeitschko et al. [1]. This compound has an important role on the resulting mechanical properties. Also, a great interesting is associated with the fact that an unusual combination of properties metals and ceramics can be attained [2, 3]. Despite the excellent properties presented by Ti_3SiC_2 compound, single-phase synthesis is still a challenge. A great number of manufacturing processes have been used, i.e., chemical vapor deposition (CVD) [4], magnetron sputtering (MS) [5], self-propagating high-temperature synthesis (SHS) [6] or self-propagating high-temperature synthesis-hot isostatic pressing (SHS-HIP) [7], arc-melting and post annealing [8], reactive sintering [9], solid-liquid reaction synthesis and simultaneous densification [10], hot isostatic pressing (HIP) [2], spark plasma sintering (SPS) and pulse discharge sintering (PDS) [11–13], pressureless sintering [14, 15], and mechanical alloying (MA) [16]. However, the single-phase preparation remains as an open question. As alternative, the microwave heating has been used to obtain density ceramics [16–22]. With this approach, both the micrometric and nanometric particles are possible to be obtained [18]. The heating by microwave energy is a process where the materials volumetrically absorb the electromagnetic energy and the heat is converted. This mechanism differs from those conventional methods when an electric energy or heated steam is transferred to materials by conduction, radiation, and convection mechanisms. The main reasons that have motivated the processing of ceramics through microwave energy are rapid heating, high densification rate [16], and low costs when compared with conventional heating [16–22]. Studies have also reported [17–20] a diffusion process increased, reduction of energy consumption, and considerable decrease in processing time and sintering temperature. Besides, better physical and mechanical properties, simplicity, unique properties, and low environmental risks are also attained [17–22].

Nowadays, the microwave sintering using the MAX phases has scarcely been explored [23–26]. In particular, the Ti_3SiC_2 phase has a low dielectric loss, which promotes the interaction of the microwaves with atoms of the material. Therefore, the electromagnetic energy can be converted into kinetic energy. The manufacture of the Ti_3SiC_2 compounds by using both

✉ Ausdinir D. Bortolozo
ausdinir.bortolozo@fca.unicamp.br

[1] School of Applied Sciences/FCA, Research Group in Manufacturing Advanced Materials, University of Campinas, UNICAMP, Campus II, Pedro Zaccaria, 1300, POB 1068, Limeira, SP, Brazil

[2] School of Technology, University of Campinas–UNICAMP, Campus I, Limeira, SP 13484-332, Brazil

grinding and microwave sintering was investigated by Wang et al. [23]. They have reported that of about 98% (in vol.) of the mentioned compound was attained. As also reported by Komarenko [26], Wang et al. [23] have argued that a drastic variation of temperature occurs due to the self-propagation process (SHS) is observed. This temperature variation promotes a silicon loss required to work with Si excess in order to a high purity of the examined samples be attained. Therefore, the authors have concluded that the reaction by SHS is not favorable for the Ti_3SiC_2 phase formation [24, 25]. Li et al. [24] have synthesized the Ti_3SiC_2 compound by using microwave heating. Distinctively from that previous study reported by Wang [23], a certain Al amount as the catalyst for the reaction was used [24]. Li et al. [24] have produced a high purity compound associated with a relative density higher than 90%. It is also demonstrated that the mechanical properties of the sintered compounds via microwave are higher than the HIP sintered. The results obtained in this work show that the Vickers hardness obtained by microwave sintering is similar to the hot press (HP) process, the flexural strength is 14% higher, and the modulus of elasticity is 3% slightly higher than that mentioned. Based on previous reported investigations, in this study, the aim is focused the influence of the resulting microstructural array on the mechanical properties of Ti_3SiC_2 manufacturing by powder metallurgy using microwave heating technology.

2 Experimental procedure

Commercial powders Ti of 99.4% (100 mesh), Si 99.59% purity (325 mesh), 99% purity C (325 mesh), and 99.8% purity Al were mixed in a stoichiometric ratio of 3Ti:1Si:1Al:2C in order to a Ti_3SiC_2 (TSC) composition sample be constituted. The powder mixture was compacted into a cylindrical steel die with 8 (± 0.5) mm in diameter and 2 (± 0.5) mm of height. A compaction pressure of 680 (± 50) MPa for 2 min was applied. The compacted specimens were sealed in a quartz ampoule under vacuum (10^{-2} mbar). The samples were heat treated at 1200 °C for 1 to 24 h by using both the conventional and microwave heat treatments (FMO1600; Fortelab). It is remarked that at least duplication was considered in order to guarantee the reproducibility of the proposed experimentations. A 2.45-GHz microwave frequency with maximum input power of 1.2 kW was applied. An X-ray diffraction (XRD) using an X'Pert Panalytical diffractometer in Bragg–Brentano geometry equipped with CuKα radiation and Ni filter was used. A full profile fitting procedure based on the Rietveld method [27], implemented in GSAS-EXPGUI software [28, 29] was employed to compare the crystallographic information such as phase abundances and lattice parameters. Scanning electron microcopy (SEM) images (using a JEOL JX 840A microscope) associated with the Energy-Dispersive X-ray Spectroscopy (EDS) spectra were obtained. Back-scattered electron (BSE) images were also obtained. The relative densities of the samples were obtained by using Archimedes' method and for this purpose, a specific gravity measurement kit SMK-401 from Shimadzu was used. Triplicate experimentations were also carried out to guarantee the reproducibility of the values attained. Vickers Hardness values of the samples were determined. For this purpose, the two indentation forces of 1.0 N and 9.8 N using dwell time of 10 s on the polished surface were used. A universal machine, model WDW-100E (Equilan®), using a strain rate ~ 2.5×10^{-4} s^{-1} and a speed cross-head of 0.25 mm/min was used in order to the experimental results of the compressive and yield strengths (0.2% proof stress) measurements be carried out. The compressive testing was carried out according to ASTM standard E 8 M/04. Triplicate experimentations were also considered when microhardness and compressive experimentations were carried out. Throughout the proposed manuscript, the corresponding averages are indicated.

