

## SUSCEPTOR-ASSISTED RAPID MICROWAVE SINTERING OF Al-KAOLIN COMPOSITE IN A SINGLE-MODE CAVITY

The present research addresses the low-temperature sintering of 4% kaolin clay reinforced aluminium composite using susceptor-aided microwave sintering at 2.45 GHz frequency. Kaoline clay the naturally available mineral in the north-eastern regions of india. The study aims to convert this kaoline clay into the value added product with enhanced mechanical properties. The Al-x% Kaolin (x = 2, 4, 6, 8, 10) composite was fabricated through the powder metallurgy process by the application of 600 MPa compaction pressure. The composite corresponding to optimum ultimate tensile strength (U.T.S) was subjected to single-mode cavity microwave-assisted sintering by varying the sintering temperatures as 500°C, 550°C and 600°C. The effect of incorporating kaolin clay on the dielectric characteristics of composite powders, as well as the effect of sintering temperature on the microstructural changes and mechanical characteristics of Al-4%Kaolin composites were also examined. Results concluded that the addition of 4 wt% kaolin powder improves the dielectric characteristics of the composite powder. The maximum Hardness. Compression strength and U.T.S of 97 Hv, 202 MPa and 152 MPa respectively achieved for the Al-4% Kaolin composite sintered at 550°C. The higher fracture toughness of 9.56 Ma. m<sup>1/2</sup> reveals the ductile fracture for the composite sintered at 550°C.

*Keywords:* kaolin reinforcement; microwave sintering; mechanical properties; powder metallurgy

### 1. Introduction

In the present era, composites are predominantly used in the automobile sector to develop the components such as brake arms, piston rods and cylinder heads as the composites possess better resistance to corrosion, high ductility and tailorable properties [1,2]. Metal matrix composites can be synthesized by incorporating numerous reinforcements such as TiC, ZrC, ZrO<sub>2</sub> and SiO<sub>2</sub> etc. The high hardness and anti-wear characteristics of these reinforcements make the composite brittle and absence of self-lubricating properties [3]. In addition to this, the higher cost of ceramic reinforcements makes its utility in industrial applications. To eradicate its effect, nowadays metal matrix composites were synthesized by incorporating naturally available particles such as coconut shell particles, bamboo-leaf-ash, rice husk particles and mineral clays [4]. Composite can be fabricated by using liquid-state processing or solid-state processing techniques. The presence of higher porosities and the oxidation of powder particles during the liquid state melting leads to the initiation of agglomerations at the interface regions which leads to composite to fail at the lower intensity

of applied loads [5]. However, the powder metallurgy process of composite synthesis eliminates the oxidation phenomenon as the matrix and reinforcements were not undergone the melting process [6]. In the powder metallurgy process, the obtained composites from the compaction process are sintered through various sintering techniques such as conventional sintering, spark plasma sintering (SPS) and microwave-assisted sintering (MAS) techniques [7]. The heat dissipation from the core part of the particle to the outside leads to the initiation of strong bonds between the neighbouring atoms which accelerates the dislocation piling at the grain boundaries and improves the mechanical strength of the MAS composite when compared to conventional sintered composite [8]. K. Ravi Kumar et al., [9] studied the strength analysis of Al-ZrO<sub>2</sub>-coconut shell particle composite and the results indicate that the U.T.S of the synthesized composite was found to be 201 N/mm<sup>2</sup> by reinforcing 2 wt% CSA and 8% ZrO<sub>2</sub> particles. Incorporation of ZrO<sub>2</sub> particles of more than 10 wt% leads to decreases in the U.T.S of the composite due to the formation of brittle intermetallics. B. Praveen Kumar et al., [10] analyzed the stir-casted Al-Bamboo leaf ash (BLA) composite. Researcher findings reveal that the mohrs hardness

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and U.T.S of the synthesized composite found to increased up to 104 Mohrs hardness and 176 MPa with the incorporation of 4.5 wt% Cu and 5 wt% BLA particles. Ch. Hima girish [11] investigates the stir casted Al-Aloe vera, Al-Fly ash composite and the obtained results concluded that the an 55.62% enhancement in U.T.S of the Al-Aloe vera composite than the unreinforced aluminium.

