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Thermal stability of n-TiB_2 reinforced α-Al_2O_3/SiC based nanocomposite sintered at 1600˚C using TG/DTA under controlled Ar atmosphere

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ABSTRACT

Innovation in material science progresses the usage of Al_2O_3/SiC based nanocomposites in gas turbine engine components, the harp-shaped structure of hypersonic rocket engine cutting tools for Ni, Al alloys, and clutch plate for two-wheelers. Thermal stability is one of the significant properties of gas turbine and rocket engine materials. Future engines may have to operate at very high temperature that may require high thermally stable material. In this research, an attempt is made to enhance the thermal stability of the Al_2O_3/SiC based nanocomposite by reinforcing 5-20 Vol. % nano Titanium Boride. Fabrication of α-Al_2O_3/SiC with 5-20 Vol. % n-TiB_2 was carried out through pressureless sintering at 1600˚C followed by cold compaction. The fabrication process was carried out at a controlled Ar atmosphere. Thermal stability of the sintered samples was analyzed by NETZSCH STA 449F3 thermogravimetric analyzer with a heating rate of 10˚C/min and compared with Al_2O_3/SiC. The composite α-Al_2O_3/SiC/(5-20 Vol. %) n-TiB_2 showed good thermal stability up to 1488˚C with 6% less mass change than Al_2O_3/SiC. The addition of n-TiB_2 enhanced the collaboration between the atoms and postponed the decomposition temperature. The microstructure of the 20 vol % n-TiB_2 added α-Al_2O_3/SiC was captured by 20 kV JSM-5600J Scanning Electron Microscopy and confirmed the presence of n-TiB_2. Also, the presence of Ti, Si, Al, O, and B in the nanocomposite was confirmed by energy dispersive analysis of X-beams (EDS).

Keywords: Al_2O_3/SiC; Nanocomposite; Sintering; Thermal stability; Titanium boride.

INTRODUCTION

Aluminum oxide and silicon carbide based composites have potential applications in many engineering fields (Niihara and Nakahira 1988, Niihara, Nakahira et al. 1989, Niihara and Nakahira 1990, NAKAHIRA and NIIHARA 1992, Stearns, Zhao et al. 1992, 中平敦 and 新原晧一 1992, Zhao, Stearns et al. 1993, Kim and Moorhead 1994, Borsa, Jiao et al. 1995, Thompson, Chan et al. 1995, Chou, Chan et al. 1996, Jeong and Niihara 1997, Wu, Lawrence et al. 1998, Abovyan, Nersisyan et al. 2001, Tamilselvam, Varsha et al. 2019). Especially, innovation in the material science progresses the use of Al_2O_3/SiC based materials in cutting tools for Ni, Al alloys (Jianxin, Lili et al. 2005)

(AM, Kaleemulla et al. 2019), armor, and NIJ ballistic protection equipment (Zawrah and Aly 2006), rocket engine nozzle throat (Samanta, Dhargupta et al. 2001), surface tile of the space shuttle (Samanta, Dhargupta et al. 2001), piston crown and cylinder of IC engine (Jeong and Niihara 1997), the nose cone of the hypersonic reentry vehicle, and the harp-shaped structure of hypersonic rocket engine. Thermal stability is one of the significant properties of these applications. Aeronautical and aerospace vehicle's hotspot components necessitate operating at a very high-temperature environment that requires high temperature and thermally stable materials. As the innovations lift the technology to the next era, the Future aerospace vehicles might have to operate at very high temperature that may require high temperature materials. Therefore, the hotspot components of these vehicles

made up of Al_2O_3 & SiC based materials need to be replaced with new material, which has enhanced thermal stability. Reinforcement of metal borides and carbides change the thermal stability unthinkably because of their ultra-high temperature behavior. In this research, an attempt is made to enhance the thermal stability of the Al_2O_3/SiC based nanocomposite by reinforcing 5-20 Vol. % nano Titanium Boride.

Metal boride-based ceramics are called structural ceramics, because these metal borides have higher structural integrity at elevated temperature. Structural ceramics possess a very high melting point and higher thermal withstanding capability. Titanium boride is one of the structural ceramics having a high melting point about 3225ºC, high oxidation resistance, and resistant to mechanical destruction (Carbide 1997, Munro 2000, Basu, Raju et al. 2006, Cahn and Hassen 2006, Murthy, Basu et al. 2006, Schmidt, Boehling et al. 2007). Titanium boride is a prospective candidate for many engineering applications, where enormous temperature encompasses because it unveils sturdy covalent bonds at elevated temperatures(Sackheim 2006). It also called ultra-high temperature ceramic (UHTC) because of its excellent properties like high thermal expansion coefficient, high melting point, high hardness, good electrical and thermal conductivity, and importantly, excellent chemical stability (Levine, Opila et al. 2002, Gasch, Ellerby et al. 2005, Basu, Raju et al. 2006, Johnson 2011).

