Sintering, Mechanical, Electrical and Oxidation Properties of Ceramic Intermetallic TiC-Ti₃Al Composites Obtained from Nano-TiC Particles

Zhezhen Fu, Kanchan Mondal, Rasit Koc*

Department of Mechanical Engineering and Energy Processes, Southern Illinois University, 1230 Lincoln
Drive, Carbondale, Illinois, US

Abstract

The paper discusses the development of a new material system for interconnect application in Solid Oxide Fuel Cells (SOFC) based on TiC-Ti₃Al. Nano-sized TiC powders utilized in this research were synthesized using carbon coated TiO₂ precursors from a patented process. The pressureless sintering of TiC-Ti₃Al in a vacuum was applied at temperatures between 1100-1500°C and content of Ti₃Al was varied in the range of 10-40 wt. %. X-ray diffraction (XRD) and scanning electron microscope (SEM) were used for phase evaluation and sintering behavior. Relative density increased markedly with increasing sintering temperature because of grain growth and formation of the Ti₃AlC₂ secondary phase. Dense products (>95% TD) were prepared from nanosized TiC powders with 10 and 20 wt. % Ti₃Al, but with about 8 to 10% porosity for 30 and 40 wt. % Ti₃Al. The mechanical properties were determined from Vickers hardness and fracture toughness calculations. Vickers hardness decreased and fracture toughness increased with increasing Ti₃Al content. The electrical conductivity and oxidation behavior of TiC-Ti₃Al composites were investigated to evaluate the feasibility for SOFC interconnect application. The electrical conductivity measurements in the air at 800°C for 100 hours were made using the Kelvin 4-wire method.

Key words: Nano-sized TiC; Ti₃Al; Sintering; Mechanical properties; Electrical properties

1. Introduction

Due to increases in oil prices and renewable energy issues, significant research has been conducted for lowering the operating temperatures of Solid Oxide Fuel Cells (SOFC) such that they can be employed in vehicular applications. The new developments resulted in the demand for new material

* Corresponding author. Email address: kocr@siu.edu

1 systems with further improvements that are not attainable in oxide-based (doped lanthanum chromite) and
2 metallic alloys [1]. Some of the key requirements of SOFC interconnect are high electrical conductivity,
3 low contact resistance with the electrodes, good thermal conductivity (efficient removal of heat from the
4 electrodes is vital in maintaining an even temperature distribution), adequate chemical and thermal
5 stability, very low permeability to reactant gases, good mechanical strength, corrosion resistance, thin and
6 lightweight construction, easy manufacturability and low cost [1,2]. Titanium carbide (TiC) combines the
7 advantages of a high melting point, good mechanical properties, high electrical conductivity, and high
8 resistance to both oxidation and corrosion. These advantages make TiC a potential candidate for
9 application in SOFC. Carbothermal synthesis of TiC from carbon-coated titania powder was first
10 developed by Koc [3]. The synthesized TiC powders are pure and single phase, narrowly distributed in
11 size, loosely agglomerated, and spherical in shape [4]. Because of those features, synthesized nano-sized
12 TiC has better sintering properties compared with commercially available TiC [5]. Former research
13 showed nano-sized TiC is one of the most suitable electrically conductive filler materials in proton
14 exchange membrane (PEM) fuel cells [6]. This increases the potential application of TiC in SOFC. Ti_3Al
15 has excellent potential as advanced aerospace and elevated temperature structure materials because of
16 their high specific strength, good oxidation resistance and high temperature strength [7]. In titanium
17 aluminum alloys, Kirkendall diffusion pores will influence the density of final products due to difference
18 between the partial diffusion coefficient of Ti and Al [8, 9]. In order to eliminate high porosity in the
19 product, pressure is usually applied during the sintering process [10, 11,12]. Preparation and properties of
20 titanium aluminum alloy-ceramic composites have been extensively studied with pressure applied [13, 14,
21 15]. Compared with sintering with applied pressure, pressureless sintering has the advantage of higher
22 efficiency and improving the ability to satisfy shape requirements. Although $\text{Ti}_3\text{AlC}_2\text{-Al}_2\text{O}_3\text{-TiAl}_3$ [16]
23 and $\text{Ti}_2\text{AlC-B}_4\text{C-Al}$ composites [17] fabricated through pressureless sintering have been reported, more
24 detailed research is needed on preparing TiC- Ti_3Al composite.