3 Results and discussion

In order to produce the proposed Ti_3SiC_2 phase, a heat treatment at 1200 °C at 24 h was carried out. Figure 1 shows the diffractograms (XRD patterns) for both the conventional and microwave heat treatments. The analysis of these XRD plots predominantly demonstrates that the formations of the intermediate phases of $TiSi_2$ and TiC are reached. A small fraction of the Ti_3SiC_2 phase is observed. It is assumed that at this treating time, the phase undergoes a decomposition reaction [30, 31], as shown in Eq. 1:

$$Ti_3SiC_2 \rightarrow 3TiCx + Si(g) \quad (1)$$

It was previously reported by Rocault et al. [30] that a ternary phase treated under vacuum has involved the evolution of gaseous silicon with ΔG (1600 K) = 181 kJ/mol^{-1}. This seems to be associated with carbon morphology. It was used a crucible graphite, which provides a good crystallization condition (cooling rate). On the other hand, in this present investigation, graphite powder was utilized. It is speculated that an increased boundary of the graphite domains is attained, which promotes the gaseous silicon evolution.

Based on those obtained XRD patterns, it is induced that the heat treating during 24 h is not satisfactory. Then, it was decided to use a shorter heat treating period time. With this, a heat treatment during 2 h at the same temperature was carried out. Figure 2 shows the results of XRD plots, which evidences a majority of Ti_5Si_3 phase formation. It is possible to observe the TiC phase and graphite formation (shown as "*" inside of Fig. 2). This fact reinforces the hypothesis that an incomplete reaction is prevalent. This is different from the previously proceeded. This is because

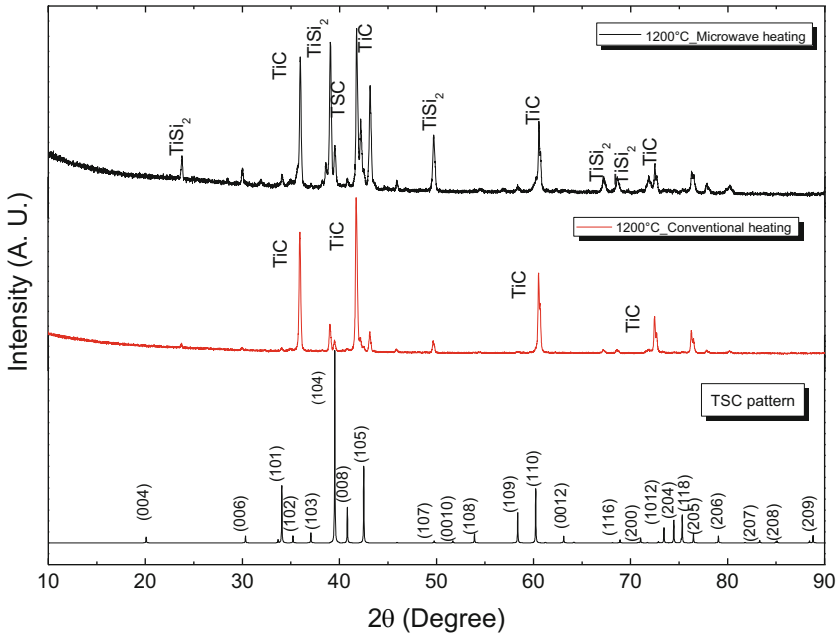

Fig. 1 XRD patterns of the samples heat treated at 1200 °C for 24 h using both microwave and conventional heating

at that moment, it is possible to identify precursor elements, whereas for the treatment of 24 h, intermediate phases were formed. By means of this result, it is suggested a kinetic order of phase formation, according to Eq. 2.

$$Ti_5Si_3 + TiC + C \rightarrow Ti_3SiC_2 \quad (2)$$

Based on a Ti-Si phase diagram, at 1200 °C, the Ti5Si3 phase is in equilibrium state with respect to Ti-α (dual-phase region). With this, it is induced that the way to the final transformation firstly intends to binary phase formation, and sequentially, the graphite diffusion occurs and the ternary is constituted. This fact is reasonably understood when the evolution of the ternary at the XRD pattern is observed. Besides, it was previously reported by Wu et al. [32] that the intermediate TiCx and Ti5Si3Cx phase formations in sintering reaction are involved. The obtained results in this present investigation are according with this aforementioned report [32].

It should also be considered that at a first stage in the reaction between Ti, Si, and C is the formation of titanium carbide. Upon the saturation of titanium in carbon, the remained titanium reacts with silicon in order to the titanium silicide be constituted. Further diffusion of titanium and carbon into Ti5Si3 causes a reconfiguration in the atomic ratio and the

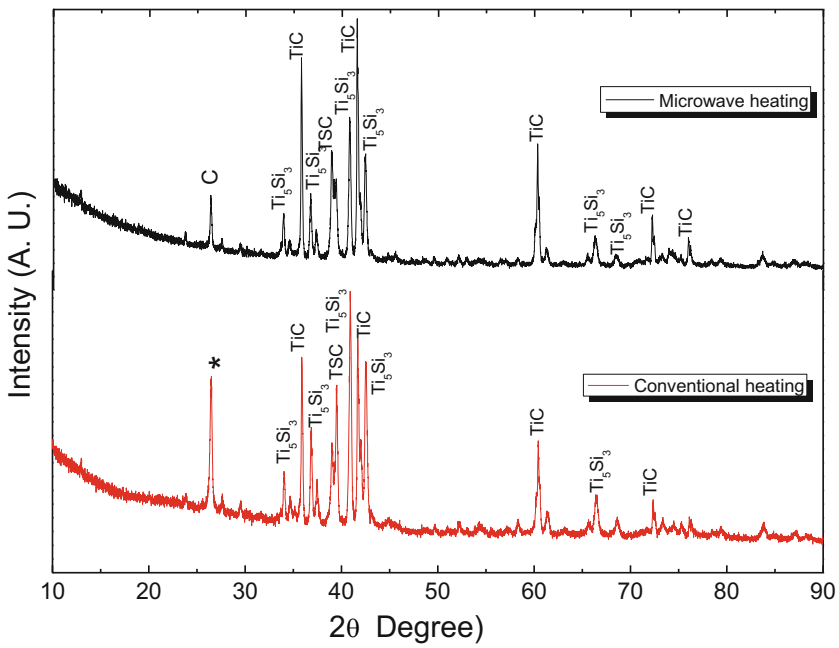

Fig. 2 XRD patterns of the heat-treated samples at 1200 °C for 2 h using both microwave and conventional heating

Ti3SiC2 phase is formed. This observation is similar to previously reported by Choi et a1. [33]. The reaction layers in the Ti-Sic composites treated at high temperatures revealed the presence of titanium carbide and the titanium silicide adjacent to a thin layer of the ternary phase.

