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Investigation the effects of compaction pressure and sintering temperature on mechanical properties of Al-x%TiC composite

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Abstract. Al-*x*TiC (*x*=5, 10, 15 and 20 wt.%) composites were fabricated using powder metallurgy (P/M) technique from Al and TiC powders. The mixture of Al and TiC powders were homogeneously mixed for 4 hours before compacting using hydraulic pressing. The effects of compacted pressure and sintering temperature on the mechanical properties of Al-5 wt.% TiC composite were investigated in the ranges of 100 to 500 MPa, and 450 to 600 °C, respectively. The best investigated fabrication parameters of Al-5 wt.% TiC composite were applied for the fabrication of other compositions, i.e. Al-10, 15 and 20 wt.%TiC. After sintering, the phase formation and the microstructure of the Al-5 wt.%TiC composite were investigated using the X-ray diffraction (XRD) and Scanning electronic microscopy (SEM). As the results, sintered Al-5 wt.% TiC composite showed no oxidation or the formation of second phases, while SEM images indicated a homogenous distribution of TiC particles through the Al matrix. The microhardness and relative density of the composite increased with the increase of applied pressure, reaching 98.15% and 31.32 Hv at 500 MPa, respectively. The microhardness of the Al-TiC composites increased with the increase of TiC content, while the weight loss of the composites decreased with the increase of TiC content. The maximum microhardness of 39.2 HV and minimum weight loss of (8.1 mg/1000 m) were obtained for the Al composite with 20 wt.% TiC reinforcement.

Keywords: Al composite; mechanical properties; powder metallurgy; sintering

1. Introduction

Alumium (Al)-based materials are among the most extensively used lightweight materials for structural applications due to their high strength-to-weight ratio, superior corrosion resistance and machineability. Various industrial sectors, in particularly the automotive and aerospace industries, are prioritizing on the reduction of weight of the structures (Abdalkareem *et al.* 2023, Adil *et al.*

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2023, Shuang *et al*. 2023). This initiative aims to improve the fuel efficiency, reduce climate changing, and environmental damage. The most effective approach to achieve significant weight reduction is through density decrease, which directly resulting in reduced overall weight (Froes 1994). Vazuzan reported the possibility of substituting Al-based materials for cast iron, offering a substantial weight reduction of 12% for a small economy car (about 100 kg in total) and 15 to 17% for a diesel truck, which primarily composed of cast iron (Varuzan 1999). Typically, Adebisi *et al*. estimated that in automobile applications, a 7 kg iron brake rotor produced using an Al-based composites (AMCs) can reduce of 50 wt.% weight, which could result in a savings from $1.23 to $12.25 (Adebisi 2011). Other researches have also indicated that a 10% reduction in vehicle weight could lead to a 6 - 8% increase in fuel efficiency of (Sohail *et al*. 2020, William 2012).

To enhance the mechanical properties of Al for practical applications, various approaches have been employed, including alloying and composite fabrication. The manipulation of mechanical properties in Al alloys can be constrained by the challenges associated with dissolving alloy elements in the host metal. Alternatively, the mechanical properties of AMCs can be widely manipulated by reinforcing with various hard materials. These materials come in a with wide range volume fractions, particle sizes, and shapes such as SiC, TiC, B_4C Al_2O_3, CNT, and graphene etc. (Priyaranjan *et al*. 2020, Kaka *et al*. 2017, Singh *et al*. 2022). Among these reinforcements, TiC is a stable refractory material with a high melting point (3140°C), remarkable hardness (3.17×10^4 MPa), high modulus (420 GPa), and a low coefficient of thermal expansion (7.74×10^{-6} K^{-1}). These characteristics make TiC suitable for enhancing the wear resistance and strength of the Al-based composites at both room and evaluated temperatures (Huabing *et al*. 2018, Wei-Si *et al*. 2018, Anand *et al*. 2013). Recently, Al-TiC composites have attracted attention from numerous researchers due to their high mechanical performances, including high strength, wear resistance, and service temperature. These properties make them suitable for various applications in the automobile and aerospace, such as break discs, rotors, connecting rod etc. (Varuzan 1999, Priyaranjan *et al*. 2020, Kaka *et al*. 2017, Singh *et al*. 2022, Shi *et al*. 1999, Dong *et al*. 2014, Sahoo *et al*. 1991).