25 In this research, nano-sized TiC synthesized from carbon coated titania and commercially
26 available Ti_3Al powders were selected. A pressureless sintering method was applied to increase sintering
27 efficiency. Different sintering temperatures and Ti_3Al contents were studied in order to produce dense
28 products. Densities, phase evolution, microstructure, mechanical, electrical and oxidation properties were
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investigated in detail. Based on those results, the composites' potential application in SOFC are discussed.

2. Experimental procedure

Nano-sized TiC powders were produced using the carbon coating process [3]. The process consists of two steps. The first step is the coating of TiO₂ (P25 Degussa Corp., Ridgefield Park, NJ) with carbon by decomposing a hydrocarbon gas (C₃H₆) at a temperature of 600°C. This step was performed in a rotating chamber furnace consisting of a 10 cm ID by 35 cm stainless steel cylinder. Approximately 100 g of TiO₂ powder was placed in the vessel. The vessel was evacuated, purged with argon, and evacuated again. The vessel was then heated to 600°C and filled with propylene gas until the pressure reached about 3 atm. After 5 minutes, the remaining gas was released and the vessel was filled with fresh propylene. The coating step was repeated until the desired amount of carbon was deposited. The weight percentage of the carbon in the precursors was determined using thermogravimetric analysis in the air and was found to be 31 wt. %. The second step involved the formation of TiC powders by promoting the carbothermal reduction of the carbon coated TiO₂ precursors at 1550°C for 4 hours in an argon gas flowing at 1 L/min. This step was performed in an atmosphere-controlled tube furnace (Model CTF 17/75/300, Carbolite, Sheffield, UK) with 70 mm ID. The precursor material and produced TiC powders were characterized using X-ray diffraction (XRD) (Rigaku, Tokyo, Japan) with Cu K_α radiation and transmission electron microscopy (TEM) (Hitachi, Model H-7650 TEM, ToKyo, Japan).

The produced TiC and commercially available Ti₃Al powders (99.5%, Alfa Aesar, Ward Hill, MA), as shown in Fig 1, were mixed using the dry mixing method in a plastic bottle with two methacrylate balls using a Spex Model 800 Mixer/Mill (Spex Corp., Metuchen, NJ) for 1 hour. The content of Ti₃Al varied from 10 to 40 wt. %. Pellets were made in a single action, uni-axial press in a hardened steel die (ID=12.95 mm) under pressure of a 50MPa. Densification was conducted over the temperature range 1100 to 1500°C for 1 hour in a graphite heated vacuum furnace. Densities of sintered pellets were determined by Archimedes' method. Theoretical densities were obtained from mixing TiC (4.93 g/cm³) and Ti₃Al (4.3 g/cm³). Percentage of theoretical densities, %TD, were then calculated using measured densities divided by theoretical densities. The surface of the samples were ground, then analyzed by XRD. A scanning electron microscope (SEM) equipped with Energy-dispersive X-ray

spectroscopy (EDS) (FEI Quanta FEG450, Hillsboro, OR) was used to investigate polished surfaces. Vickers hardness values were measured at least 10 times for each sample by a hardness tester (Shimadzu, HSV-20, Kyoto, Japan) using a load of 5 kg. The fracture toughness was determined using the Vickers hardness indentation method. By measuring the crack length and applying the following equation, derived by Anstis [18], the fracture toughness can be calculated:

$$K_{IC} = \delta \left(\frac{E}{H_V} \right)^{1/2} P C_0^{-3/2}$$

where: K_{IC} is fracture toughness, δ is a constant value of 0.016 ± 0.004 , E is the Young's modulus (taken from literature data [19] and the law of mixture was applied for composites), H_V is the Vickers hardness, P is the indentation load and C_0 is the crack length measured from the center of the indent.

The electrical conductivity measurements in the air for sintered samples with size of $3 \times 3 \times 10$ mm at 800°C for 100 hours were made using Kelvin 4-wire method, which were conducted on an AC resistance bridge (Liner Research Inc. LR-700, San Diego, CA). Each sample was weighed before and after the test by an electronic microbalance with an accuracy of 10^{-4} g to calculate weight gain. SEM and EDS were used to observe the polished cross sections of tested samples.

