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Enhancing Zirconia-Toughened Alumina with Polyethylene Glycol (PEG) and Empty Fruit Bunch-Derived Solid Carbon

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ABSTRACT

Zirconia-toughened alumina (ZTA) is a widely utilized oxide ceramic known for its exceptional toughness, wear resistance, and corrosion resistance, making it suitable for applications such as cutting tools and biomedical devices. Despite its advantages, the burnout of binders like polyethylene glycol (PEG) at high temperatures leaves porous structures in ZTA composites, potentially hindering densification and material performance. This study aims to enhance ZTA composite properties by using PEG to create pores that facilitate carbon (C) infiltration through the chemical vapor infiltration process, utilizing biomass-derived carbon from empty fruit bunches (EFB). The ZTA composites were impregnated with different amounts of PEG, ranging from 0 to 5 wt%, to increase the porosity of their microstructures, as the composites had to be porous to facilitate efficient C deposition, and achieve optimal strength. Empty fruit bunches (EFBs) were used as the C source. The scanning electron microscopy (SEM) results revealed that the PEG formed pores on the surface of the ZTA composites, possibly, because the higher PEG concentration decreased the density of the ZTA composites. However, the density of the ZTA composites increased post C infiltration as the C filled the pore spaces. The C-infiltrated ZTA composite containing 3 wt.% of PEG had the highest density (4.145 g/cm^3) as well as the highest hardness (1911 HV) and fracture toughness (6.98 MPa.m$^{1/2}$). It also required the lowest Raman spectra intensity ratio (I_D/I_G, 1.26), which indicated that it contained a high percentage of sp2 hybridised C atoms and a higher degree of graphitising C. Our findings had proved that during the sintering process, the binder evaporated, leaving pores that were later filled with C. This improved fracture resistance, hardness, and density because fewer pores remained, and porosity was reduced. However, as the PEG content increased to 4 wt.%, mechanical properties declined. This was due to agglomeration, which created more pores in the microstructure, making it easier for cracks to spread.

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1. Introduction

Zirconia-toughened alumina (ZTA) is a very important oxide ceramic that is widely used as an economical material for cutting tools, biomedical devices, and wear parts [1]. The addition of zirconia (ZrO_2) to alumina (Al_2O_3) strengthens the matrix via tetragonal transformation to monoclinics when cracks form. This phase transformation toughening method causes about a 4 to 5 % expansion in volume, which increases its compressive stress and decreases the spread of the cracks [2]. As such, ZTA has been the centre of considerable research interest due to its resistance to wear and corrosion as well as high strength and toughness performance [3,4].

The purpose of adding a binder to a ceramic system is to form bridges between its particles and increase the strength of green-body ceramics [5,6]. Multiple binders have examined for use in the ceramics industry, the most common of which is polyethylene glycol (PEG). Polyethylene glycol (PEG) is a polymer that is easily soluble in various organic solvents; such as alcohol, benzene, and ether [7]. It is also non-toxic as it is organic, odourless, colourless, and non-volatile, which perfect for the ceramics industry [6]. However, PEG changes the microstructure of composites in terms of the degree of material densification [7]. Furthermore, binders evaporate and leave a porous structure in sintered Al_2O_3 bodies as they completely burnout at high temperatures of between 600 to 700°C in oxidising atmospheres [8]. The residue that it leaves may also cause poor densification in sintered composites [6]. One of the objectives of this present study was to use PEG to form pores on ZTA composites.

The pores that binder burnout leaves in ceramic composites facilitate carbon (C) infiltration, which can increase the density of a composite. Solid C is common reinforcement for ceramic matrix composites as well as a wide range of applications in various fields [9]. The addition of C to a composite increases the properties of the base material as it possesses excellent properties that can decrease the brittleness of ceramics and developing high quality of materials. Empty fruit bunch (EFB) biomass, which comprises C, oxygen, and hydrogen, is a well-known renewable energy source. As it is high in C, it could, potentially, be used to infiltrate C into ceramic composites. This could decrease environmental issues, such as uncontrolled waste disposal; and is cost effective as it is abundantly available in the oil palm industry [10]. This present study used chemical vapour infiltration to facilitate C infiltration into the ZTA ceramic composite via the fast pyrolysis of EFB, which produces biochar, biotar, and gases [11,12]. Tar vapour, which is the main product of fast pyrolysis, decomposes after it comes into contact with the pore surface of ceramic composites, thereby enabling its C to infiltrate into the ceramic composite.