Since the reaction was incomplete, a second heat treatment for 2 h was carried out. After this second treatment, it was determined that ~ 90% of the desired phase formation using the two methods is constituted, as shown in Fig. 3. It is worth noting that a remained amount of the TiC phase is still formed. Analyzing the two heating methods, it is evidenced that the microwave assisted sintering promotes a better quality of the material constituted, as also depicted in Fig. 3. Analyzing the peaks near to reflections $2\theta = 35°$, a small amount of the spurious phases is observed. In addition, the microwave sintering has narrower peaks, which suggests that a refinement in particle size is formed. Considering these aforementioned XRD results, it is noted that intermetallics have not been formed. This evidences that Al has not participated in none reaction to constitute TiAl, Ti_2Al, Ti_3Al, or $TiAl_3$.

Although a percentage of the phase higher than 90% was obtained, it was decided to carry out a third heat treatment. This is in order to verify if the increase of the TSC phase formation is consolidated, or if the selected time to promote the decomposition in binary species is enough. From this point, it is important to remark that a statistical planning/modeling was not provided. This is due to only the sintering time was experimented or modified. The sintering temperature was selected based on the Ti-Si-C phase diagram, which indicates that at 1200 °C, the desired Ti_3SiC_2 phase will be provided.

Based on aforementioned, a 3rd heat treatment was carried out at 1200 °C for a further 2 h, totaling 6 h of heat treatment. Also, it was not efficient, so it was disregarded. Thus, it was adopted that the subsequence samples would be produced only with two treatments to avoid such problems.

In order to improve those previously obtained results, an isothermal sintering was chosen. For the first heat treatment, it was adopted a heat rate of 10 °C/min up to 400 °C for 1 h. Sequentially, the temperature was raised at the same heating rate up to 550 °C, holding for a further 1 h and finally 1 h at 1200 °C. It is evidenced that by applying an isothermal heat treatment, the TSC phase is promoted. The second treatment was performed at 1200 °C for 2 h; the experimental XRD patterns concerning to the mentioned procedure is shown Fig. 3. With this procedure, of about 93% (in volume) of the desired Ti_3SiC_2 phase is formed. This is a good result when compared with literature, since this purity value is very close to those obtained by other studies, such as Barsoum et al. [1] and Racault et al. [30].

It is remembered that the powder metallurgy is one of the most economical and capable of producing parts with more complex geometries and high chemical quality control. For this result, the Rietveld refinement was carried out for the compound synthesized by using both the conventional and microwave heating, as shown in Fig. 4. The results obtained from the Rietveld analyses are demonstrated in Table 1. As it can be seen, the network parameters are in accordance with previously reported data [1–3]. Based on the convergence of the parameters χ^2 and Rwp (Table 1), it is induced that the refinement is stable. Additionally, a mass fraction higher than 94% of the proposed phase is attained.

Since a certain amount of the desired phase is attained, it is suggested that the processing route is defined. The corresponding results of the specific mass of the produced materials are

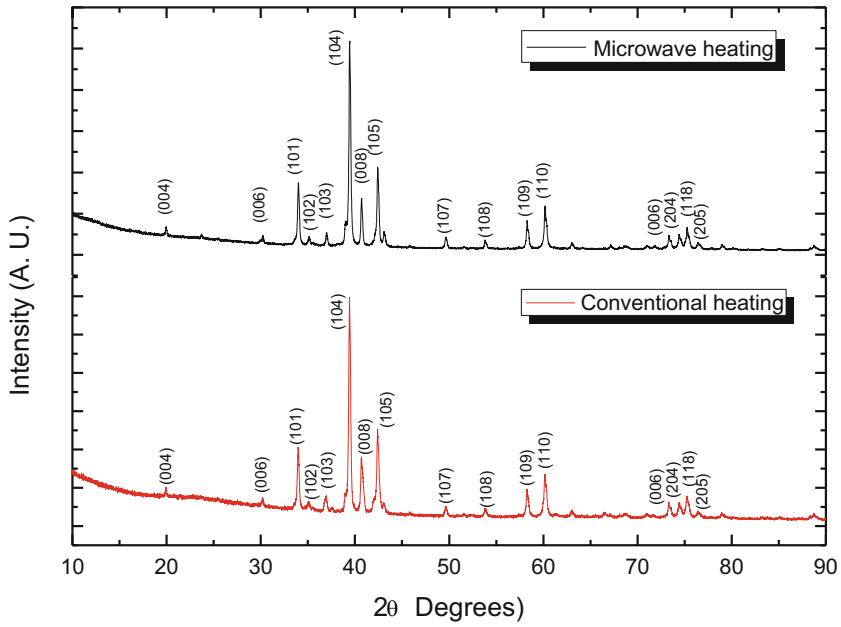

Fig. 3 XRD patterns of the heat-treated samples at 1200 °C for 2 added by 2 h using microwave and conventional heating

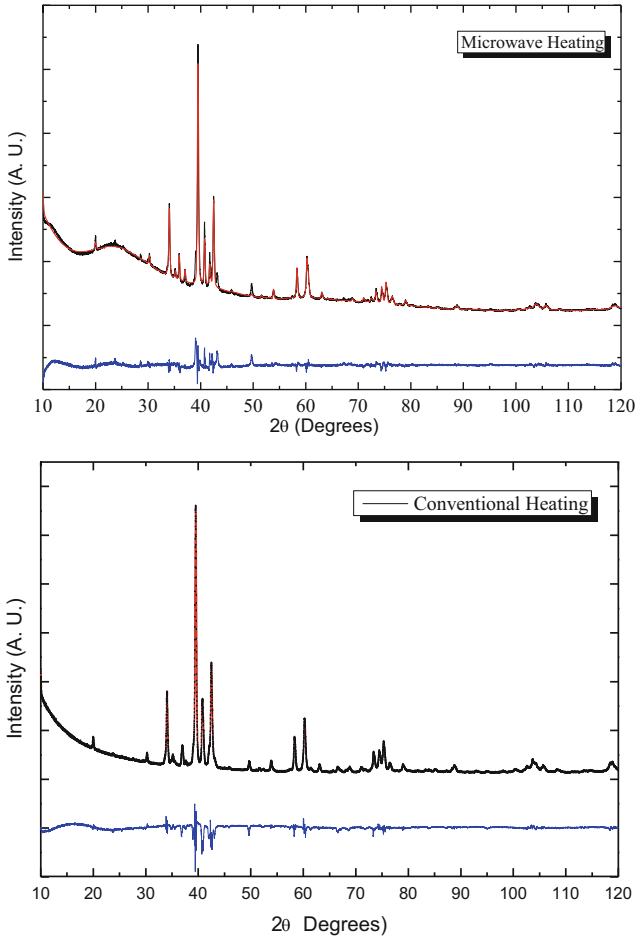

Fig. 4 Rietveld analyses of the heat-treated samples at 1200 °C for 2 added by 2 h using microwave and conventional heating