3. Results and discussion

3.1 TEM micrographs and XRD patterns of carbon-coated TiO_2 and produced TiC

Fig 2 and Fig 3 show TEM micrographs and XRD patterns of precursors and the produced TiC. As shown in Fig 2(a), a uniform coating of carbon on TiO_2 is apparent. Synthesized powders have a particle size around 100 to 300 nm as shown in Fig 2(b). Fig 3 shows the XRD patterns for the TiC formed from precursors which reacted at 1550°C for 4 hours in flowing argon. No secondary phase was detected. The lattice parameter of the product is $4.331 \text{ \AA}$ which is consistent with past research [20]. As mentioned before, the coating procedure forms an intimate mixing of reactants with high surface contact between carbon and titania. It also eliminates impurities such as iron and boron. Under these conditions, high purity TiC with fine particle sizes and narrow size distributions as well as loose agglomerations can be produced under a relatively lower temperature. This kind of TiC has better sintering properties compared with commercial TiC [5].

3.2 Sintering properties of TiC and Ti_3Al

XRD patterns of samples sintered at different temperatures and Ti₃Al contents are shown in Fig 4 and Fig 5. Densities and relative densities are shown in Fig 6 and Fig 7. It is observed in Fig 4 that the sintering temperature has an obvious effect on phases composition. As sintering progresses, chemical reactions between TiC and Ti₃Al lead to the formation of Ti, TiAl, Ti₃AlC, Ti₃AlC₂ and Ti₂AlC secondary phases. At 1100°C, TiC, Ti₃AlC, Ti₂AlC, Ti, TiAl and Ti₃Al phases can be observed. When the temperature is increased to 1200°C, the same phases can be found, but peak intensities for Ti₃AlC and Ti decrease while the peak intensity for Ti₂AlC increase. As the sintering temperature is further increased to 1300°C, Ti₃AlC and TiAl phases disappear while TiC, Ti, Ti₃Al, Ti₂AlC, and Ti₃AlC₂ phases are still present. Further increase in the sintering temperature to 1400°C reveal the disappearance of Ti₂AlC and Ti₃Al phases with a concomitant increase in the content of Ti₃AlC₂ (as shown by the increasing intensity of (002) peak at 9.5°). The Ti₃AlC₂ content increases when the sintering temperature is 1500°C. Phase evolution with increasing sintering temperature is summarized in Table 1. The lattice parameters of TiC decreased from 4.331 Å to 4.316 Å after samples sintered at 1500°C. Based on Holt's research [21], a decrease in carbon content in TiC_{1-x} leads to a decrease in the lattice parameter, which means that substoichiometric TiC_{1-x} was formed. Increasing the sintering temperature leads to the decrease of lattice parameters because more Ti₃Al reacts with TiC, thereby lowering the carbon/titanium ratio. Previous research [22] about the synthesis of Ti₃AlC₂ from elemental powders showed two reaction stages as follows:

In our current research, the raw materials included a large amount of TiC and no carbon black or graphite. The reaction between TiC and Ti₃Al should lead to TiC_{1-x}. The changes of lattice parameters, which are summarized in Table 2, also demonstrate that the source of carbon in Ti₃AlC₂ or TiAlC is from TiC. Based on XRD patterns regarding phase evolution as shown in Fig 4, and previous research [22, 23, 24], the following reactions are expected at different sintering temperature. Below or at 1200°C:

Between 1200°C and 1300°C, eq 4 would continue because more Ti_2AlC phases can be found and TiAl disappeared within this temperature range from XRD results. At the same time, Ti_3AlC also disappears because of the following reaction:

Ti_3Al exists at 1300°C and disappears at 1400°C, which leads the authors to hypothesize that the direct reaction between TiC and Ti_3Al may also occur:

The above reactions, specifically eq 4, eq 7, and eq 8 result in increasing the content of Ti_2AlC at 1300°C. As the sintering temperature approaches 1400°C and 1500°C, a decrease in the Ti_2AlC phase is observed until it is completely undetectable. Thus, an increase in temperature would result in the reaction between Ti_2AlC with TiC to form Ti_3AlC_2 [25]:

At the same time, with the temperature range between 1400 and 1500°C, the disappearance of Ti_3Al as shown in Fig 4 (d) and (e), and decrease of $1-x$ from Table 2, also indicate the direct reaction between TiC and Ti_3Al to form Ti_3AlC_2 :

Fig 6 shows relative densities increase with the change of the sintering temperature. Relative density was observed to go up from 71% to 96% as the temperature increased from 1100°C to 1500°C. The increase in the density as a function of temperature is much lower at temperatures below 1300°C as compared to the response at higher temperatures. Thus, in order to prepare dense products (higher than 95% TD) through pressureless sintering, a high temperature for $\text{TiC-Ti}_3\text{Al}$ composites is needed.