Titanium boride reinforced composites are possessing enhanced mechanical, thermal, and tribological properties. Investigations on titanium boride based materials are reported by many researchers (Anal, Bandyopadhyay et al. 2006, Murthy, Basu et al. 2006, Raju, Mukhopadhyay et al. 2009, Ramesh, Ahamed et al. 2010, Suresh, Shenbag et al. 2012, Yu. Popov, Sivak et al. 2015, Chen, Kang et al. 2016, Alizadeh, Geraei et al. 2018). Titanium boride addition augmented the hardness, wear resistance, and high-temperature stability of the Fe matrix based composite due to the solid solution strengthening of iron by aluminum and silicon. Even at higher loading, the composite has shown no particle pullout. It designates the excellent bonding between the Fe matrix and TiB_2 particles. The high specific modulus of TiB_2 enhanced the load-bearing capacity and bonding capability (Anal, Bandyopadhyay et al. 2006), which helped improve the steel's tensile strength (Murthy, Basu et al. 2006, Raju, Mukhopadhyay et al. 2009). During the fabrication processes like reactive hot pressing, the TiB_2 reacted with Al and B_2O_3 exothermically and produced efficient boding between TiB_2 and Al_2O_3. Such exothermic reaction enhanced the fracture toughness of the composite greatly (Yu. Popov, Sivak et al. 2015). Further, Al-TiB_2-Al_2O_3 composite material fabricated through the in situ process clacked good microstructure and strength at elevated temperature (Alizadeh, Geraei et al. 2018). TiB_2 reinforcements also improvise the Al 6063 metal matrix composite (Ramesh, Ahamed et al. 2010, Suresh, Shenbag et al. 2012). In addition, particulate reinforcement of TiB_2 upsurges the material strength by increasing the quality of the alloy matrix and interaction between the matrix and hard particles (Raju, Mukhopadhyay et al. 2009, Chen, Kang et al. 2016).

A number of researchers witnessed that the high-temperature behavior of materials enhanced by Titanium Boride reinforcement (Dallaire and Champagne 1987, Ma and Tjong 2000, Telle, Sigl et al. 2000, Wang, Shun et al. 2006, Raju and Basu 2009, Ziemnicka-Sylwester 2013, Nallusamy and S 2020). TiB_2 based ceramic composites fabricated through self-propagating high-temperature synthesis displayed greater hardness and good microstructure homogeneity at elevated temperature (Ziemnicka-Sylwester 2013). On the other hand, Titanium boride reinforcement

increases the hardness and flexural strength of $TiSi_2$ up to 1200ºC (Raju and Basu 2009). Furthermore, Ti's lattice diffusion on Cu increased the high temperature creep resistance of in situ processed TiB_2/Cu (Ma and Tjong 2000). Out of these researches, none of the research articles discussed the thermal stability of TiB_2 reinforced Al_2O_3/SiC particulate composite. In this research, the high-temperature thermal stability of TiB_2 reinforced Al_2O_3-SiC composite discussed using Thermogravimetric analysis. The difference in mass percentage for various temperatures with a standard heating rate was recorded and presented.

MANUFACTURING OF COMPOSITE AND EXPERIMENTAL PROCEDURE

The nanopowders of Al_2O_3, SiC and TiB_2 of average particle size 90-100 nm procured and processed directly without any preprocessing. The essential properties of the procured raw materials are displayed in **Table 1.**

Table 1. Important Properties of raw materials.

Name of the material	Titanium Diboride	Silicon Carbide	Aluminum Oxide
Chemical formula	TiB_2	SiC	α-Al_2O_3
Density	4.52 g/cm^3	3.21 g/cm^3	3.986 g/cm^3
Melting point	3230 ºC	2730ºC	2,072°C
Molar mass	69.489 g/mol	40.11 g/mol	101.96 g/mol
Average Particle size	90-100 nm	90-100 nm	90-100 nm

Sample S_1 is prepared by mixing 95 vol% of α-Al_2O_3 (99.5% purity)-SiC(99% purity) and 5 vol% of n-TiB_2 (99.5% purity) in a planetary ball milling machine for a cycle of 60 minutes at 250 rpm. The ball milling jaw was cleaned with acetone before starting the process. The jaw has 25 Zirconium balls for sizing and mixing. The remaining samples S_2, S_3, S_4 have also processed through the same ball milling machine as per the stoichiometric ratio mentioned in **Table 2.** Then, the processed samples were pelletized to the size of 10 mm diameter and 10 mm height by the uniaxial hydraulic press with a 5-tonne load. For pelletizing, a die made of piston metal was used. After pelletizing, each pellet is placed inside the tubular furnace for pressureless sintering. A crucible made of alumina (Al_2O_3) was used as a sintering tray. The sintering process was carried out as per the details mentioned in

Table 3. Initially, for the first two hours, the furnace's temperature was raised to 30°C using 25 amps current. Then the temperature was raised to 1200ºC using 45 amps current in the next two hours. At the end of the 6th hour, the furnace's temperature was 1600ºC, and it was achieved by passing 65 amps current. During the seventh and eight hours, the pellets were allowed to soak at 1600ºC. Then all the pellets were cooled at room temperature for 6 hours in vacuum with argon gas purging(Rabiezadeh, Hadian et al. 2014). After cooling, all the pellets' thermal stability was carried out using NETZSCH STA 449F3 Thermogravimetric analyzer. The NETZSCH STA 449F3 TG/DTA analyzer's testing chamber was blushed with pure argon gas before starting the analysis. The thermal stability analysis was carried out from room temperature to 1500°C by maintaining 10°C/min as heating rate throughout the investigation. The response of the material in terms of thermal degradation was noticed with respect to the temperature rise. Additionally, Xpert-Pro Diffractometer characterized the S4 with Cu Kα radiation (XRD). The micrographs of S4 was recorded using 20 kV JSM-5600J Scanning Electron Microscopy equipped with Energy dispersive X-Ray spectroscopy (EDS) for further analysis.