While zirconia-toughened alumina (ZTA) has been extensively studied for its toughness and wear resistance, there is limited research on the effective integration of biomass-derived carbon (e.g., from empty fruit bunches) into ZTA composites to improve their mechanical properties. Existing studies have largely focused on binders like polyethylene glycol (PEG) for pore formation, but the impact of carbon infiltration via chemical vapor infiltration (CVI) from renewable sources on the microstructure and densification of ZTA composites remains unexplored. Addressing this gap could provide insights into sustainable material enhancements while using renewable resources.

2. Methodology

The ZTA composite powder was prepared by mixing a 4:1 ratio of Al_2O_3 and Yttria-stabilized zirconia (YSZ) while the PEG solution was prepared by adding 1 to 5 g of PEG flakes to a beaker filled of 100ml of distilled water and stirring it with a magnetic stirrer until it dissolved. The PEG solution and the ZTA composite powder were then mixed. A hydraulic carver press was set at 300 MPa pressure for six minutes to compact the samples. The green compacts then underwent pressureless

sintering in an electrical furnace set at 1600°C for one hour at a heating rate of 5°C/min. The EFBs were dried in an oven at 100°C for 24 hours before they were sieved. Glass wool was, first, placed inside the mould followed by 50g of EFB pellets and glass wool inserted compressively in the mould. The mould was then placed in a 500°C furnace at a heating rate of 10°C/min for four hours to begin the pyrolysis and infiltration process. The surfaces of the sintered samples were analysed using field emission scanning electron microscopy (FESEM). The other characteristics of the sintered samples; such as density, firing shrinkage, Vickers hardness, fracture toughness, and Raman spectra; were also investigated. Figure 1 shows the summary of the sample preparation and its respective characterizations.

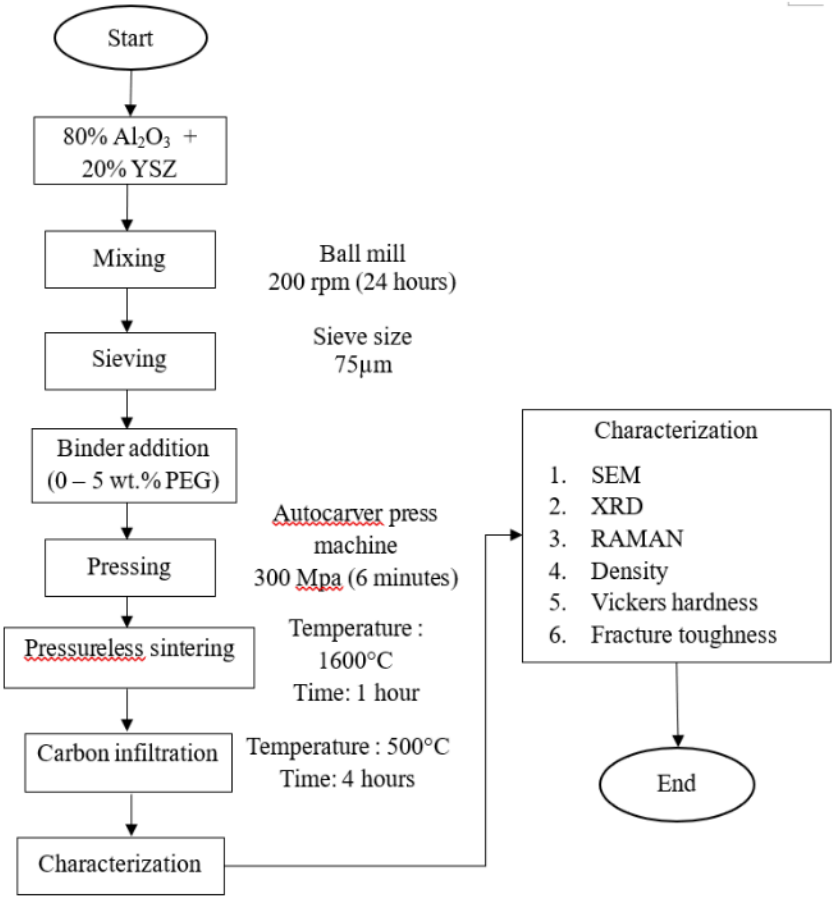

Fig. 1. Flowchart of the sample preparation and characterizations

3. Results

3.1 Phase Analysis

Figure 2(a) shows the phase analysis results of ZTA containing 1 to 5wt.% of PEG. The main phases found were α-Al_2O_3, which was in the corundum phase, and ZrO_2 in the tetragonal phase. This oxide group is, usually, used as a highly temperature resistant material that is suitable for use in cutting tools [13]. The ZrO_2 used in this present study was in the tetragonal phase, which is more stable than the cubic and monoclinic phases. Therefore, when ZrO_2 is in the tetragonal phase, it can become metastable [14].