Kaolin is an abundantly available mineral in the northeast regions of India. This mineral consists of ceramic compounds such as silicon oxide ( $\text{SiO}_2$ ), aluminium oxide ( $\text{Al}_2\text{O}_3$ ), Titanium oxide ( $\text{TiO}_2$ ) and Magnesium oxide ( $\text{MgO}$ ) which processes higher hardness and self-lubricating characteristics. Hence, this study explores the conversion of kaolin clay into a value-added product. The Kaolin particles were reinforced in the aluminium matrix as 2 wt%, 4 wt%, 6 wt% and 8 wt% to investigate the effect of kaolin reinforcement on the mechanical characteristics of the MAS sintered composite. Further, the composite with the optimal mechanical properties was subjected to a single-mode cavity MAS process by varying sintering temperatures to study the sintering temperature impact on the mechanical properties of the synthesized Al-Kaolin composite.

## 2. Materials and fabrication process

### 2.1. Preparation of composite powders

The aluminium and kaolin powders with an average particle size (APS) of 40  $\mu\text{m}$  were purchased from Nanoshell Pvt Ltd, Delhi. The constituent elements present in the As-received kaolin powders was shown in TABLE 1. The particle morphology, EDX spectrum and APS of aluminium and kaolin particles were depicted in Fig. 1. The ball milling was performed to the mixture of aluminium and x% kaolin ( $x = 2, 4, 6, 8$ ) powders by rotating the vial at 300 rpm for 2 h [12]. The powder mixture was surrounded by an Ar gas atmosphere to avoid aluminium powder oxidation during the milling.

### 2.2. Fabrication of the composite

The milled composite powders were filled in the H-13 steel die and then compacted by applying 600 MPa pressure using the hydraulic-operated pallet press by the application of 600 MPa

TABLE 1

Elemental composition of Kaolin reinforcement

| Elemental Constituents | $\text{SiO}_2$ | $\text{Al}_2\text{O}_3$ | $\text{TiO}_2$ | $\text{Fe}_2\text{O}_3$ | $\text{K}_2\text{O}$ | $\text{CaO}$ | $\text{MgO}$ | $\text{Na}_2\text{O}$ | L.O.I. |
|------------------------|----------------|-------------------------|----------------|-------------------------|----------------------|--------------|--------------|-----------------------|--------|
| Weight Percentage      | 45.6           | 38.4                    | 1.5            | 0.4                     | 0.18                 | 0.06         | 0.05         | 0.03                  | 13.8   |