measured. The specific mass obtained for the green sample (only compacted) is of about 3.13 g/cm^3. After the heat treatments, the resulting densities of 3.47 (± 0.1) g/cm^3 and 4.154 (± 0.1) g/cm^3 are attained associated with the conventional heating and via microwave treating, respectively. A theoretical specific mass 4.51 g/cm^3 associated with the compound Ti_3SiC_2 is reported [2]. By means of the proposed two distinct routes (i.e., conventional and microwave), the resulting densification levels are 77% and 92%, respectively.

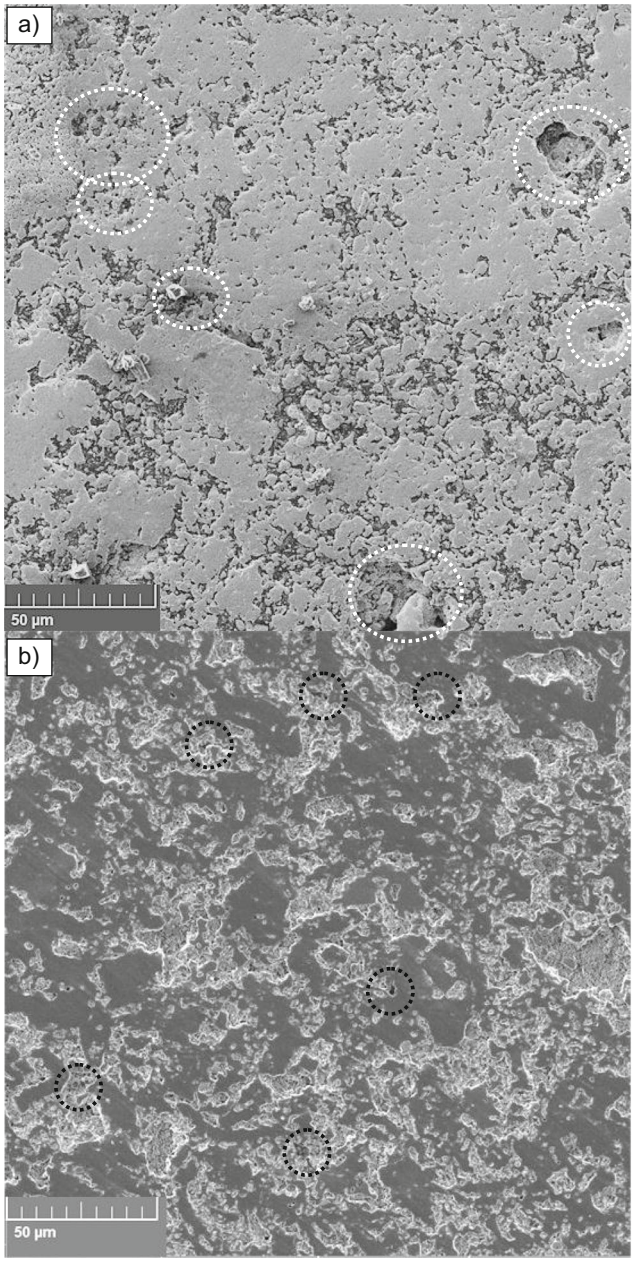

Fig. 5 Typical SEM micrographs of the heat-treated samples at 1200 °C for 2 added by 2 h using **a** microwave and **b** conventional heating

Table 1 Refinement for Rietveld parameters obtained of the Ti_3SiC_2 compound

Formula	Ti3SiC2 CN heating	Ti3SiC2 MW heating
Space group	$P6_3/mmc$ (194)	$P6_3/mmc$ (194)
Lattice constants (nm)	a = 0.3077	a = 0.30269
	c = 1.768	c = 1.768
Atomic positions Ti1 (4f)	(1/3, 2/3, 0.363)	(1/3, 2/3, 0.363)
Si (2b)	(0, 0, 1/4)	(0, 0, 1/4)
Ti2 (2a)	(0, 0, 0)	(0, 0, 0)
C (4f)	(1/3, 2/3, 0.926)	(1/3, 2/3, 0.926)
Theoretical density (g/cm^3)	4.505	4.505
Rwp	8.5%	7.97%
CHI**2	12.88	25.01
R(F**2)	0.1841	0.9059
Cycles	396	413
Ti_3SiC_2	94.8%	97%
TiC	4.6%	2.6%
TiO	0.6%	0.4%

When a potential application using this material is intended, for instance, the metallic foams, both pore size and distribution controlling, are required. Barsoum and El-Raghy [2] have obtained a densification of 99%, when HIP process is used. They have also reported that a densification of about 93% has also been attained when the microwave heating was used [2]. In other previous investigations, processes such as SP and HIP have demonstrated values for the resulting densifications of 98.9% and 98% respectively [7–16, 23]. However, these mentioned manufacturing processes provide a higher cost than that proposed in this present study.

Mechanisms or processing routes are required in order to increase the densification level since the remained porosity has a deleterious effect upon the resulting mechanical behavior. To confirm the results obtained in X-ray diffraction and densification analyses, scanning electron microscopy measurements were performed. From analysis of Fig. 5, it is evidenced that a compound sample with remained high porosity is prevalent. This corroborates with those densification analyses. In addition, the used manufacturing process routes promote formation of distinctive microstructural arrays. Figure 5 a and b shows typical resulting microstructural arrays provided by using both the conventional (CN) and microwave (MW) sintering,