Other than that, although binders are used in ceramic systems, they burn out and remove organic components from green bodies during heat treatment. As such, binders do not appear at the diffraction peaks. According to Zare *et al.,* [15], organic binders evaporate and leave a porous

structure in Al_2O_3 sintered bodies at high temperatures. Their residue also reduces the level of crystallinity in a ceramic system. Therefore, increasing the amount of PEG in a composite will decrease the intensity of its X-ray diffraction (XRD) peaks as diffraction peaks decrease at higher PEG concentrations.

Figure 2(b) depicts the XRD patterns of C-infused ZTA composites containing different amounts of PEG. The Al_2O_3 and ZrO_2 phases were observed in the composite, but C peaks were not. According to extant studies, C appear largely similar in XRD with main peaks appearing at 24° and corresponding well to the (002) planes and 43° corresponding to the (100) planes [16-18]. However, C was not observed in Figure 2(b), possibly, because the C content may have been very low. Furthermore, as many crystal structure elements exhibit higher intensities when approaching 8000 units, it may have overshadowed the 120 units at low C intensities [19]. Nevertheless, ZTA composites containing PEG had higher C intensity patterns, which proved that the organic PEG binder evaporated and left porous structures in the sintered body and facilitated the infiltration of C into the pore spaces.

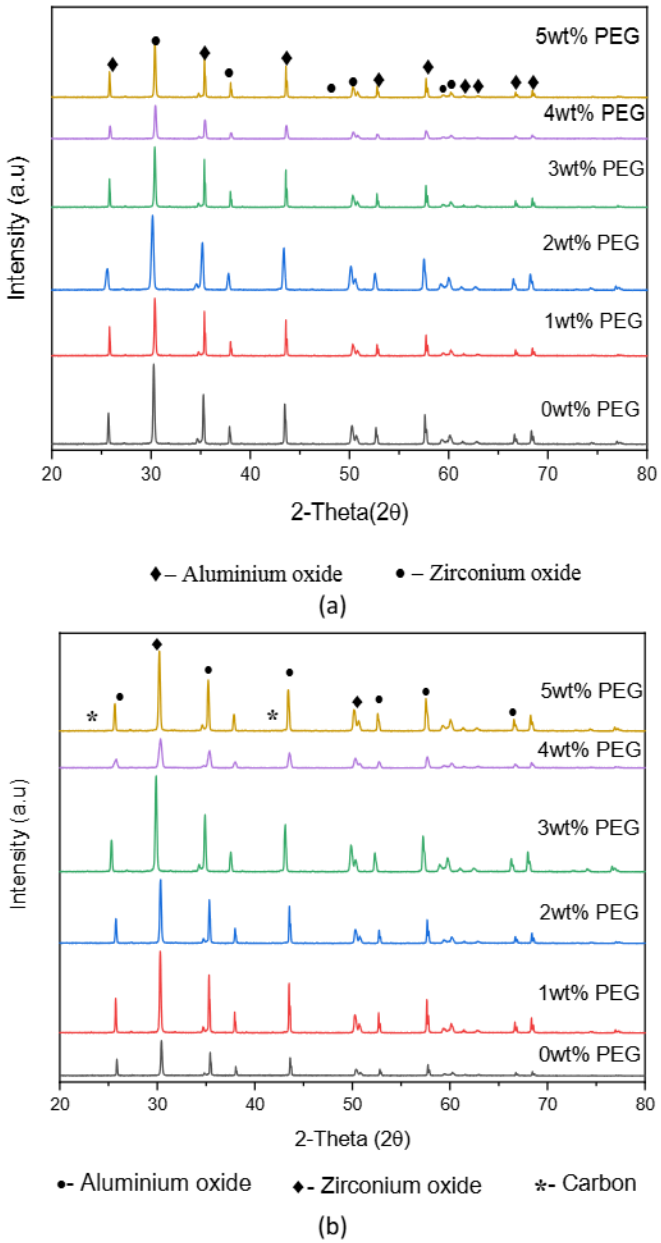

Fig. 2. The XRD patterns of the (a) ZTA composite and (b) C-infiltrated ZTA composites containing 0 to 5 wt% of PEG