Samples with Ti_3Al whose content varied from 10 wt. % to 40 wt. % were sintered at 1500°C. All four samples are found to have two phases of TiC and Ti_3AlC_2 as shown in Fig 5. The content of the Ti_3AlC_2 phase increases with increasing content of Ti_3Al as expected. The lattice parameters of TiC

decrease with an increase in the content of Ti_3Al . This observation validates the discussions about the reactions between TiC and Ti_3Al . With increasing Ti_3Al content, more Ti_3Al or intermediates react with TiC which leads to substoichiometric TiC_{1-x} being formed. The estimated lattice parameters of these products are also summarized in Table 2. Densities and relative densities with the change of Ti_3Al content are shown in Fig 7. The relative density of the sintered product decreases with an increase in the Ti_3Al content. The authors believe that the reason for this decrease in %TD is due to the following reasons: (1) An increase in the Kirkendall diffusion pores [8, 9] (pores generated by different diffusion rates of Ti and Al atoms). This would lead to a decrease in the relative densities of the final products; (2) The differences of the densities of TiC and Ti_3Al as compared (on which the theoretical calculations are based) with TiC_{1-x} and Ti_3AlC_2 . Ti_3AlC_2 has a theoretical density between 4.2 to 4.29 g/cm³ [23], which is slightly lower than Ti_3Al (4.3 g/cm³). At the same time, density of TiC_{1-x} decreases with an increase in the value of x [26]. As mentioned earlier, the value of 1-x decreases with an increase in the content of Ti_3Al . Due to these reasons, the deviations between calculated theoretical densities and actual theoretical densities increases with increasing content of Ti_3Al .

SEM micrographs of samples sintering at different temperatures with 20 wt. % Ti_3Al are shown in Fig 8. When the sintering temperature was equal or less than 1200°C, as shown in Fig 8(a) and (b), TiC , Ti-Al alloy and $\text{Ti}_x\text{Al}_y\text{C}_z$ phases are observed. Based on the previously obtained relative density values, it's believed that very little densification occurred at this temperature range. When the sintering temperature was increased to 1300°C, similar surface morphology is observed with only some nano-sized, spherical shape TiC particles clearly appeared on the enlarged micrograph. The particles are also covered by some other small white particles. Commercially available Ti_3Al has a particle size range from a few microns to about 50 μm . In addition to the reaction with TiC , part of the Ti_3Al is believed to form a liquid phase and promotes densification. When the sintering temperature increased to 1400°C, nano-sized TiC particles no longer can be seen as compared with Fig 8(c), which demonstrates grain growth of TiC . TiC grain growth can eliminate a portion of the pores. Since a significant amount of pores are still observable in the samples, this grain growth is not sufficient to eliminate the porosity. As XRD patterns show in Fig 4(b), there are only TiC and Ti_3AlC_2 phases in this sample. However, Ti_3AlC_2 is not observed on the polished micrographs. Since only TiC and Ti_3AlC_2 phases were present in the sample, aluminum and titanium elements mapping was used to identify the Ti_3AlC_2 phases and estimate the grain size.

1 Ti_3AlC_2 grains have a needle shape with a size of approximately 5 to 20 μm in length and 3 to 5 μm in
2 width as shown in Fig 9. Comparing the particles which appear on the surface of TiC in Fig 8(a), (b) and
3 (c), this clearly shows the grain growth of Ti_3AlC_2 . As the sintering temperature reached to 1500°C, a
4 smooth surface with very few pores is shown Fig 8(c). Ti_3AlC_2 has a grain size of about 15 -35 μm by 5 -
5 10 μm which is larger and more uniform than the sample obtained at 1400°C. The grain growth of Ti_3AlC_2
6 also eliminates the pores. SEM micrographs of samples from 1100°C to 1500°C show evidence of the
7 liquid phase formation and the resulting grain growth of both TiC and Ti_3AlC_2 . All these factors
8 contribute to accelerated sample densification as the temperature was increased.