AMCs can be fabricated using various techniques such as liquid-state casting, powder metallurgy (P/M) processing. Both liquid and solid-state processes are being mainly applied to produce AMCs. Casting is particularly attractive due to its economical and practical technique. However, a significant main drawback of the casting process is the agglomeration of reinforcements, and containing high porosity, which leads to the reduction in mechanical properties. In contrast, P/M technique commonly used for the manufacturing of metal matrix composites because it offers several advantages compared to casting technique. These include a reduction the possibility of chemical reaction between the matrix and reinforcement phases, as the fabrication process takes place at low temperatures, and increased volume fraction of reinforcements incorporated into the matrices (Ruth 2021, Sangita *et al*. 2016, Halil *et al*. 2019). Another advantage of P/M is the uniformity distribution of reinforcements in the matrix. This uniformity not only enhances the structural properties but also improve the reproducibility levels in the resulting properties (Torralba *et al*. 2003). Finally, the products of P/M are near - net shape, minimizing the need for subsequent machining process (Priyaranjan *et al*. 2020, Halil *et al*. 2019, Sivaraj 2017, Adel 2008).

In this research, Al-*x*TiC (5, 10, 15 and 20 wt.%) composites were fabricated using P/M technique from Al and TiC powders. The formation of new phases, microstructure and interaction between the matrix and reinforcement were investigated after sintering. The effects of compacted pressures and sintering temperatures on the mechanical properties of the Al-TiC composites were systematically investigated. In addition, the composites's resistance to weight loss was also assessed.

2. Experimental procedure

In this research work, the starting materials were of Al powder (99.7% purity, particle size $\leq$ 44 μm) and TiC powder (99.6% purity, particle size $\leq$ 5 μm). The composition of composites was corresponding to Al-5, 10, 15, and 20 wt.% TiC. The mixtures of Al and TiC powders were homogeneously mixed for 4 hours using low-energy milling apparatus with the ball to powder ratio of 5 to 1 in dry condition before cold-pressing.

The mixed Al-5 wt.% TiC powders were compacted using hydraulic press at different pressures of 100, 200, 300, 400 and 500 MPa in a stainless-steel die with a diameter of 10 mm. The compacted samples were sintered at different temperatures of 450, 500, 550 and 600 ℃ for one hour in Ar atmosphere using Nabertherm tube furnace. The best-fabricated parameters of Al-5 wt.% TiC were applied for the fabrication of other compositions, i.e. Al-10, 15, and 20 wt.% TiC.

After sintering, new phase formation and microstructures of samples were characterized using X-ray diffraction (Bruker D8 Advance diffractometer with Cu Kα radiation 1.54059 Å), and a field-emission scanning electron microscope (FE-SEM, S4800, HITACHI), respectively. Densities and microhardness of sintered samples were measured using Archimedes' method and microhardness tester Axiovert 250 with a load of 4.9 N for 10 s, respectively, whilst the weight loss was measured using Tribo-Technic apparatus with sand papers # 800, disc rotation speed of 200 mm/s, and load of 12 N for different distances of 250, 500, 750 and 1000 m.

3. Results and discussion

3.1 Phase stability and microstructure

In order to study the phase stability, the XRD of the Al-xTiC (x= 5, 10, 15, and 20 wt.%) composites before and after sintering was analyzed, and the results is given in Fig. 1. Before sintering, the XRD pattern of the Al-5 wt.% TiC sample indicates only XRD peaks of starting elements, i.e. Al and TiC (Fig. 1(a)). After sintering at 600 ℃, the XRD pattern of the composite indicates the formation of no other new phases such as Al_2O_3 or the formation of Al_3Ti intermetallic during sintering, except XRD peaks of starting materials (Fig. 1(b)). Similar results were observed for others Al – 10, 15 and 20 wt.% TiC composites, except the increase of XRD peak intensity of TiC due to the increase of TiC content (Fig. 1c-e). These results indicate good protection of the composites from oxidation during sintering in the conventional furnace by using high-purity Ar gas.