3.2 Microstructure Analysis

Figure 3(a) to 3(f) depict the microstructural evolution of ZTA composites containing 1 to 5 wt% of PEG. Figure 3(a), which depicts a pure ZTA sans the addition of a binder, was similar to that of previous studies [20,21]. More specifically, the microstructure was homogeneous and did not contain aggregates or pores. Abnormal grain growth was not observed either. As seen in Figure 3(b) to 3(f), the number of pores increased as the PEG content increased. The addition of 1 wt.% of PEG did not significantly change the microstructure of the composite. However, the addition of more than 2 wt.% of PEG increased the appearance of pores. As such, higher concentrations of PEG will yield ZTA composites that are relatively inhomogeneous. Furthermore, as the PEG granules did not significantly deform, they caused poor densification in the ZTA composites. These microstructural observations prove that the addition of a PEG binder decreases the density of the ZTA composites as the PEG burns out at high temperature, leaving behind pores. Therefore, increasing the amount of binder will increase the porosity as well as decrease compactness and density [22].

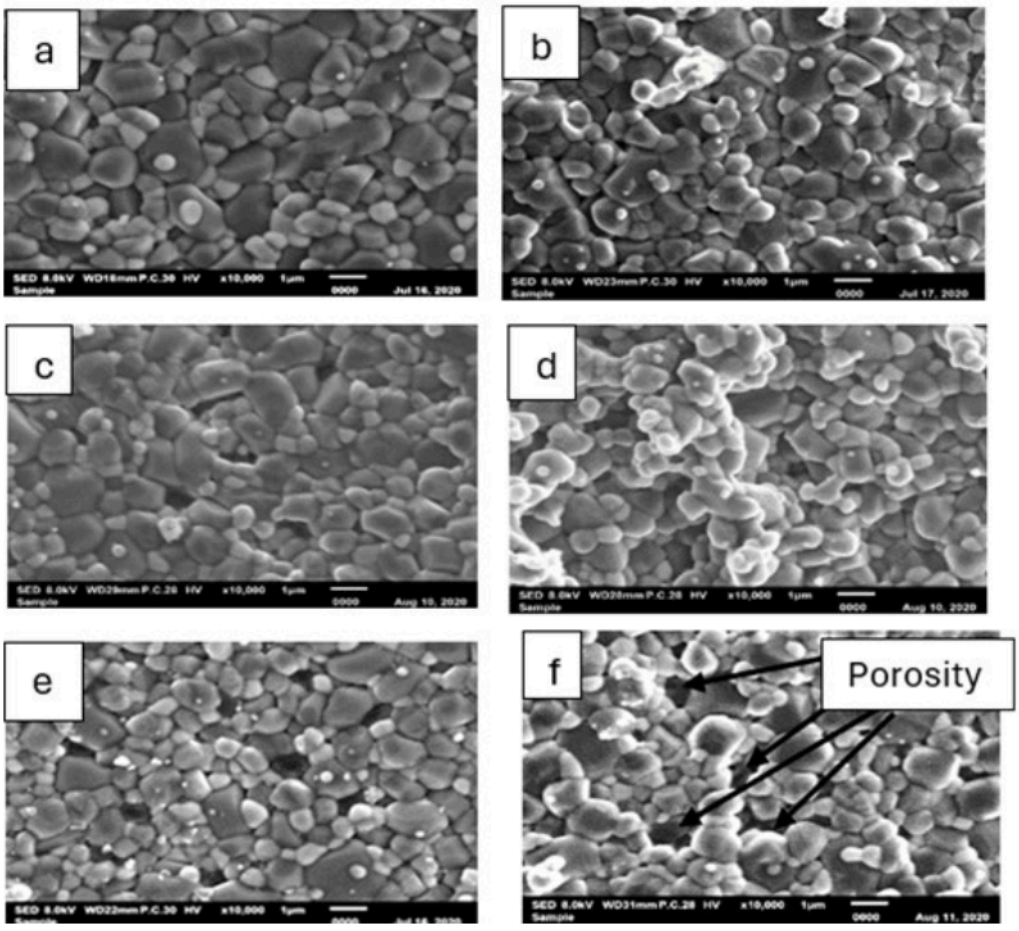

Fig. 3. The microstructural images of ZTA composites containing (a) 0, (b) 1, (c) 2, (d) 3, (e) 4, and (f) 5 wt% of PEG at 10000x magnification