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17 Microstructures of samples sintered at 1500°C and varying Ti_3Al content from 10 to 40 wt. % are
18 shown in Fig 10. Aluminum mappings clearly show the presence of Ti_3AlC_2 phases. With an increase in
19 the Ti_3Al content, the area of Ti_3AlC_2 phases on the micrograph also increased. When the Ti_3Al contents
20 used was 10 and 20 wt. %, (Fig 10 (a) and (b)) TiC is the main phase and needle shaped Ti_3AlC_2 grains
21 are surrounded by TiC phase. As the Ti_3Al content used was increased to 30 and 40 wt. %, Ti_3AlC_2
22 became the dominant phase and appeared to prevent the TiC grain growth. At the same time, grain
23 growth of Ti_3AlC_2 is also observed. Grain sizes are found to be larger in samples with higher Ti_3Al
24 content. It is observed from the SEM micrographs that the open pores also increase with an increase in
25 the Ti_3Al content. This is consistent with the previous discussion regarding relative densities. Although
26 the higher Ti_3Al content in the samples resulted in increased liquid phase formation, the pores resulting
27 from the differences in the diffusion rates of different atoms dominated the progression of the sintering
28 process.

3.3 Mechanical properties of sintered samples

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42 The Vickers hardness and fracture toughness as a function of Ti_3Al content are shown in Fig 11.
43 The hardness decreases from 16.24 GPa to 4.89 GPa with an increase in the Ti_3Al content. This is
44 primarily due to the fact that Ti_3AlC_2 has a Vickers hardness of 2.5 GPa[23], while TiC has a relative
45 high Vickers hardness (>20 GPa). Three other possible reasons for the decrease in the hardness values
46 are: the lower relative densities of samples with 30 and 40 wt. % Ti_3Al ; lower Vickers hardness of TiC_{1-x}
47 with an increase in the value of x; and larger Ti_3AlC_2 grain size. Similar to $\text{Ti}_3\text{AlC}_2/\text{TiB}_2$ composites [27],
48 fine cracks initiated from the diagonals of the indent and a few cracks in the periphery of indents were
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observed for all samples. It was also observed that an increase in the content of Ti_3Al resulted in the increase in the frequency of half-penny like cracks on the periphery of the indents.

Fracture toughness increases with an increase in the content of Ti_3Al . The fracture toughness was calculated to be as high as $7.9 \text{ MPa}\cdot\text{m}^{1/2}$ when Ti_3Al content was 40 wt. %. As compared with TiC , Ti_3AlC_2 has a much higher fracture toughness [28] (fine grain $7.76 \text{ MPa}\cdot\text{m}^{1/2}$, coarse grain $9.46 \text{ MPa}\cdot\text{m}^{1/2}$). The increase in the fracture toughness value can be attributed to the increasing content of tougher Ti_3AlC_2 phase. On the other hand, Ti_3AlC_2 grain size is much larger for samples with 30 and 40 wt. % Ti_3Al as compared with samples with 10 and 20 wt. %. This may also lead to high fracture toughness. Finally, since the pores also inhibit the propagation of cracks, the presence of pores lead to high fracture toughness when the crack length measurement method is employed.

3.4 Electrical conductivities and oxidization properties

The values of electrical conductivity for the samples with different Ti_3Al content over time is shown in Fig 12. Weight gain per unit area, XRD patterns of tested samples, backscattered electron (BSE) micrographs of polished cross sections and their elemental distribution are shown in Fig 13, Fig 14, Fig 15 and Fig 16, respectively. In Fig 12, all $\text{TiC-Ti}_3\text{Al}$ composites show excellent electrical conductivity at elevated temperature. The values are all above 250 S/cm with the highest above 1200 S/cm . Samples with low Ti_3Al content have high conductivity. Electrical conductivities for all samples are slightly decreased with increased holding time. Fig 13 shows that increasing the Ti_3Al content resulted in a higher weight gain per unit area. XRD patterns in Fig 14 show the oxide scales are mainly TiO_2 with a very small amount of Al_2O_3 , even with the concomitant increase in Ti_3Al content. The strongly adhering oxide scale formed on the surface of each tested sample, as shown in Fig 15. Thickness of the oxidation layer increases with an increase in the Ti_3Al content. It must be noted that the outer edge of the scale contains more Al_2O_3 and the inner part of oxide scales are mainly TiO_2 , as shown in Fig 16 (a) and (b). The substrate matrix can be divided into TiC rich and $\text{Ti}_x\text{Al}_y\text{O}_z$ rich phases which both contain less oxygen compared with the oxide layer (Fig 16 (c) and (d)).