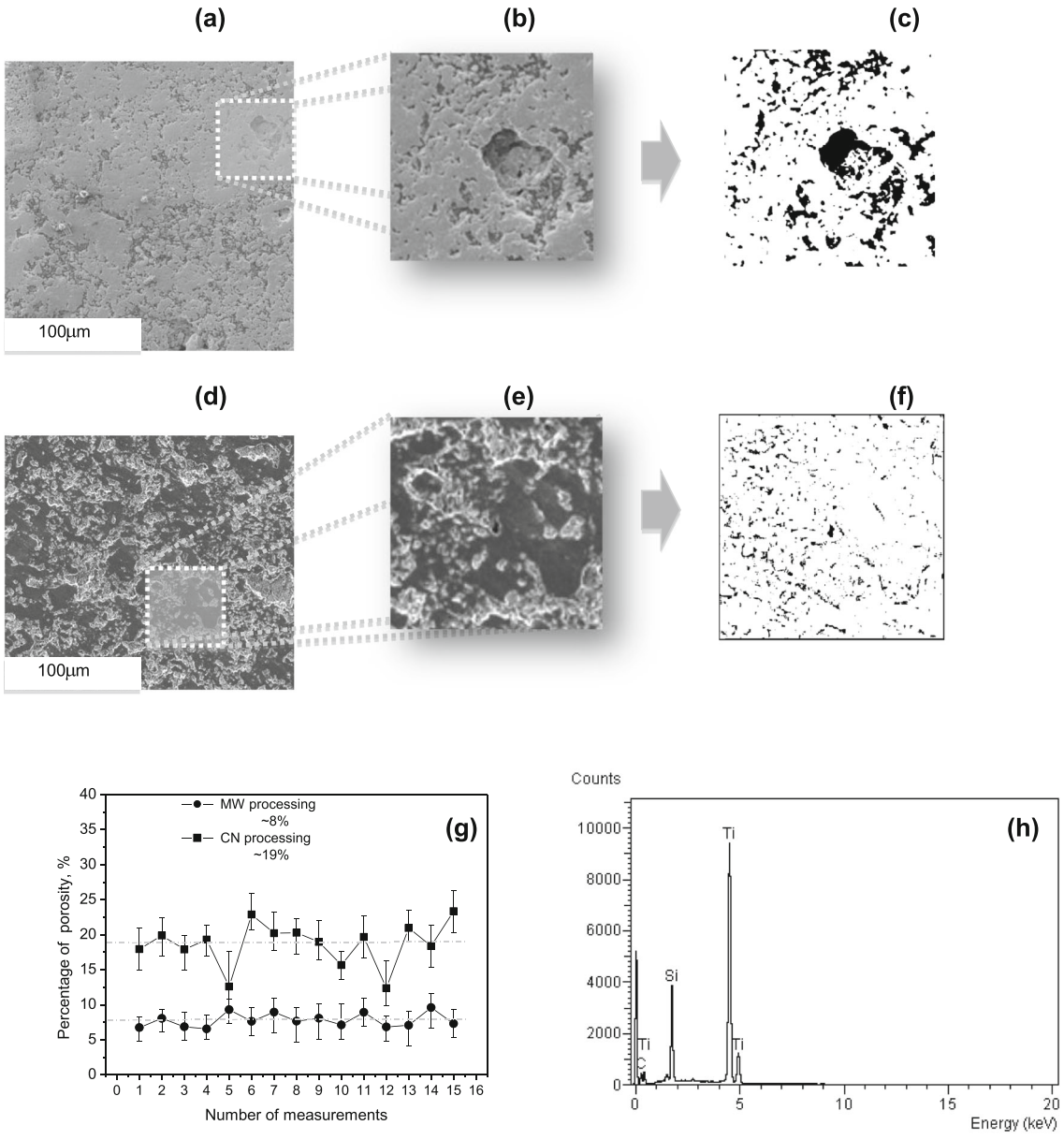

Fig. 6 SEM micrographs evidencing the porosity formation (a) and (d),; and considered regions containing substantial pores (b) and (e), and its corresponding binary images for the CN and MW sintering treatments (c) and (f), respectively. The average porous percentages (g) for both CN and MW sintering and (h) typical EDS analysis for the MW heat-treated sample

respectively. It can be observed that the CN sintering induces to a deeper (white doted-circle) porosity level than the MW sintering. It is remarked that equivocate interpretation of the resulting porous can be induced. Both the CN and MW heat treatments have provided similar surface textures. From those SEM micrographs, using ImageJ® software, contrast, brightness, and threshold are reworked and the binary images are produced, as shown in Fig. 6. Typical SEM images of the sample heat treated by using the CN and MW processes are shown in Fig. 6(a) and (d), respectively. Their corresponding regions characterizing deeper porosity are shown in Fig. 6(b) and (e), respectively. Figure 6(c) and (f) depicts the corresponding binary images. The average percentages of the porosity for each examined sample using CN and MW routes are shown in Fig. 6(g). An EDA results corresponding with the sample heat treated with the MW process is also demonstrated in Fig. 6(h).

Based on the resulting binary images (Fig. 6c and f), the corresponding average porous percentages of each one of the examined samples produced using the CN and MW sintering are determined. It is observed that the resulting porosity levels (i.e., ~ 18 and 8%) are similar to those results of the relative densifications obtained, i.e., ~ 77% and 92%, respectively. It is speculated that a higher relative densification is attained when the MW sintering is carried out. This seems to be intimately associated with a higher absorption. When the microwave sintered sample is analyzed, a slightly trend to refinement of the microstructure is characterized. As expected, the studies previously reported by Wang et al. [23] and Li et al. [24] demonstrate that the microwave treatment favors the formation of finer grains arrangement. This means that the smallest presence of pores and a denser microstructural array is attained.

Figure 7 evidences a typical SEM micrograph of a heat-treated MW sample with finer microstructural array formation with quasi fiber-like shape of the TiC (TC) phase embed into the matrix Ti-Si rich phase. Figure 7a depicts a fiber-like TiC phase longitudinally disposed, while a transversal section of this TiC phase is shown in Fig. 7b. The analysis of the dispersive energy spectroscopy (EDS) was also carried out in order to verify the formation of the TSC phase and impurities, as shown in Fig. 6(h). A typical EDS analysis of the samples treated using the microwave reveals an atomic ratio of 3Ti:1Si. The carbon element was not considered due to its low spectral energy, which leads to a considerable inaccuracy. In both spectra obtained by the EDS, the presence of the Al that was used as the additive in the starting material was not explicit. These obtained results are very close to that previously demonstrated for the Ti_3SiC_2 [24]. By means of the EDS analysis of the TiC phase was identified, in addition, regions with Si concentration were observed. Additionally, it is possible that some Si rich phase, such as Ti_5Si_3, is formed. Due to its low concentrate in the samples examined, this phase was not identified when the XRD analysis was carried out.