Figure 4(a) to 4(f) depict the formation of the biochar that filled the ZTA composites' pores as narrow pores were visible post tar carbonisation in the pores. Rozhan *et al.,* [12], similarly, found that when the pyrolysis vapours meet the pore surface of the composite, the decomposed tar is placed inside the pores, leaving behind a thin layer of solid C. Therefore, pyrolysis at high temperatures facilitates the production of tar vapours, which increases the C content of the pores. The C agglomerates observed in Figure 4(b) to 4(f) may have formed due to poor C diffusion in the ZTA

matrix. This would, in turn, affect the density of the ZTA composite and deteriorate its mechanical properties, such as its hardness. According to Molok *et al.,* [23], excessive amounts of C will agglomerate in a composite and adversely affect densification.

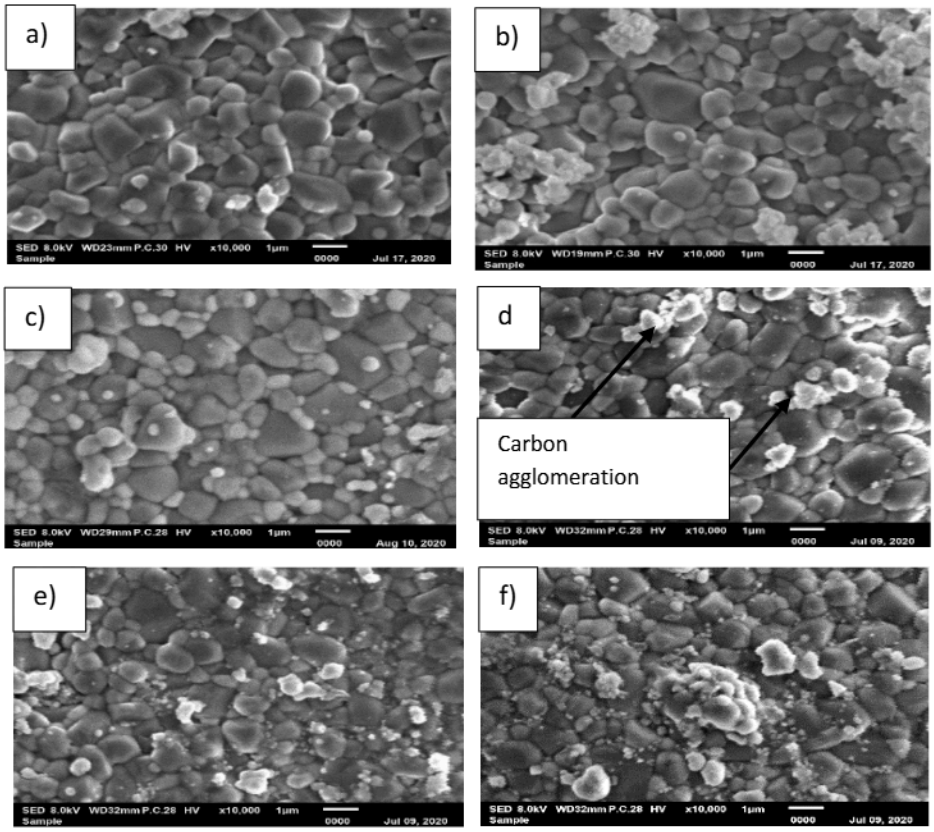

Fig. 4. The microstructural images of C-infiltrated ZTA composites containing (a) 0, (b) 1, (c) 2, (d) 3, (e) 4, and (f) 5 wt% of PEG at 10000x magnification

3.3 Density

Figure 5 shows the density data of pure ZTA and C-infiltrated ZTA composites containing 0 to 5 wt% of PEG. As seen, increasing the amount of PEG, from 0 to 5 wt. %, decreased the density of the ZTA composite from 4.043 to 3.866 g/cm^3. The ZTA composites containing 1 wt. % of PEG had the highest density. Therefore, as little as 1 wt. % of PEG will enhance the density of a composite. The density decreased upon the addition of more than 1 wt. % of PEG. This may be due to PEG burnout at higher temperatures, which causes pores to form in the microstructure. Moreover, the addition of binder fortifies the granules of the composite, leading to fewer granule fragments and, thus, lowering the density of green bodies [24]. According to Zare *et al.,* [15], the density decreases depending on the amount of PEG added. When the grains of a composite are pressurised, the force causes them to move and rearrange. The higher the binder content, the higher the movement for rearrangement, which decreases its density. However, it is noteworthy that Zare *et al.,*'s study only examined pure Al_2O_3 while this present study examined the addition of ZrO_2 to Al_2O_3.