Table 2. Volume percentage and initial weight of TiB_2 in $TiB_2/Al_2O_3/SiC$ composite.

Sample name	TiB2 (Vol. %)	Initial mass of the samples (g)
S_1	5	10.21
S_2	10	10.30
S_3	15	10.52
S_4	20	10.85

Table 3. Processing duration, heating rate and temperature rise during muffle furnace sintering.

Processing duration	Current supplied (Amps)	Temperature rise (°C)	Heating rate (°C/min)
1^{st} -2^{nd} hours	25	30°C	10 °C/min
3^{rd} -4^{th} hours	45	1200°C	
5^{th} -6^{th} hour	65	1600°C	
6^{th}- 12^{th} hour	NA	Slow cooling	Room temperature cooling

RESULT AND DISCUSSION

Thermogravimetric Analysis

Thermogravimetric analysis (TGA) is one of the scientific methods used to understand thermal behavior. In this analysis, the material's mass or weight percentage changes have been examined by raising the temperature to the material's elevated temperature. For composites, the temperature could be increased more than the elevated or melting point (Coats and Redfern 1963). The Thermogravimetric analysis (TG/DTA) of 5-20 Vol % n-TiB_2 Reinforced α-Al_2O_3/SiC based nanocomposite was carried out through NETZSCH STA 449F3 Analyzer. To avoid oxidation, the NETZSCH STA 449F3 Analyzer was initially blushed with pure argon gas (Li, Bao et al. 2019). Besides, throughout the testing duration, the Argon atmosphere was maintained inside the chamber. The initial mass of the samples is mentioned in Table 2.

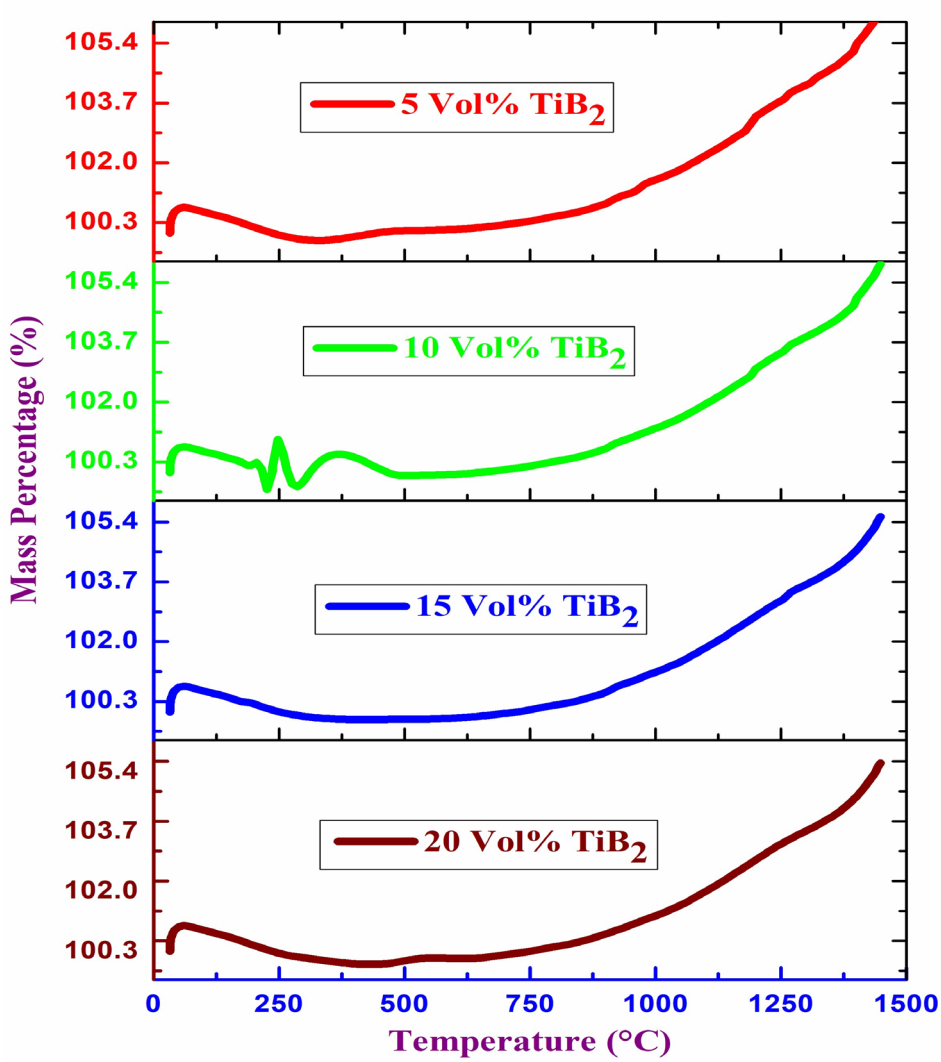


Figure 1. Mass percentage variation of S1, S2, S3, and S4 at different temperature recorded through Thermogravimetric Analysis (TGA).