No Al_3Ti intermetallic or Al_4C_3 was formed in the composites after sintering, which indicates the advantages of the low temperature of P/M technique compared to casting technique. In general, P/M technique process can be carried out at a temperature below the melting point of the Al matrix, while casting technique need to be carried out at the temperature higher than the melting point. Ranjit (2009) reported that the fabrication of in-situ Al-TiC composite by melting method at high temperatures from 700 to 1000 ℃ resulted in the formation of Al_3Ti intermetallic. This needle intermetallic phase is brittle, which led to the reduction in the ductile of the composite. In other researches (Tong *et al*. 2001, Contreras *et al*. 2007), intermetallic. i.e. Al_3Ti and Al_4C_3 were also found to be formed in the Al-TiC composite during melting at high temperature of 900 ℃ and 1350 ℃, respsectively.

To further investigate the composition of the sintered composite, the EDX analysis has been carried out, as shown in Fig. 2. The EDX spot on the matrix indicated Al, whilst a small amount of oxygen was also detected, which can be explained due to the formation of thin Al_2O_3 layer on the

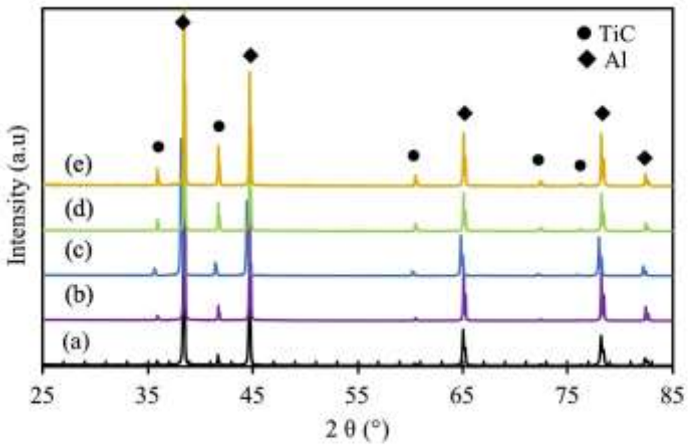


Fig. 1 XRD pattern of Al-*x*TiC (*x* = 5, 10, 15, and 20 wt.%) composites (a) a mixed Al-5 wt.% TiC powder before sintering and composites after sintering at 600 ℃, 1 h: (b) Al-5 wt.% TiC, (c) Al-10 wt.% TiC, (d) Al-15 wt.% TiC, and (e) Al-20 wt.% TiC

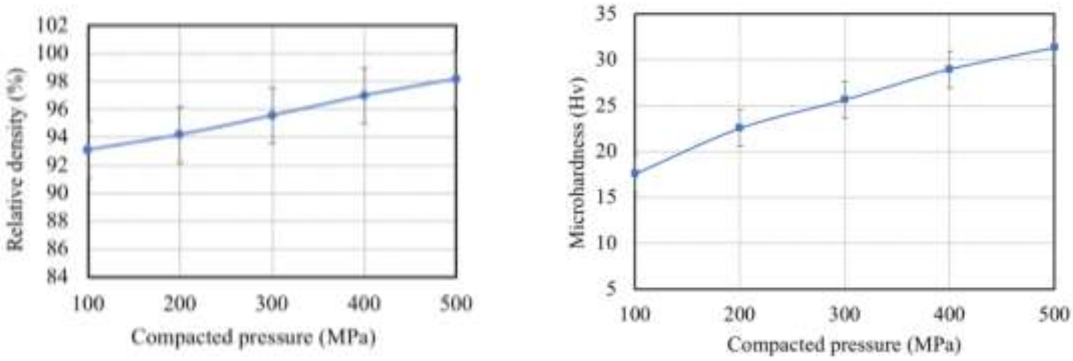
surface of the composite during exposing to the air (Fig. 2(a)). The EDX spot on the particle indicated the TiC particle reinforcement (Fig. 2(b)). This result is in agreement with the XRD results.