The microhardness values of the CN and MW sintering samples were obtained. Their corresponding analysis shows that the conventional sintering process is compatible with that hardness reported in the literature. For the distinctive types of processes such as HIP, HP, and HIP-SHS, the obtained hardness of about 4 (± 0.5) GPa is reached [33–39]. However, the microwave sintering positively has implied the resistance of the material to penetration. Thus, the hardness values obtained were two times higher than the conventional heat treatment, i.e., ~ 8 (± 0.5) GPa. These results confirm the expectation that the microwave heating promotes a particle refinement and, consequently, an improvement in the mechanical properties is also provided.

The experimental compressive strength (CS) results of both the MW and CN heat treatments are shown Fig. 8a. From analyzing the obtained data correlated with the MW processing evidences an UCS (ultimate compressive strength) of about three times higher (i.e., 241 ± 24 MPa) than the CN treating (75 ± 12 MPa). These results are agreed with the porous Ti_3SiC_2 samples also previously reported [36–45]. It is worth noting that the deformation has occurred without noticeable reduction in the cross-sectional area.

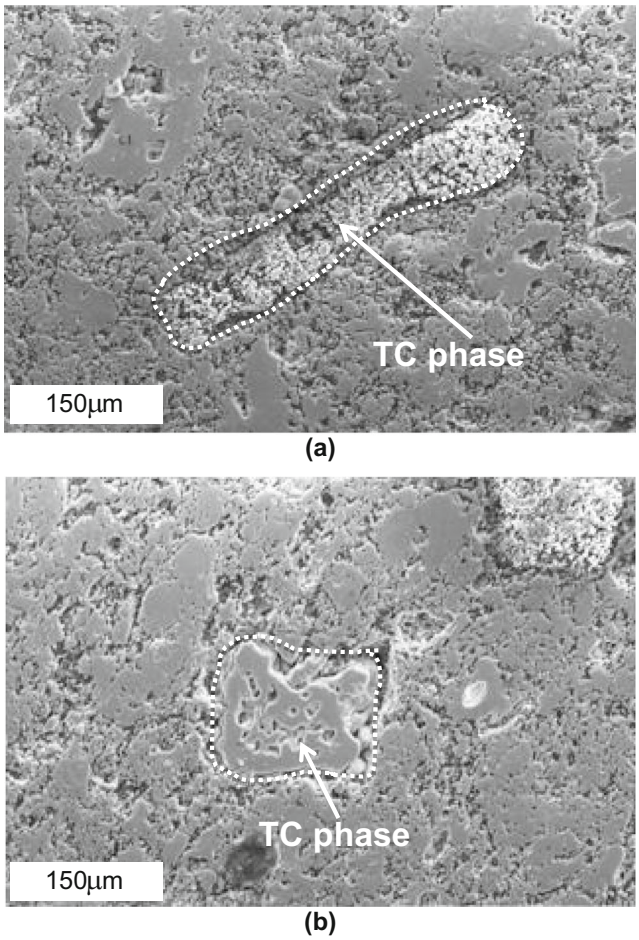

Fig. 7 Typical SEM micrograph showing **a** fiber-like TiC particle longitudinally disposed, **b** transversally sectioned of a sample heat treated by MW process

Furthermore, it is noticed that the formation of shear bands, inclined at 45° to the loading axis [38–40] are attained. This is a typical response of a MAX material. Additionally, it seems that the yield strengths (YS) are ~ 25 (± 5) MPa and ~ 150 (± 18) MPa for the heat-treated samples using the CN and MW processing, respectively. Based on these aforementioned results, the MW method proved to be more efficient than the CN in terms of the mechanical properties, which seems to be intimately associated with the sintered densification levels and microstructural array refinement provided. Although other properties (e.g., flexural strength) were not explored, it is speculated that the microwave treating provides a particle refinement. With this, an improvement in the mechanical properties is also attained. The compressive strength result indicates this influence provided by microwave process and an UCS ~ 240 MPa is reached, while the conventional heat treating attains only of about 75 MPa. Based on this assertion, it is assessed that the mechanical behavior improvement is due to a combination of factors. In fact, the microstructural refining acts as barrier for the dislocation of defects, and consequently, an increase in mechanical strength is promoted.

Figure 8 b and c shows the correlation between the relative sintered densification and the results for both the UCS (ultimate compressive strength) and YS for the examined samples treated by using both the CN and MW treatments. It is clarified that the examined mechanical behavior is more susceptible to the porous distribution level than the microhardness. This seems to be associated with the fact that the penetration of the indentator is not sufficiently deeper in order to engage a reasonable porous quantity to indicate a deleterious effect on the results of microhardness examined. On the other hand, when the compressive strength is evaluated, a great number of both the surface and internal porous are taken in account. Based on this, it is confirmed that the samples with higher porosity levels than other ones have consequently lower sintered densifications and lower mechanical parameters are provided.

Another analysis that provides interestingly results is the evaluation of the UCS-to-density relation, commonly designated as the specific strength (SS). This analysis provides a concatenate investigation between the effective strength and the lightweight effect provided by each of the samples examined. When a certain range of UCS value is considered, the MW processing indicates a range of the SS results between 53