The thermal response of the 5-20 Vol % n-TiB_2 Reinforced α-Al_2O_3/SiC (S_1, S_2, S_3, S_4) recorded from room temperature to 1500˚C with a heating rate 10 °C/min as displayed in the Figure 1. Initially, a small rise in the mass percentage was recorded, and that may be because of the impurities present in the test chamber or crucible. These impurities are of a negligible amount. But they produce a significant amount of Archimedes force that interns produce the buoyancy effect (Bhatt 1992, Raffaitin, Monceau et al. 2006). Further, the increase in temperature brought the mass percentage change to null at a particular time because of the impurities' evaporation. The temperature of the test chamber was increased continuously with the warming rate of 10 °C/min. 0.28%, 0.88%, and 3.8 % of average mass change were recorded at 500°C, 950°C, and 1300°C respectively, in all the samples. Reinforcement of n-TiB_2 enhanced the samples' thermal stability greatly by strengthening the bond between each molecule of the material. S_1, S_2, S_3, and S_4 are having enhanced thermal stability up to 1386°C, 1401°C, 1423 °C, and 1448°C with less than 5 percentage mass change. The recorded mass changes were comparatively less than the average mass change of raw materials (Hui-Mei, Chang-Wei et al. 2006, Shahbahrami, Bastami et al. 2010, Sadabadi, Aftabtalab et al. 2013,

Sathyaseelan, Baskaran et al. 2013, Blokhina and Ivanov 2015, Tishchenko, Ilchenko et al. 2015) as well as the base material (Al_2O_3/SiC)(Pathak, Bandyopadhyay et al. 2001). The mass change recorded for samples S_1, S_2, S_3 and S_4 at 1488.8°C is 6.57%, 5.55%, 5.35%, and 4.77%, respectively. Samples S_1, S_2, S_3 are recorded with higher mass percentage change than the sample S_4 (Al_2O_3/SiC+20 vol % n-TiB_2). Sample S_4 recorded less mass change, about 4.77%, which shows good thermal stability. From the comparison, it is evident that the addition of n-TiB_2 enhanced the thermal stability of the Sample Al_2O_3/SiC. Besides, the thermal stability increases with n-TiB_2 volume percentage up to 20 Vol %. The Thermogravimetric analysis of S_4 presented in **Figure 2** in detail. In the beginning, though the chamber is blushed with Ar Inert gas, a considerable gain in mass was recorded at 66°C. This mass change may be due to the Archimedes force, which creates a buoyancy effect because of the test chamber's impurities (Bhatt 1992, Raffaitin, Monceau et al. 2006). 0.7015% mass change was recorded at 66°C. Further increase in chamber temperature decomposes the impurities, which brought the mass percentage change nearer to 0 at 266°C. At 166.5°C, an endothermic peak was recorded with a small shoulder at 325°C with no recorded mass change. Moving towards the higher temperature decrease the mass percentage due to the reaction mechanisms of thermal decomposition. Kissinger's strategy can figure out the activation energy of the decomposed material. But the decomposition rate recorded up to 475°C was not significant because the mass change was almost null.

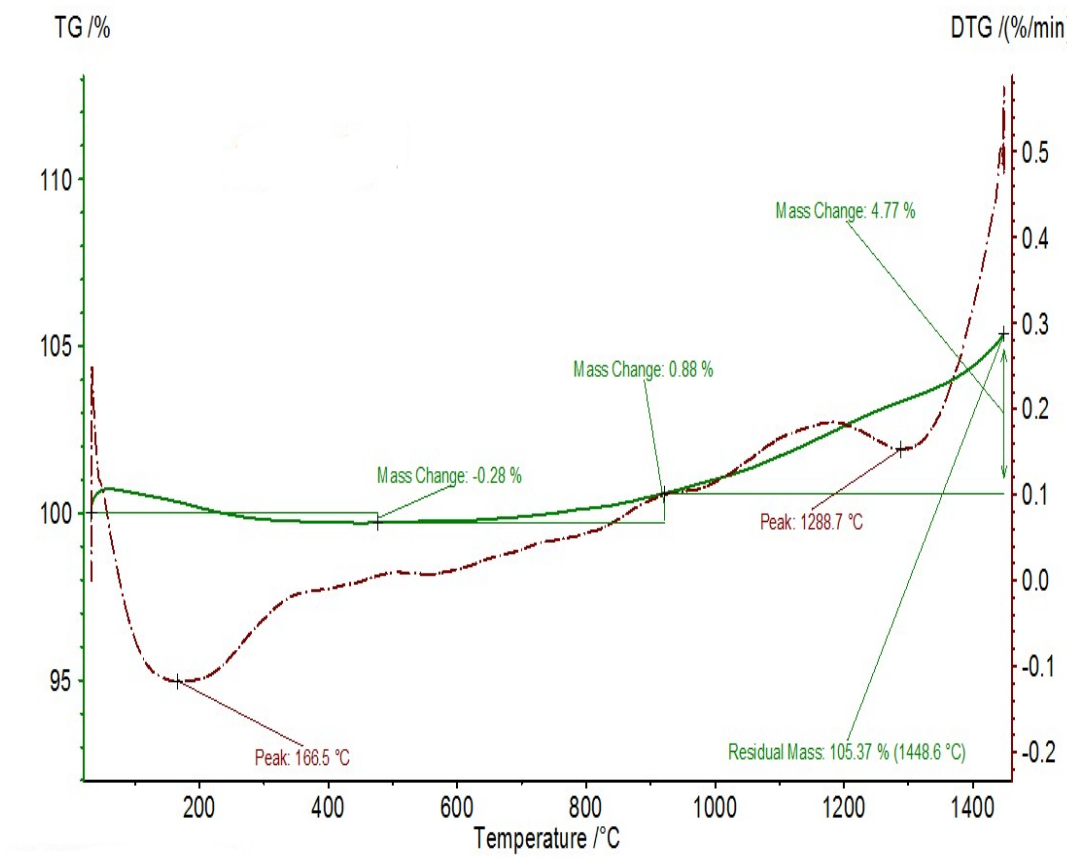


Figure 2. Thermal response of 20 Vol % n-TiB_2 reinforced Al_2O_3/SiC based composite under controlled Ar atmosphere.