The sample's conductivity is determined by both ceramic matrix and surface oxide scale. As discussed before, samples with higher Ti_3Al content are expected to have more Ti_3AlC_2 and, consequently a higher value of x in the substoichiometric TiC_{1-x} . High substoichiometric TiC_{1-x} have

much lower conductivity as seen from Willams' report [29]. In this case, although Ti_3AlC_2 has a slightly higher electrical conductivity compared to TiC [23], the overall result is a decrease in electrical conductivity of the ceramic matrix with an increase in Ti_3Al content. The oxide scale which mainly contains TiO_2 and Al_2O_3 has a much lower conductivity compared with TiC_{1-x} or Ti_3AlC_2 . Al_2O_3 has also more electrical resistivity than TiO_2 at 800°C [2]. It is important to note that the thickness and content of the oxide scale dominates the electrical resistance. As shown in Fig 14, a sample with higher Ti_3Al content should have slightly more Al_2O_3 in the oxide layer. At the same time, as shown in Fig 15, thicker oxide layer is also formed for higher Ti_3Al content sample. This is also the primary reason for the decay in the observed electrical conductivity of samples with high Ti_3Al content at longer holding times. The authors believe that the Al_2O_3 scales are not continuous since all samples had relative good conductivity. Discussions about both ceramic matrix and oxide scale indicate conductivity difference for samples with different Ti_3Al content as a function of increasing holding time, as shown in Fig 12.

Former research showed monolithic Ti_3AlC_2 has better oxidation resistance than TiC or TiC/ Ti_3AlC_2 composite and low TiC percentages have deleterious effects on the Ti_3AlC_2 oxidation resistance [30]. It is interesting that a sample with lower Ti_3Al content, which means low Ti_3AlC_2 percentages, appears to have better oxidation resistance in this research. As the oxidation progresses, a higher diffusion rate of Al would lead to these atoms escaping from the Ti_3AlC_2 lattice to form non-continuous Al_2O_3 . Subsequently, the oxidation of Ti would result in the formation of TiO_2 layer. This TiO_2 scale prevents further oxidation. For samples with higher Ti_3Al content, because of high porosity, samples would be easier to oxidize due to lowered mass transfer resistance for the oxygen.

Based on above discussions, composites obtained from TiC- Ti_3Al appeared to have excellent mechanical properties, electrical conductivities and oxidation resistance compared with doped lanthanum chromite and metallic interconnect. The thermal expansion coefficient of TiC and Ti_3AlC_2 is $9.0 \times 10^{-6} \text{ K}^{-1}$ and $9.2 \times 10^{-6} \text{ K}^{-1}$ respectively, which are close to yttria-stabilized zirconia ($10.5 \times 10^{-6} \text{ K}^{-1}$) [1,2]. Thus, the application of TiC- Ti_3Al could be used as an interconnector in SOFCs. Further research into electrical conductivity in H_2 or H_2+CO atmospheres, and coefficient of thermal expansion is still needed.

4 Conclusions

In the present paper, dense products from synthesized nano-sized TiC with 10 and 20 wt. % commercial Ti_3Al mixtures and lower relative density products with 30 and 40 wt. %, were processed in 1500°C for 1 hour under a vacuum through pressureless sintering. XRD analysis indicated that the sintered samples were composed of Ti_3AlC_2 and TiC_{1-x} resulting from the reaction between Ti_3Al and TiC. SEM micrographs showed an increase in the amount of the Ti_3AlC_2 phase and grain size with an increase in Ti_3Al content. Vickers hardness decreased and fracture toughness increased with an increase in Ti_3Al content. All samples showed good electrical conductivities at 800°C for 100 hours. The formation of a dense $\text{TiO}_2/\text{Al}_2\text{O}_3$ oxide layer prevented continuous oxidation. Due to its good mechanical properties, electrical conductivity and oxidation resistance along with compatibility of the thermal expansion with yttria-stabilized zirconia, TiC- Ti_3Al composite has the potential application as an interconnector in SOFC.