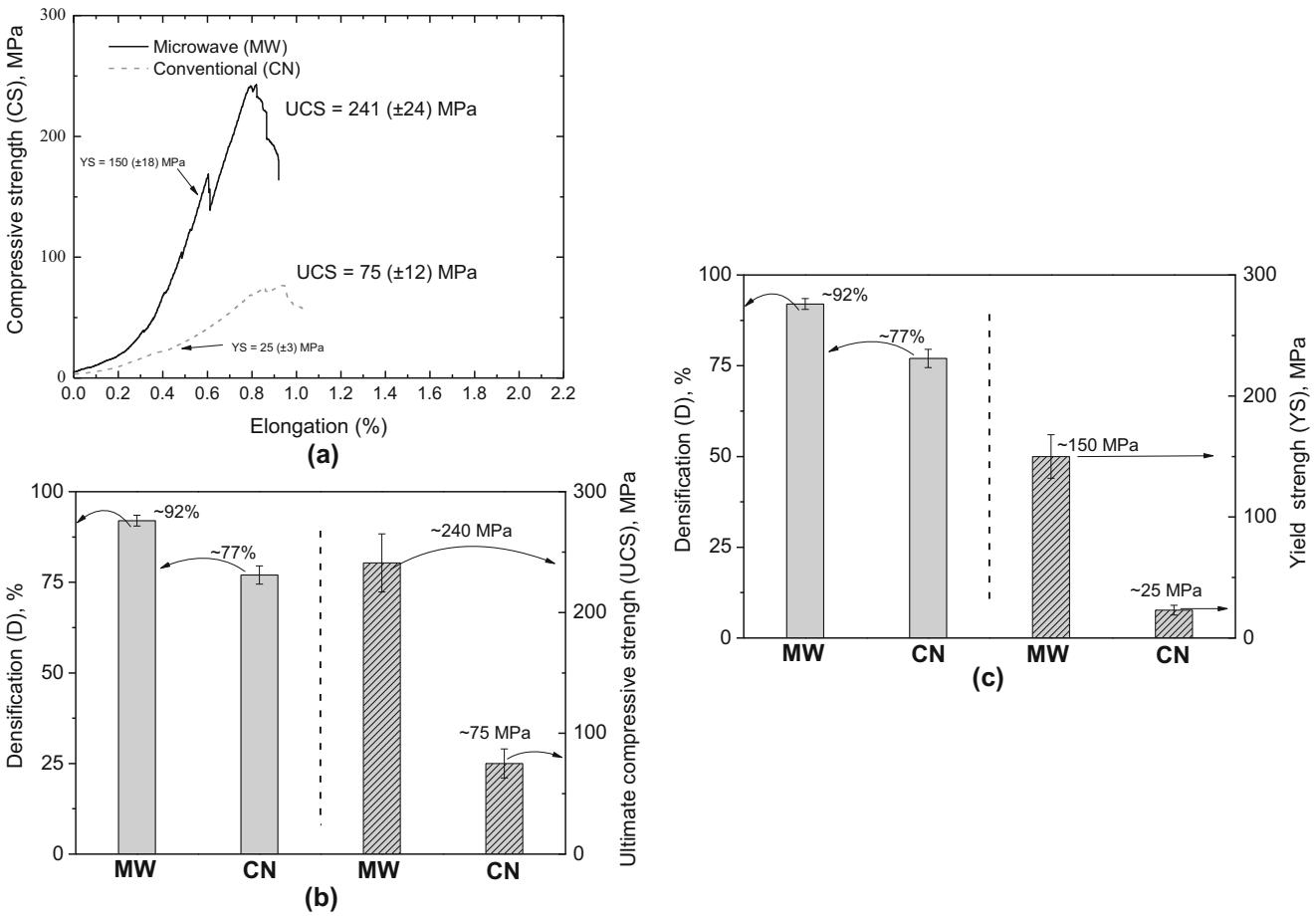

Fig. 8 **a** Typical engineering stress-strain curves obtained in TSC sample and **b**, **c** densification and UCS and YS as a function of the conventional (CN) and microwave (MW) heat treatments

and 63×10^3 m^2 s^{-2}, while the CN processing provides a range between 19 and 25×10^3 m^2 s^{-2}. This means that the mechanical strength concatenated with the lightweight effect clearly favors the heat-treated MW samples. Besides, an environmentally friendly aspect can also be attained when other manufacturing processes are compared. This is based on the fact that a lower energy level and simple construction and/or equipment are taken in account.

4 Conclusion

Powder metallurgy is used to Ti_3SiC_2 samples be sintered using both the microwave and conventional heat treatments. Based on the experimental observations and analysis, the following conclusions can be drawn:

1. There is a viability to apply the microwave heating in order to the Ti_3SiC_2 phase be produced. A volume fraction of about 90% can be attained;
2. The microhardness results have revealed presented values of the order of 8.2 GPa when the microwave processing is used, which is ~ 2× higher than the conventional (4.2 GPa); when compressive strength are considered, the microwave sintering shows values ~ 3× higher than the conventional processing; and
3. The attained results induces that the microwave treatment is suitable for the Ti_3SiC_2 production.

Funding information This work was supported by the FAPESP (Grant 16/13352-0).