The C-infiltrated composite containing 3 wt.% of PEG had the highest density (4.146 g/cm^3)**.** This was because the binder evaporated during the sintering process, leaving behind pores to be filled with C, which increased its density. A PEG binder has also been used to facilitate good C diffusion in composite matrices [25]. However, the density gradually decreases as the PEG percentage approaches 4 wt.% due to agglomeration.

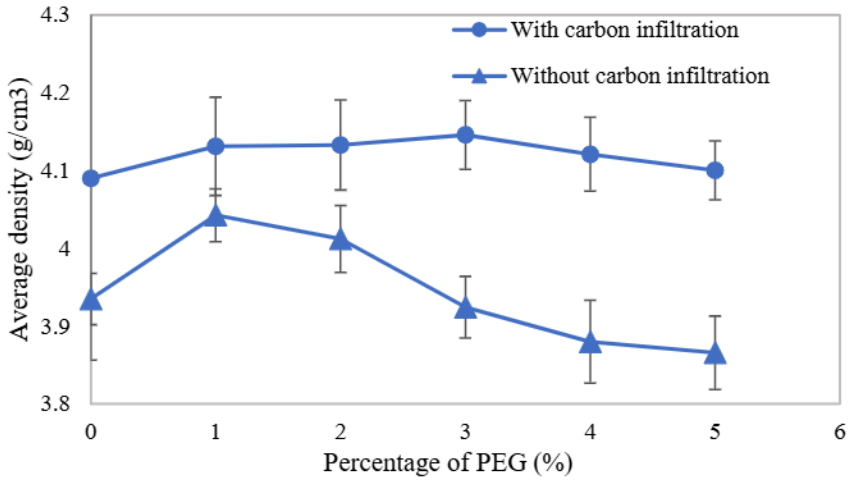

Fig. 5. The density of ZTA composites containing 0 to 5wt. % of PEG

3.4 Hardness

As seen in Figure 6, the hardness of the ZTA composite is a function of the amount of PEG that is contains. As expected, the hardness of the ZTA composite containing 0 and 3 wt. % of PEG significantly increased, from 1599 to 1685 HV. The purpose of adding binders to ceramics is to increase the strength of green bodies by forming bridges between the particles during Al_2O_3 formation [26]. However, the hardness decreased slightly when more than 3 wt.% of PEG was added to the composites. Therefore, excessive amounts of PEG will decrease the hardness of a composite as it would produce larger pores and the density of a ZTA composite is lower when the PEG content is high, which decreases the hardness.