3.2 Distribution of TiC in the Al matrix

The distribution of TiC particles in the Al matrix shows in Fig. 3. It can be seen that TiC particles homogeneously distributed in the Al matrix (Fig 3. (a)-(b)), however, the size of TiC particles is not uniform. Under higher magnification, SEM image shows no sign of needle-like intermetallic phases Al_3Ti or Al_4C_3 (Fig. 3c). This observation is consistent with the XRD results presented in Fig. 1. In addition, it also could not observe any crack at the interfaces between the Al matrix and TiC particles, which indicates good bonding between the matrix and reinforcement. In addition, no pores can be observed in these SEM images. These results suggest P/M route is a suitable fabrication method for the Al-TiC composite. Tong *et al*. reported that the formation of Al_4C_3 particles in the casted Al – TiC composite led to the formation of micro-void coalescence, which inferior tensile strength and toughness of the composite (Tong *et al*. 2001). Ehsan *et al*. reported that Al-15 wt.%TiC composite was prepared using conventional technique from the mixture of Al and TiC powders, which was cold pressed at 250 MPa and then sintered in a conventional furnace at 700 ℃ (Ehsan *et al*. 2017). This composite showed microcracks around TiC particles. Ranjit *et al*. reported that TiC was observed to segregate at the grain boundaries in the as-cast Al-TiC composite, which fabricated by melting method at 1200 ℃ (Ranjit *et al*. 2011).

3.3 Effect of compacted pressure on mechanical properties

In order to investigate the effect of compacted pressures on mechanical properties of Al-5 TiC composites, the mixed powders were compacted at different pressures from 100 to 500 MPa and sintered at 550 ℃ (~ 0.8 T_m of Al) for one hour in Ar atmosphere. As the results, the relative density of Al-5 wt.%TiC composites increased with the increase of compacted pressures. At the compacted pressure of 100 MPa, the relative density of the composite was ~ 93 % of theoretical density.



(a) Relative density (b) Microhardness

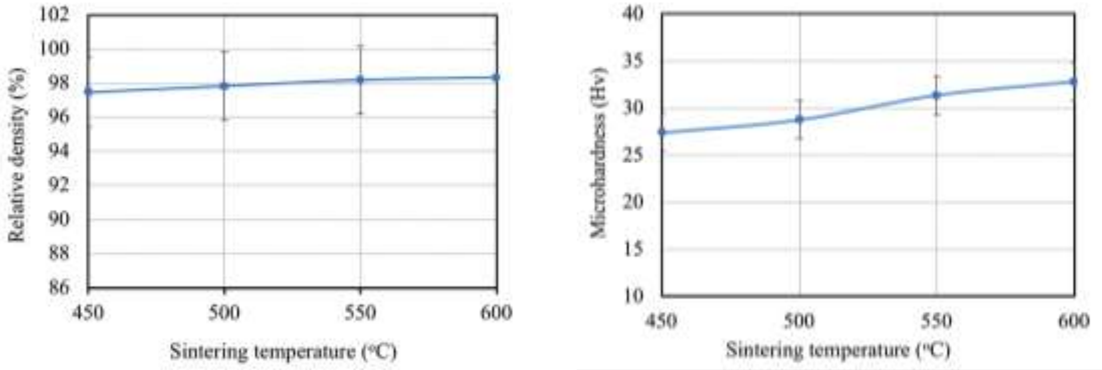
Fig. 4 Effect of compacted pressure on mechanical properties of Al-5 wt.%TiC composites

However, it reached a maximum value of 98.15 % of theoretical density at the compacted pressure of 500 MPa (Fig 4. (a)). In the P/M, the increase of compacted pressure leads to a higher degree of arrangement of particles and a reduction spaces between particle powders, which resulting in higher green density. Consequently, this reduces the mass transfer distances in the sample during sintering, which leads to the increase of sintered density (Randall, 1996). The high measured density of 500 MPa compacted pressure sample is in good agreement with the observations from SEM images in Fig. 3. This results also indicates the P/M is a simple and suitable technique for practical fabrication of Al-5 TiC composite without the need of hot pressing or other advanced sintering techniques. The relative density of composite samples in present work was comparable with the same composite, which fabricated using hot pressing at 400 MPa and 400 ℃ in research (Sangita *et al*. 2016). In this research, the achievement high density of composite sample by hydraulic press in combination with conventional sintering without applying pressure during sintering can be explained due to the achievement of high green density (96.8% of theoretical density) of the sample before sintering. The high green density was possible to achieve by hydraulic press because the Al matrix is soft, ductile and constitutes 95 wt.% of the composite.