References

1. Jeitschko W, Nowotny H (1967) Die Kristallstruktur von Ti_3SiC_2 - ein neuer Komplexearbid-Typ. Monatsh Chem 98(2):329–337
2. Barsoum MW, El-Raghy T (1996) Synthesis and characterization of a remarkable ceramic: Ti3SiC2. J Am Ceram Soc 79(7):1953–1956
3. Okano T, Iseki T, Yano T (1995) Synthesis and high temperature mechanical properties of Ti3SiC2/SiC composite. J Mater Sci 30:3087–3090
4. Goto T, Hirai T (1987) Chemically vapor deposited Ti_3SiC_2. Mater Res Bull 22(9):1195–1201
5. Palmquist J-P, Jansson U (2002) Magnetron sputtered epitaxial single-phase Ti3SiC2 thin films. Appl Phys Lett 81:835–837
6. Riley DP, Kisi EH, Wu E, Mccallum A (2003) Self-propagating high-temperature synthesis of Ti3SiC2 from 3Ti+SiC+C reactants. J Mater Sci Lett 22:1101–1104
7. Lis J, Miyamoto Y, Pampuch R, Tanihata K (1995) Ti_3SiC-based materials prepared by HIP-SHS techniques. Mater Lett 22(4):163–168
8. Arunajatesan S, Carim AH (1995) Synthesis of titanium silicon carbide. J Am Ceram Soc 78:667–672
9. Li JT, Miyamoto Y (1999) Fabrication of monolithic Ti_3SiC_2 ceramic through reactive sintering of Ti/Si/2TiC. J Mater Synth Process 7:91–96
10. Zhou Y, Sun Z, Chen S, Zhang Y (1998) In-situ hot pressing/solid-liquid reaction synthesis of dense titanium silicon carbide bulk ceramics. Mater Res Innov 2:142–146
11. Zhou W, Mei B, Zhu J, Hong X (2005) Synthesis of high-purity Ti_3SiC_2 and Ti_3AlC_2 by spark plasma sintering (SPS) technique. J Mater Sci 40(8):2099–2100
12. Zhang ZF, Sun ZM, Hashimoto H, Abe T (2002) Application of pulse discharge sintering (PDS) technique to rapid synthesis of Ti_3SiC_2 from Ti/Si/C powders. J Eur Ceram Soc 22(16):2957–2961
13. Lagos MA, Pellegrini C, Agote I, Azurmendi N, Barcena J, Parco M, Silvestroni L, Zoli L, Sciti D (2019) Ti3SiC2-Cf composites by spark plasma sintering: processing, microstructure and thermo-mechanical properties. J Eur Ceram Soc 39:2824–2830
14. Li JF, Matsuki T, Watanabe R (2003) Fabrication of highly dense Ti_3SiC_2 ceramics by pressureless sintering of mechanically alloyed elemental powders. J Mater Sci 38:2661–2666
15. Zhou C, Wu X, Ngai TL, Li L, Ngai S, Chen Z (2018) Al alloy/Ti_3SiC_2 composites fabricated by pressureless infiltration with melt-spun Al alloy ribbons. Ceram Int 44:6026–6032
16. Li S-B, Zhai H-X (2005) Synthesis and reaction mechanism of Ti_3SiC_2 by mechanical alloying of elemental Ti, Si, and C powders. J Am Ceram Soc 88(8):2092–2098
17. Oghbaei M, Mirzaee O (2010) Microwave versus conventional sintering: a review of fundamentals, advantages and applications. J Alloys Compd 494:175–189
18. Asadian K, Ebadzadeh T (2013) Synthesis of Nanosized $Ba(Zn_{1/3}Nb_{2/3})O_3$ Using microwave heating process. J Mater Eng Perform 22:898–902
19. Kirill I, Rybakov EA, Olevsky E, Krikun V (2013) Microwave sintering: fundamentals and modeling. J Am Ceram Soc 96(4):1003–1020
20. Venkateswarlu K, Saurabh S, Rajinikanth V et al (2010) Synthesis of TiN reinforced aluminium metal matrix composites through microwave sintering. J Mater Eng Perform 19:231–236
21. Chandrasekaran S, Ramanathan S, Basak T (2011) Microwave material processing—a review. J Rev 58(2):330–363
22. Wu H, Chen F, Xu J (2017) Preparation and Characterization of (Mo, W)Si2-SiC Composites by In Situ Microwave Reaction Sintering. J Mater Eng Perform 26:3239–3244
23. Wang C, Hu S, Cai Y, Sakka S, Grasso Q, Huang (2014) Synthesis of high-purity Ti_3SiC_2 by microwave sintering. Int J Appl Ceram Technol 11(5):911–918
24. Li F, Zhang H, Wang Q, Qu D, Zhou T, Kim B, Sakka Y, Hu C, Huang Q (2014) Microwave sintering of $Ti_3Si(Al)C_2$ ceramic. J Am Ceram Soc 97(9):2731–2735
25. Liu W, Qiu C, Zhou J, Ding Z, Zhou X, Du S, Han Y-H, Huang Q (2015) Fabrication of Ti 2 AlN ceramics with orientation growth behavior by the microwave sintering method. J Eur Ceram Soc 35:1385–1391
26. Komarenko P, Clark DE (2009) Synthesis of Ti3SiC2-based materials using microwave-initiated SHS. Ceram Eng Sci Proc 15:1028–1035
27. Rietveld HM (1967) Line profiles of neutron powder-diffraction peaks for structure refinement. Acta Crystallogr 22(1):151–152
28. Larson AC, Von Dreele RB (1994) General Structure Analysis System (GSAS), Los Alamos National Laboratory Report LAUR, pp 86–748
29. Toby BH (2001) EXPGUI, a graphical user interface for GSAS. J Appl Crystallogr 34:210–213
30. Racault C, Langlais F, Naslain R (1994) Solid-state synthesis and characterization of the ternary phase Ti3SiC2. J Mater Sci 29:3384–3392
31. Li X, Xu L, Chen QN, Cao X, Liu L, Wang Y, Zhang H, Meng C, Wu Q (2018) Investigation of formation mechanism of Ti3SiC2 by high pressure and high-temperature synthesis. High Pressure Res 38:440–447

32. Wu E, Riley DP, Kisi EH, Smith RI (2005) Reaction kinetics in Ti3SiC2 synthesis studied by time-resolved neutron diffraction. J Eur Ceram Soc 25:3503–3508
33. Choi SK, Chandrasekaran M, Brabers MJ (1990) Interaction between titanium and SiC. J Mater Sci 25:1957–1964
34. Li B, Cheng L-F, Zhang L-T (2002) Identification of damage tolerance of Ti3SiC2 By hardness indentations and single edge notched beam test. Mater Sci Technol 18:231–233
35. El-Raghy T, Zavaliangos A, Barsoum MW, Kalidindi SR (1997) Damage mechanisms around hardness indentations in TiSiC. J Am Ceram Soc 80:513–516
36. Fraczkiewicz M, Zhou AG, Barsoum MW (2006) Mechanical damping in porous Ti3SiC2. Acta Mater 54:5261–5270
37. Radovic M, Barsoum MW, El-Raghy T, Wiederhorn SM, Luecke WE (2002) Effect of temperature, strain rate and grain size on the mechanical response of Ti_3SiC_2 in tension. Acta Mater 50:1297–1306
38. Radovic M, Barsoum MW, El-Raghy T, Seidensticker J (2000) Tensile properties of Ti_3SiC_2 in the 25–1300°C temperature range. Acta Mater 48:453–459
39. Pechkovskii EP, Firstov SA (2003) Structure and mechanical properties of porous titanosilicon carbide Ti_3SiC_2. Powder Metall Metal Ceram 42:424
40. Li J-F, Pan W, Sato F, Watanabe R (2001) Mechanical properties of polycrystalline Ti_3SiC_2 at ambient and elevated temperatures. Acta Mater 49(6):937–945
41. Sun ZM, Murugaiah A, Zhen T, Zhou A, Barsoum MW (2005) Microstructure and mechanical properties of porous Ti_3SiC_2. Acta Mater 53(16):4359–4366
42. Velasco B, Gordo E, Hu L, Radovic M, Tsipas SA (2018) Influence of porosity on elastic properties of Ti_2AlC and Ti_3SiC_2 MAX phase foams. J Alloys Compd 764:24–35
43. Yue Z, Yang L, Gong J, Gao J (2018) Synthesis and comparison of Two cBN composites with starting ternary carbide binders. J Mater Eng Perform 27:2124–2130
44. Gao H, Luo F, Nan H, Wen Q, Qing Y, Wang C, Zhou W, Zhu D (2019) Improved mechanical and microwave absorption properties of SiC fiber/mullite matrix composite using hybrid SiC/Ti_3SiC_2 fillers. J Alloys Compd 791:51–59
45. Wang Z, Zhang H, Liu X, Jiang Y, Gao H, He Y (2018) Reactive synthesis of porous nanolaminate Ti3(Si,Al)C2 intermetallic compound. Mater Chem Phys 208:85–90