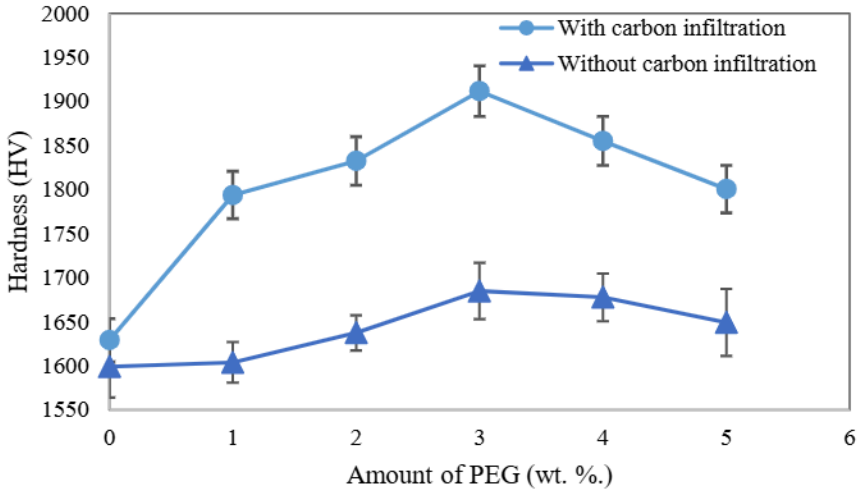

Fig. 6. The hardness of ZTA composites containing 0 to 5wt. % of PEG

The hardness of C-infiltrated composites containing 0 and 3 wt. % of PEG increased steadily, from 1629 to 1911 HV. Composites containing C were harder than composite without C. This increase in hardness may relate to the microstructure, where the solid C was uniformly distributed on the surface and in the Al_2O_3 grains, which increased the effective load transferred from the matrix to the reinforcement [9]. Lee *et al.,* [27], similarly, found that C improves mechanical properties. However, the addition of more than 3 wt.% of PEG slightly decreased the hardness of the composite due to

poor C distribution. Agglomeration may occur at this stage if the grains and binders did not mix evenly, leading to low densification and hardness. The agglomeration of C also enables the matrix to more easily separate, creating a weak load while the pressure was transferred [28].

3.5 Fracture Toughness

As seen in Figure 7, the fracture toughness of the ZTA composites with C infiltration increased gradually, from 6.002 to 6.567 MPa.m$^{1/2}$, with the addition of 0 to 3 wt. % of PEG. However, it decreased slightly when more than 3 wt. % of PEG was added. The density and hardness, similarly, decreased when more than 3 wt. % of PEG was added. This low fracture toughness may be due to the large number of pores that formed in the microstructure, which could have facilitated the spreading of cracks. Therefore, the addition of the PEG binder to the ZTA composite created pores post-sintering, which decreased its fracture toughness during loading. The fracture toughness of C-infiltrated composites was higher than that of composites without C infiltration. The C-infiltrated ZTA composite containing 3 wt.% of PEG had the highest fracture toughness (6.98 MPa.m$^{1/2}$). As such, its fracture resistance, hardness, and density are higher as fewer pores had formed in its microstructure and its porosity was lower as the C had infiltrated and filled its pore spaces.

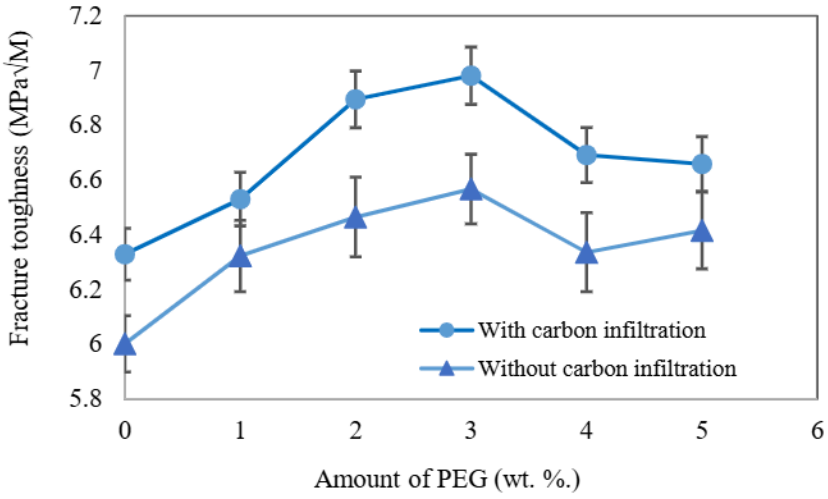

Fig. 7. The fracture toughness of ZTA composites containing 0 to 5 wt. % of PEG

3.6 Raman Spectra

Figure 8 depicts the Raman spectra of C-infiltrated ZTA composites containing 0 to 5 wt. % of PEG. Two characteristic bands of the C structure, the D- and G-bands, appeared around 1350 and 1580 cm^{-1}, respectively [29,30]. Carbon (C)-infiltrated ZTA composites containing 0 to 5 wt. % of PEG exhibited C diffusion peaks at the D- and G-bands, indicating the presence of C. As seen in Figure 9, the intensity ratio of the D- and G-bands (I_D/I_G) increased as the amount of PEG increased from 0 to 2 wt.% but decreased when 3 wt.% of PEG was added to the composites. The C-infiltrated ZTA composites containing 3 wt.% of PEG had the lowest in I_D/I_G (1.26), indicating that it contained a higher percentage of sp2 hybridised C atoms and an increase in the degree of graphitising C [31]. Higher I_D/I_G values indicated a high degree of impurities and structural defects as well as low crystallizable C, which will deteriorate the properties of the ZTA composite [32].