Fig. 4(b) shows the increase of microhardness value of the composite as a function of compacted pressure. The results indicate the increase of the microhardness value increases by about 80% when increasing the compacted pressures from 100 MPa to 500 MPa. At compacted pressure of 100 MPa, the microhardness value of the composite was about 17.58 HV, which can be attributed to low relative density, as shown in Fig. 4(a). However, microhardness of the composite increased to 31.6 Hv when increasing compacted pressure to 500 MPa. In another research, Ranjit (2009) reported that the microhardness of in-situ Al-5 wt.% TiC composite is 37 Hv, which was fabricated using melting method from Al, K_2TiF_6 and graphite. The slightly higher microhardness value of Ranjit's work can be attributed to the formation of Al_3Ti intermetallic. However, this intermetallic is a brittle phase, which led to the reduction of toughness of the composite.

3.4 Effect of sintering temperature on mechanical properties

The effect of sintering temperature on mechanical properties was investigated for the Al-5 wt.% TiC composites, which were compacted at the same pressure of 500 MPa and sintered in the temperature range of 450 - 600 ℃, as shown in Fig. 5. The relative density of the composites shows a slight increase from 97.5 to 98.3% of theoretical density with the increase of sintering temperature



(a) Relative density (b) Microhardness

Fig. 5 Effect of sintering temperature on mechanical properties of Al-5 wt.% TiC composite

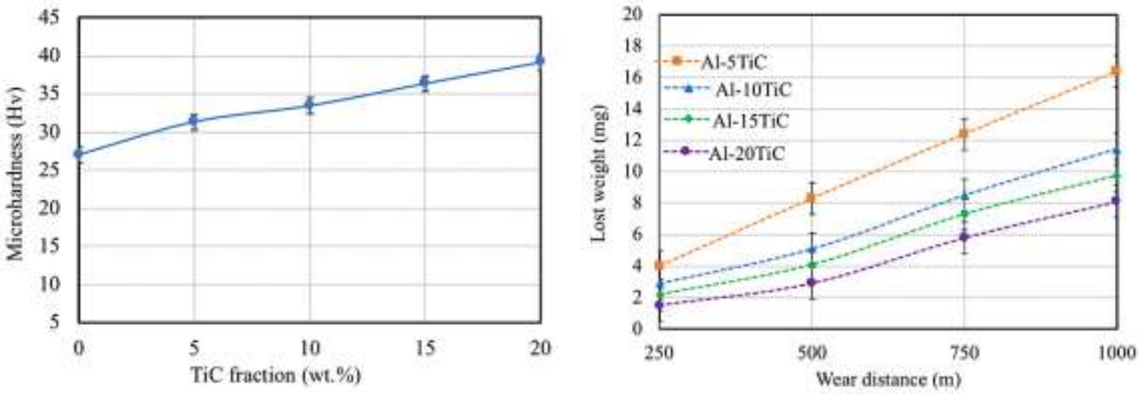
from 450 to 600 ℃, which indicates that the sintering temperature slightly influenced on the relative density of the composites. It can be explained that the green density of compacted samples has been reached a high value of 96.8% under high compacted pressure of 500 MPa before sintering. Therefore, in this case, the sintering temperature plays an insignificant role in the consolidation of the compacted sample.

Similarly, the microhardness of the composites also increases with the increase of sintering temperature, which increases about 20% from 27.4 to 32.7 HV when increasing the sintering temperature from 450 to 600 ℃. For the composite materials, the increase of microhardness can be explained due to the increase of the relative density with the increase of temperature (Ehsan *et al*. 2017), which enhances the resistance to load during testing. Despite the slight increase in density, the increase of sintering temperature still significantly contributes to bonding, thereby improving microhardness and consequently enhancing the strength of the composite (Kanhu *et al*. 2022).