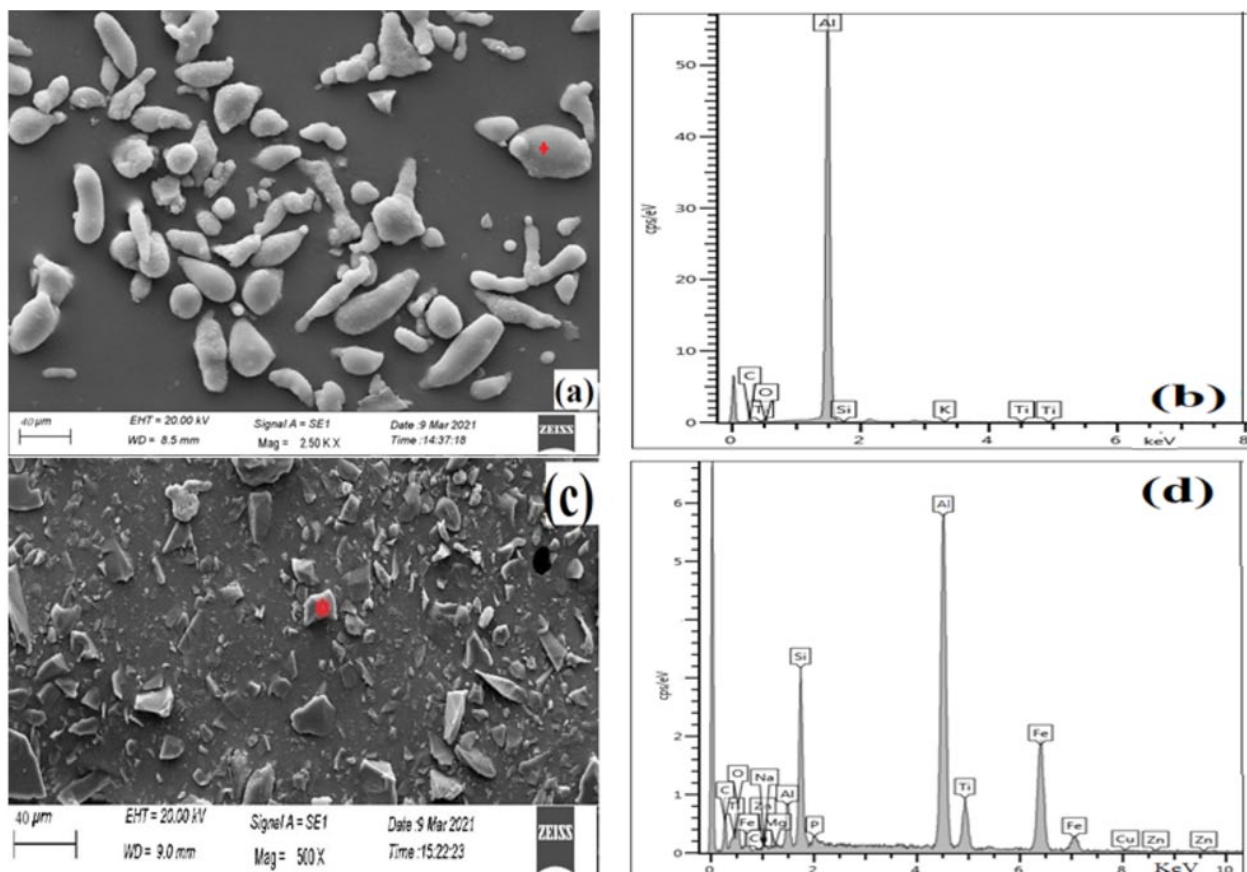


Fig. 1. (a) APS for aluminium (b) EDS Spectra for the aluminium and (c) APS for Kaolin and (d) EDS spectra for Kaolin particles

pressure [13]. The composites are synthesized by reinforcing the 2 wt%, 4 wt%, 6 wt% and 8 wt% kaolin particles. The compacted composites were microwave sintered at 500°C for 30 min with a power input of 1.5 KW and 2.45 GHz frequency to study the influence of wt % of kaolin on the mechanical properties of the composite [14,15]. The composite corresponding to maximum U.T.S was subjected to single-mode cavity microwave sintering (SAIREM, Chennai) with WR (Waveguide Rectangular) 340 waveguide by supplying 2 kW power with a frequency of 2.45 GHz. Composite samples were placed in a location where the electric field was presumably the strongest [16]. Croquesel et al., [17] and Cecilia et al., [18] stated the entire process, as well as the conditions for the single-mode cavity sintering of the composites. Fig. 2 depicts the synthesized composites sintered through the MAS technique. The codes assigned in this study are A0K, A2K, A4K, A6K and A8K represent the aluminium composite reinforced with 0 wt%, 2 wt%, 4 wt%, 6 wt% and 8 wt% kaoline particles. A4K500, A4K550, and A4K600 represent Al-4%Kaoline single-mode cavity MAS sintered at 500°C, 550°C and 600°C respectively.

### 2.3 Composite characterization methods

The dielectric properties of the as-received aluminium and kaolin powders were measured by using the network analyzer operating at a frequency of 0.5-3 GHz integrated with a dielectric kit probe [19]. Tensile and compression tests were performed as per ASTM E8 and ASTM E9 standards on the M30 model micro universal testing machine [20]. The ECONOMET VH1MD micro Vickers hardness tester was utilised to assess the hardness of the composite as per ASTM E384-16 specifications with the application of a 500 g load [21]. The presence of constituent phases was identified by performing a PANanalytical X-Ray diffraction machine. The microstructures of the composites were identified by using a scanning electron microscope (Carl Zeiss Ltd, U.S.A) integrated with an EDS analyzer by varying the accelerating voltage between 0.2 to 30 kV.