When the temperature of the test chamber raised more than 475°C, the sample started gaining weight. Initially, the mass gain is too low, but the mass gain reaches a considerable amount as the temperature rises. **Figure 2** presented

no variation in mass percentage up to 750°C, which directly infers no decomposition up to that temperature. But the Al_2O_3/SiC nanocomposite fabricated through hot pressing presented a considerable percentage mass gain at 750°C due to the thermal decomposition(Wang, Ponton et al. 1995). Consequently, the addition of n-TiB_2 enhanced the collaboration between the atoms and postponed the decomposition temperature. From 750°C to the remaining test chamber temperature, considerable mass gain recorded and propagated because of the grain growth(Perez-Rigueiro, Pastor et al. 1998). Closer to 950°C, the Al_2O_3/SiC+20 vol % n-TiB_2 showed 0.88% mass change, and at 1488°C, the quantity raised to 4.77%, as shown in **Figure 2**. The material presented an endothermic peak at 1288°C because of the 2 % mass gain with 0.15%/min DTG. Closer to 1100°C, Ti_2AlC is available. Around 1300°C, the nanocomposite $Ti_3Si(Al)C_2$ began to frame because of the high-temperature molecular interference Ti on Al_2O_3/SiC. A significant amount of $Ti_3Si(Al)C_2$ recorded at 1400°C. 20 vol % n-TiB_2 added Al_2O_3/SiC offered 4.77% mass gain at 1488˚C. The recorded mass changes are comparatively lesser amount than the mass change of raw materials reported in (Hui-Mei, Chang-Wei et al. 2006, Shahbahrami, Bastami et al. 2010, Sadabadi, Aftabtalab et al. 2013, Sathyaseelan, Baskaran et al. 2013, Blokhina and Ivanov 2015, Tishchenko, Ilchenko et al. 2015) as well as the Al_2O_3/SiC (Pathak, Bandyopadhyay et al. 2001). Differential thermal analysis curves confirmed endothermic peak at 1288˚C and stability up to 1488˚C.