Acknowledgement

Partial financial support by a grant (DE-FE0008864) from U.S. Department of Energy is gratefully acknowledged. The first author is also supported by China Scholar Council, Project of Study Aboard.

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Table 1 Phases composition with change of sintering temperature

Sintering temperature	Phases composition
1100°C	TiC, Ti ₃ AlC, Ti ₂ AlC, Ti, TiAl, Ti ₃ Al
1200°C	TiC, Ti ₃ AlC, Ti ₂ AlC, Ti, TiAl, Ti ₃ Al
1300°C	TiC, Ti ₂ AlC, Ti, Ti ₃ Al
1400°C	TiC, Ti ₃ AlC ₂
1500°C	TiC, Ti ₃ AlC ₂

Table 2 Lattice parameter of TiC from different samples

Ti ₃ Al content and sintering temperature	Lattice parameter
As produced TiC	4.331 Å
20 wt. % Ti ₃ Al, 1100°C	4.329 Å
20 wt. % Ti ₃ Al, 1200°C	4.328 Å
20 wt. % Ti ₃ Al, 1300°C	4.328 Å
20 wt. % Ti ₃ Al, 1400°C	4.326 Å
20 wt. % Ti ₃ Al, 1500°C	4.316 Å
10 wt. % Ti ₃ Al, 1500°C	4.325 Å
30 wt. % Ti ₃ Al, 1500°C	4.307 Å
40 wt. % Ti ₃ Al, 1500°C	4.304 Å

Fig 1 SEM micrograph of Ti_3Al

Fig 2 TEM micrograph of coated TiO_2 and produced nano-sized TiC. (a) TiO_2 coated with 31 wt. %, (b) produced nano-sized TiC

Fig 3 XRD pattern of produced TiC

Fig 4 XRD patterns of samples with 20 wt.% Ti_3Al sintering at different temperature. (a) 1100°C; (b) 1200°C; (c) 1300°C; (d) 1400°C; (e) 1500°C.

Fig 5 XRD patterns of samples sintering at 1500°C with different Ti_3Al weight percent. (a) 10 wt.%; (b) 20 wt.%; (c) 30 wt.%; (d) 40 wt.%

Fig 6 Relative density as a function of sintering temperature with Ti Al_3 content as 20 wt. %

Fig 7 Sintered density and **relative** density as a function of Ti₃Al content
from 1500°C

Fig 8 Microstructure of TiC with 20 wt. % Ti_3Al sintering under different temperature.
(a)1100°C, (b)1200°C, (c)1300°C, (d)1400°C, (e)1500°C

Fig 9 Elements map of samples sintering at 1400°C. (a) Aluminum, (b) Titanium

Fig 10 Microstructures with aluminum element mapping of samples sintering at 1500°C with different Ti_3Al content. (a)10 wt. % Ti_3Al , (b)20 wt. % Ti_3Al , (c)30 wt. % Ti_3Al , (d)10 wt. % Ti_3Al .

Fig 11 The Vickers hardness as a function of Ti_3Al content

Fig 12 Electrical conductivity of TiC-Ti₃Al samples as a function of time at 800 °C in air

Fig 13 Weight gain per unit area of whole test process

Fig 14 XRD of samples after electrical conductivity and oxidation test with different Ti_3Al content. (a) 10 wt. % Ti_3Al , (b) 20 wt. % Ti_3Al , (c) 30 wt. % Ti_3Al , (d) 40 wt. % Ti_3Al .

Fig 15 Microstructures of polished cross section tested samples with different Ti_3Al content. (a)10 wt. % Ti_3Al , (b)20 wt. % Ti_3Al , (c)30 wt. % Ti_3Al , (d)40 wt. % Ti_3Al .

Fig 16 EDS patterns of selected point of tested samples with different component. (a) point 1, edge of oxide layer, (b) point 2, inside of oxide layer, (c) point 3, TiC rich substrate matrix, (d) point 4, Ti_3AlC_2 rich substrate matrix.