3.5 Effect of TiC content on mechanical properties of the composite

Fig. 6 (a) shows the microhardness of Al-*x*TiC (*x* = 5, 10, 15, 20 wt.%) composites as a function of TiC content. The composites were prepared at the same conditions, which were cold compacted at 500 MPa and then sintered at 550 ℃ for 1 h. The results show that microhardness of the Al-*x*TiC composites increased with the increase of TiC content. The microhardness of Al matrix is approximately 27 HV, however its microhardness reached a maximum value of 39.2 Hv with the reinforcement of 20 wt.% TiC (Fig 6. (a)). The increase of the microhardness can be explained due the increase the formation of obstacles and the dislocation density as well as the modifications in microstructure, which prevented the movement of dislocations and thereby enhancing the microhardness of the composites (Tong *et al*. 2001).

In order to investigate the wear resistance of the composites, the weight loss of Al-*x*TiC (*x* = 5, 10, 15, 20 wt.%) composites were measured for different sliding distances and the results are given in Fig. 6(b). As expected, the weight loss of Al-*x*TiC composites decreased with the increase of TiC content. Specifically, the weight loss of sintered Al-5 wt.% TiC composite was notably higher compared to other compositions at all the testing distances. Conversely, the composite with 20 wt.% TiC reinforcement exhibited the lowest weight loss, which was approximately 1.5 mg and 8.1 mg at sliding distances of 250 m and 1000 m, respectively. At the sliding distance of 250 m, the weight



(a) Microhardness (b) Weight lost

Fig. 6 Microhardness and weight lost of Al-*x*TiC (*x* = 5, 10, 15 and 20 %) composites as a function of TiC content and sliding distance

loss of the composites was reduced about 170 % when increasing the TiC amount from 5 to 20 wt. %. At large sliding distance of 1000 m, TiC particles reinforcement exhibited more significant role in the prevention of weight loss, which was about 200% in the reduction with the increase of TiC content from 5 to 20 wt.%. In another research, e.g. Tavoosi *et al*. (2010) reported that the weight loss of Al-16 wt.% Al_2O_3 nanocomposites prepared by mechanical alloying and hot pressing was ~ 6 and 8 mg with applied forces of 10 and 15 N at sliding distance of 1000 m, respectively. While, Anand Kumar *et al*. (2014) reported that the weight loss of Al-4.5 wt.% Cu/5 wt.% TiC composite was about 40 mg with an applied load of 10 N at sliding distance of 1000 m. Similar to our research, the weight loss of these Al-based composites typically increased with the increase of applied load and sliding distance, and decreased with the increase of reinforcement content. The low weight loss of the composites with the increase of TiC content can be attributed to the hard TiC reinforcement phase, TiC restricting the flow of metal during sliding, and good interfacial bonding between TiC particles and the Al matrix etc. (Anand Kumar *et al*. 2014, Jerome *et al*. 2010, Tavoosi *et al*. 2010).

4. Conclusions

A high relative density of 98% theoretical density could be achieved for the Al-TiC composite, which was fabricated using a simple powder metallurgy technique by combining hydraulic press at 500 MPa, and sintering temperature of 550 ℃ in one hour from Al and TiC powders. The Al-TiC composites exhibited good protection from oxygen in the conventional tube furnace when using high-purity Ar gas. TiC particles reinforcement homogenously distributed in the Al matrix through conventional low-energy ball milling mixing technique. No cracks or debonding could be observed at the interfaces of Al matrix and TiC particles after sintering. At a high compacted pressure of 500 MPa, the sintering temperature has a slight influence on the relative density of the sintered composites, which can be attributed to the high green density. The microhardness of the Al-*x*TiC (*x* = 5, 10, 15, and 20 wt.%) composites increase with the increase of TiC content, while the weight loss of the composites decreased with the increase of TiC content. A minimum weight loss of 8.1 mg was measured for the Al-20 wt.% TiC composite at the sliding distance of 1000 m.

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