The microstructure of the n-TiB_2 added Al_2O_3/SiC nanocomposite

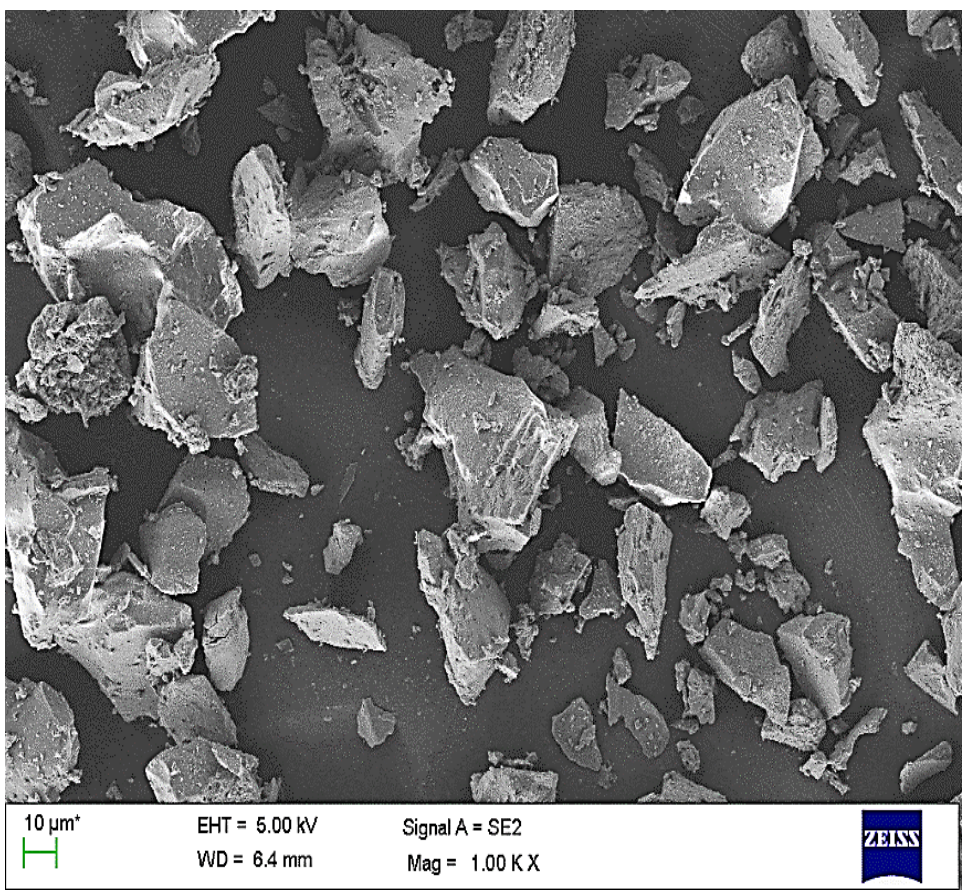
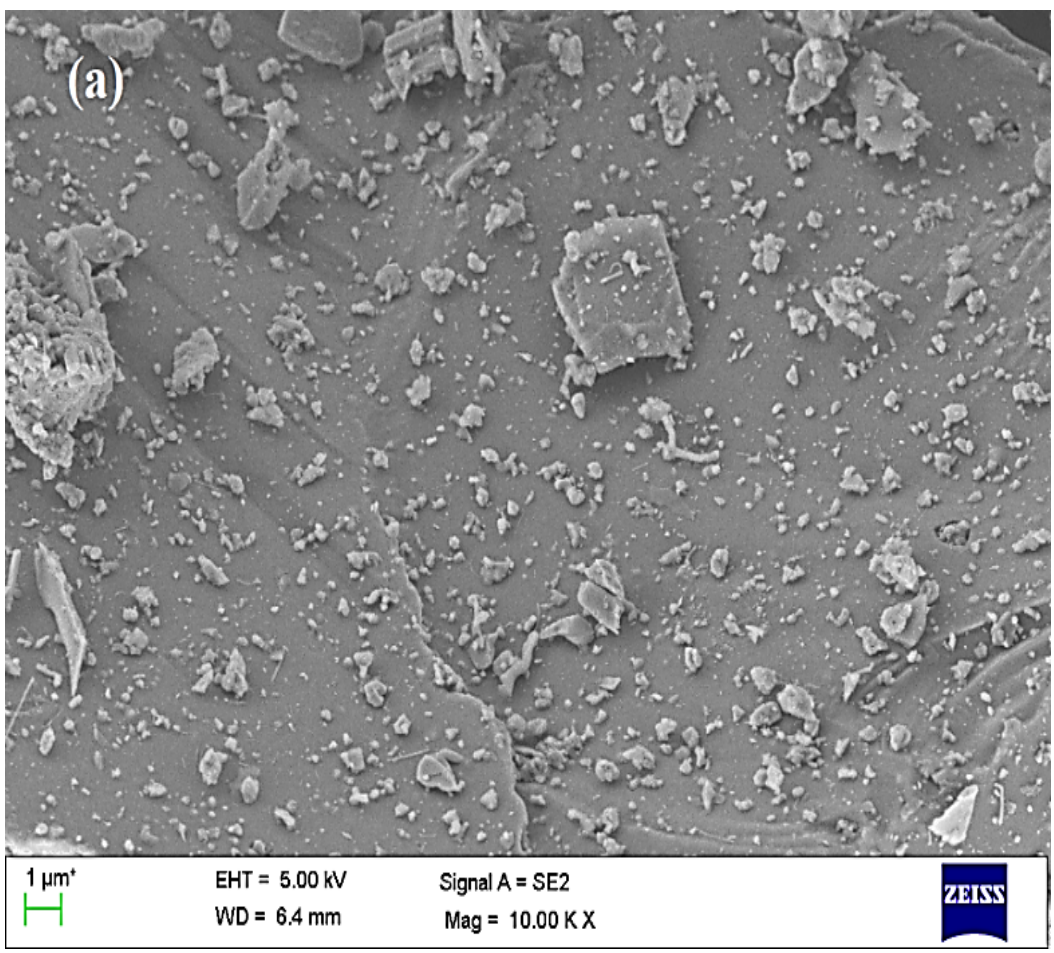


Figure 3. The microstructure of the 20 vol % n-TiB_2 added Al_2O_3/SiC green compact.

The microstructure of a composite material mainly depends on the raw material, type, shape and size of the raw material and adopted manufacturing method. Primarily, the elemental size of the raw material defines the microstructure and strength of the composite. The microstructure of the 20 vol % n-TiB_2 added Al_2O_3/SiC was captured by 20 kV JSM-5600J Scanning Electron Microscopy. As mentioned earlier in this paper, the green compact was initially compacted by using a hydraulic press. Then the microstructure of the green compact 20 vol % n-TiB_2 added Al_2O_3/SiC was captured.

Figure 3 displayed the microstructure of the green compact before sintering. On the other hand, the postsintering SEM images are shown in Figure 4. The composite's surface is smooth, and Titanium particles are distributed randomly, as displayed in Figure 4a. As discussed in TG/DTA, when the temperature value reaches 1300°C, the material started forming a composite Ti3Si(Al)C2. The postsintering microstructure of Ti3Si(Al)C2 and Titanium elements presented in Figure 4a&b. The average particle size of titanium boride found is 96 nm. Even though the Titanium particles distributed randomly, the Pressure less sintering increased the compactness between raw materials. That could be identified by comparing the pre-sintering (Figure 3) and postsintering (Figure 4) SEM images. The presence of Ti, Si, Al, O, and B in the nanocomposite confirmed by energy dispersive analysis of X-beams (EDS) as displayed in Figure 5.