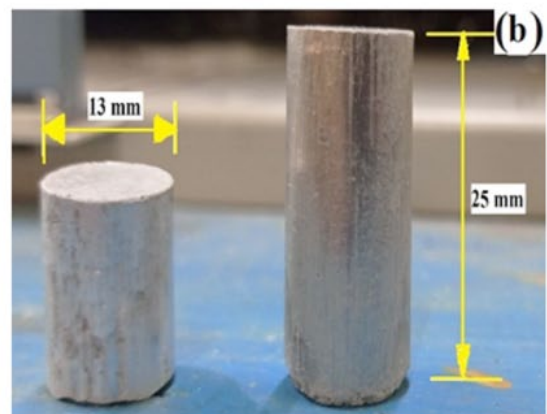
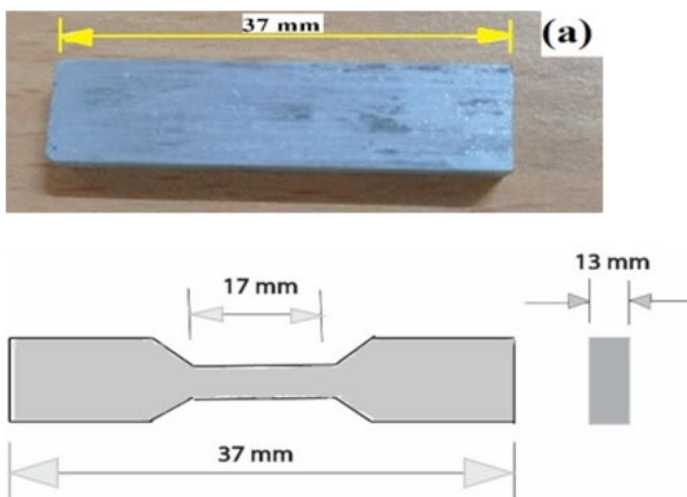


Fig. 2. Synthesized MAS composites for (a) Tensile test, (b) Hardness and Compression test

## 3. Results and discussion

### 3.1. Permittivity measurement of as-received powders

The dielectric properties of the as-received matrix and reinforcement powders were determined at 2.45 GHz frequency at ambient conditions after constrained tapping for 15 times and the obtained results were shown in TABLE 2 [19]. The addition of 4% kaolin powders to aluminium particles resulted in an enhancement of dielectric characteristics such as real, imaginary and loss tangent values.

TABLE 2

Dielectric properties of as-received powders

| Composition | Real ( $\gamma'$ ) | Imaginary ( $\gamma''$ ) | Loss tangent ( $tg\gamma$ ) |
|-------------|--------------------|--------------------------|-----------------------------|
| Al          | $1.33 \pm 0.01$    | $0.03 \pm 0.01$          | $0.004 \pm 0.01$            |
| Al-4%Kaolin | $2.18 \pm 0.01$    | $0.21 \pm 0.01$          | $0.012 \pm 0.01$            |

The loss tangent values represent the microwave absorbing capability of the given substance. From TABLE 2 it was clear that the addition of kaolin to the aluminium significantly improves the microwave absorbing behaviour of the composite powder to be sintered. The presence of aluminium oxide, silicon oxide and magnesium oxide in the kaolin powder makes the composite powder absorb the high intensity of microwaves during the microwave sintering process.

### 3.2. XRD and microstructural investigation

Fig. 3 depicts the XRD results for the as-received kaolin and aluminium powders. The obtained peaks were compared with JCPDS card numbers. The peaks corresponding to  $\text{SiO}_2$  (JCPDS card No: 46-1045 [22,23]),  $\text{MgO}$  (JCPDS card No: 59-7746 [24,25]),  $\text{Al}_2\text{O}_3$  (JCPDS card No: 00-046-1212 [14]) and  $\text{TiO}_2$  (JCPDS card No: 21-1276 [26]) confirms the as-received powder is kaolin and the peaks other than these con-