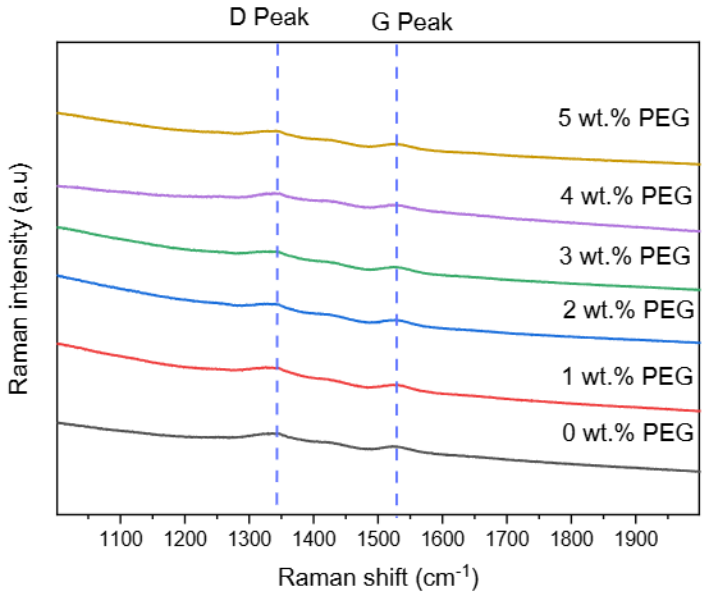

Fig. 8. The Raman spectra of C-infiltrated ZTA composites containing 0 to 5 wt. % of PEG

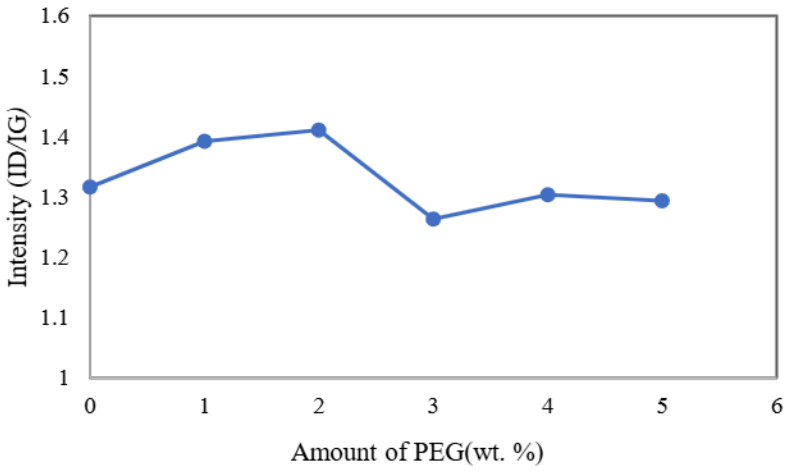

Fig. 9. The Raman I_D/I_G ratios of C-infiltrated ZTA composites containing 0 to 5 wt. % of PEG

4. Conclusions

This present study examined the addition of 0 to 5 wt. % of PEG to C-infiltrated and pure ZTA composites. Microstructural observations indicate that the addition of PEG to ZTA composites will create porous structures and facilitate the infiltration of C into the pores available on the surface. In the previous study [33], the highest density achieved was 3.787 g/cm³ for ZTA composites infused with solid carbon derived from Empty Fruit Bunch (EFB) without the addition of PEG. However, this study demonstrates a marked increase in density, reaching 4.145 g/cm³, attributed to the introduction of 3 wt.% polyethylene glycol (PEG) as a binder to create controlled pores that facilitate more efficient carbon infiltration during chemical vapor infiltration (CVI). It also reports the highest hardness of 1911 HV and fracture toughness of 6.98 MPa.m$^{1/2}$. This improvement highlights the effectiveness of the combined approach of pore formation and CVI carbon infiltration in enhancing both density and mechanical properties. The introduction of PEG in this study optimizes the

microstructural conditions for carbon infiltration, enabling better densification and mechanical reinforcement. However, excessive amounts of PEG (more that 3 wt.%) will agglomerate in a composite and adversely affect densification. Hence, reduce overall mechanical properties of the composite. This study holds significant relevance across a range of applications, especially for cutting tools, where enhancing its mechanical properties could prolong tool life and reduces maintenance costs.

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