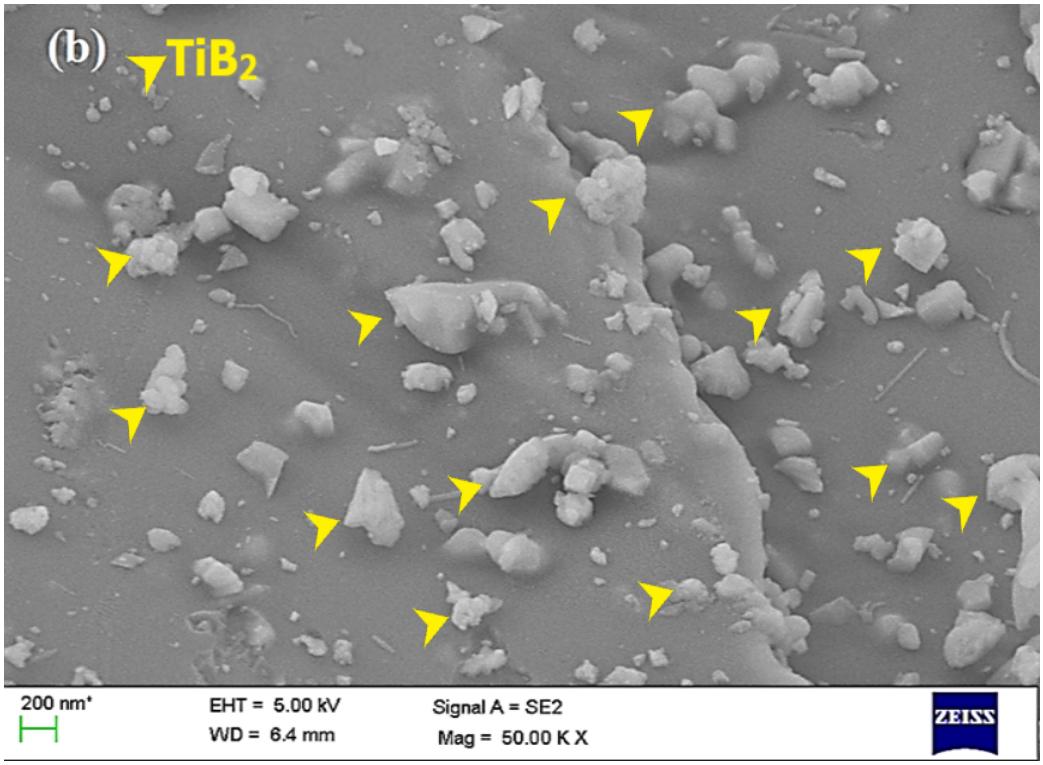


Figure 4 a&b. Microstructure of the 20 vol % n-TiB_2 added Al_2O_3-SiC nanocomposite after sintering.

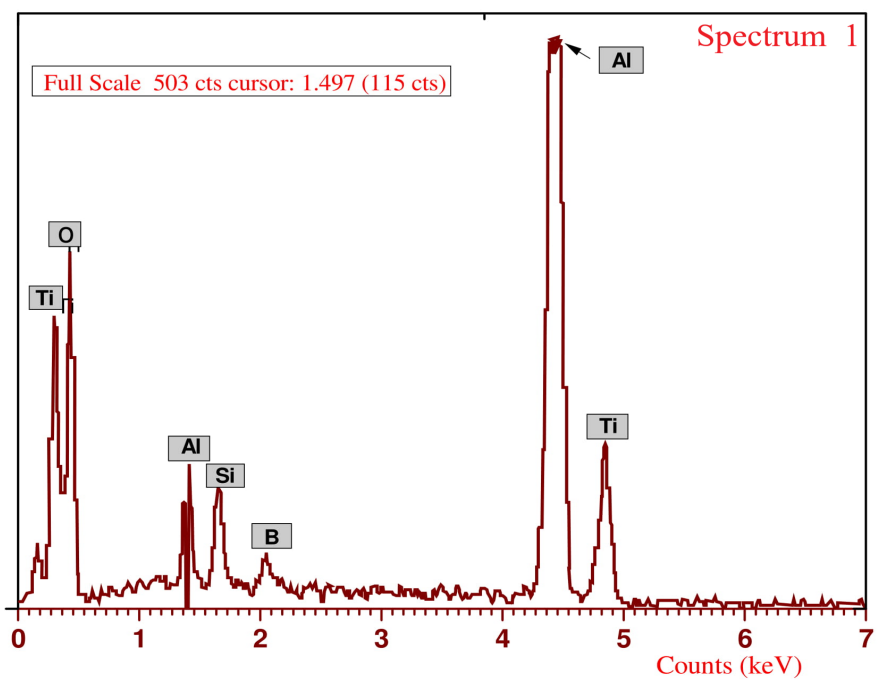


Figure 5. EDS result which confirms the presence of Ti, Si, Al, O, and B.

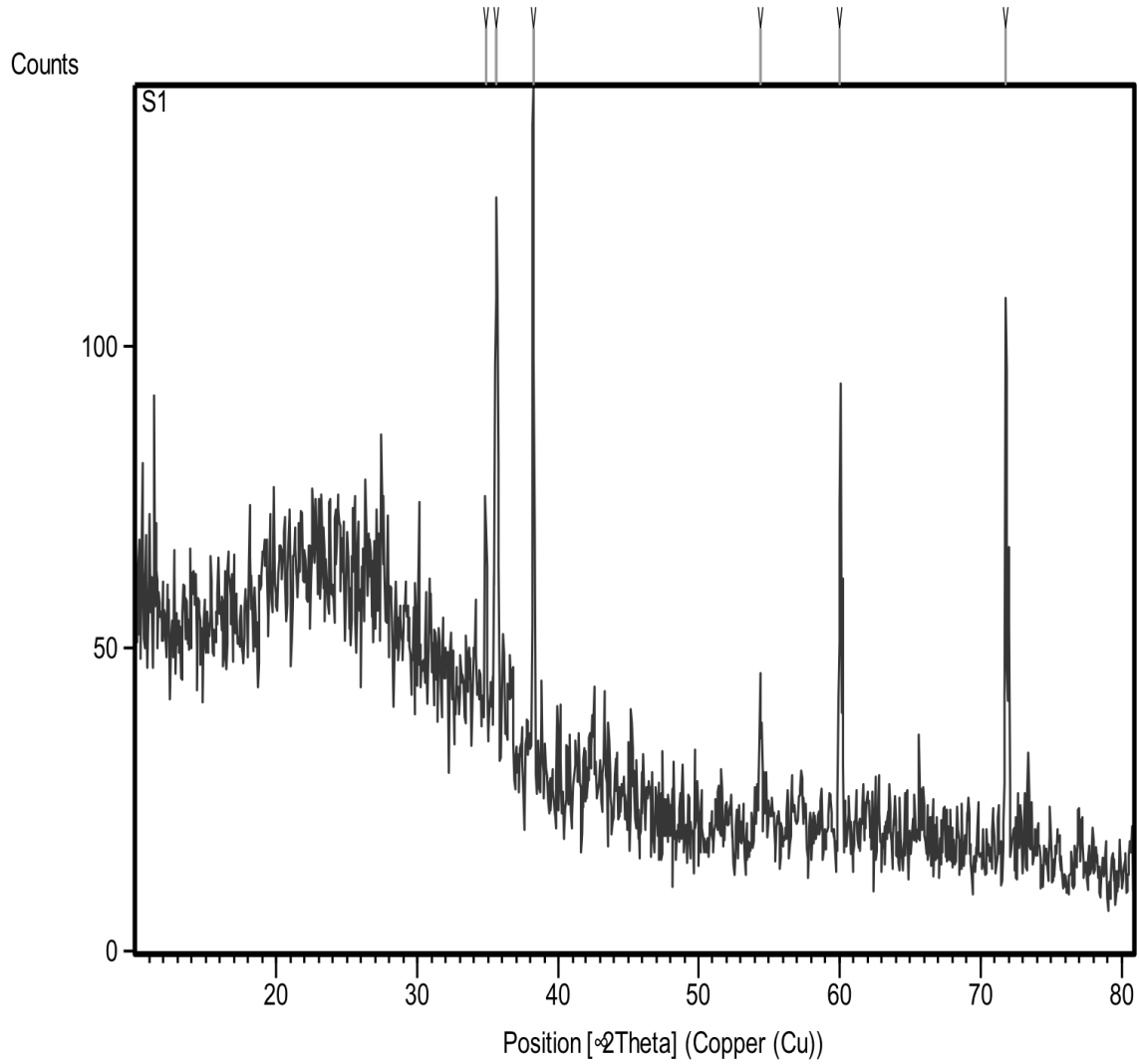


Figure 6. X-Ray Diffraction Observations of 20 vol % n-TiB_2 added Al_2O_3/SiC at 1500°C (Nallusamy 2020).

X-Ray Diffraction (XRD) analysis

XRD is a scientific technique used to identify the molecular and atomic structure of the material. The diffraction analysis on the S4 composite was carried out through Xpert-Pro Diffractometer with Cu Kα radiation. A 1.5420 N-m wavelength incident X-rays were used to irradiate the 20 vol % n-TiB_2 added Al_2O_3/SiC at 1500°C, and the intensities, scattering angles of reflected waves were recorded as shown in **Figure 6**. The recorded intensities are 29.41%, 78.99%, 100%, 12.83%, 45.16%, and 87.71% at 2θ values of angles 34.8555, 35.6227, 38.2255, 54.3916, 60.0078, and 71.7782, respectively. The highest intensity peak value of 100% was recorded at 2θ = 38.2255. Similar research reported by Yong Ning et al. (Jang, Enoki et al. 1995) on Al_2O_3-Ti_2SiC recorded a strong peak at 2θ= 38° as the second phase. In the present research, peaks have been identified at 2θ = 38.2255 with higher intensity. The

high-intensity peaks dictate the improved crystalline nature. These improvements confirmed that the TiB_2 interacted with the Al_2O_3/SiC structure and improved the crystalline nature.

CONCLUSION

Nano 5-20 Vol% TiB_2 doped Al_2O_3/SiC nanocomposites manufactured through pressureless sintering at 1600ºC under Ar atmosphere. Thermal stability of Al_2O_3/SiC/TiB_2 with different volume percentages of TiB_2 (5-20 vol. %) was analyzed by Thermogravimetric analysis. TG/DTA of Al_2O_3/SiC/20vol. %TiB_2 reveals better thermal stability, since less mass changes up to 1488ºC. The addition of TiB_2 increased the thermal withstanding capability of Al_2O_3/SiC composite. The SEM images presented a random arrangement of TiB_2 and revealed the compactness among elements. The nano TiB_2 powders were tightly packed with the other two powders by sintering at high temperatures. EDAX analysis was carried out by the EDS setup, which is attached with SEM, and the result confirmed the presence of TiB_2 elements after sintering. The XRD of 20 vol% TiB_2 added Al_2O_3/SiC recorded using Xpert-Pro Diffractometer, and the results confirmed the improved crystalline structure. This Titanium boride doped alumina-silica particulate composite would be preferable for hot-spot application, where high-temperature stability is necessitated.

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