stituents were not observed, which reveals the kaolin is free from the impurities. The XRD analysis of the single-mode cavity MAS composite at 600°C shows the presence of an  $\text{Al}_2\text{Cu}$  peak as depicted in Fig. 4. The identified  $\text{Al}_2\text{Cu}$  peak (JCPDS card No: 25-0012 [27]) was matched with the peak identified by the researcher Gatea et al., [28]. Increasing the sintering temperature beyond 550°C accelerates the chemical reactions between the kaolin and aluminium powders and prons to the diffusion of atoms among the adjacent particles. This phenomenon predominantly initiates the agglomerations formation at the boundaries of the kaolin and aluminium particles [29]. The generated brittle  $\text{Al}_2\text{Cu}$  agglomeration decreases the load withstanding capability of the composite and causes failure of the specimen at the lower magnitude of applied loads. Despite this, the presence of low MAS temperature (550°C) leads to uniform internal heat dissipation which eliminates the agglomeration formation in the composite.

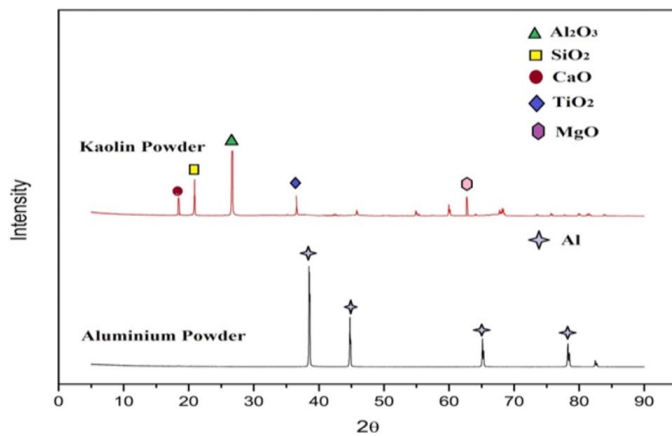


Fig. 3. XRD peaks for the As-Received Aluminium and Kaolin powder

The interface chemical bonding between the kaolin and aluminium particles plays a predominant role in controlling the mechanical characteristics of the composite. To investigate

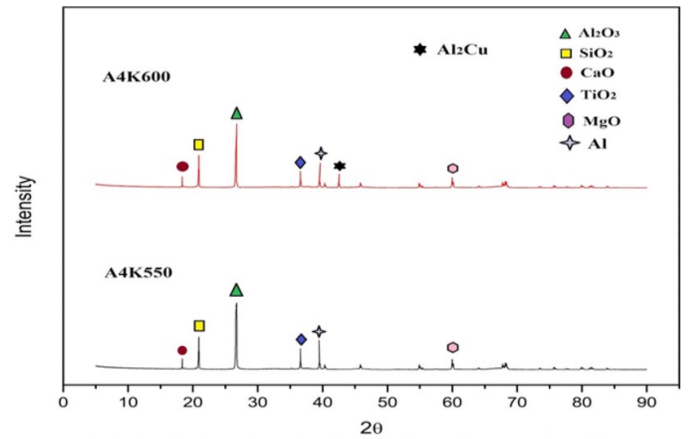


Fig. 4. XRD peaks for the single-mode cavity sintered Al-Kaolin composite at (a) 550°C and (b) 600°C

the kaolin reinforcement distribution in aluminium matrix SEM analysis was performed on the single-mode cavity MAS composite at 550°C and 600°C (Refer Fig. 5.) From Fig. 5, It was observed that the kaolin particles in the aluminium matrix were dispersed uniformly with clean interfaces and no agglomerations were identified, which indicates that the no chemical interaction among the adjacent particles during the microwave sintering. However,  $\text{Al}_2\text{Cu}$  agglomerations were identified in the SEM image for the composite sintered at 600°C due to the presence of accelerated diffusion of atoms and the initiation of chemical interactions at higher temperatures [30,31]. The reinforced kaolin particles absorb the higher intensity of microwaves at elevated temperatures and increase the kinetic energy of the particles located near the grain boundaries [15]. This phenomenon generates the brittle  $\text{Al}_2\text{Cu}$  agglomerations along the grain boundaries as shown in Fig. 5(b). The oxides layers were observed on the surfaces of the MAS composite sintered at 600°C due to the interactions between the agglomerations and the aluminium particle. The oxidized surface of the composite and the SEM image for the oxidized surface was depicted in Fig. 6.

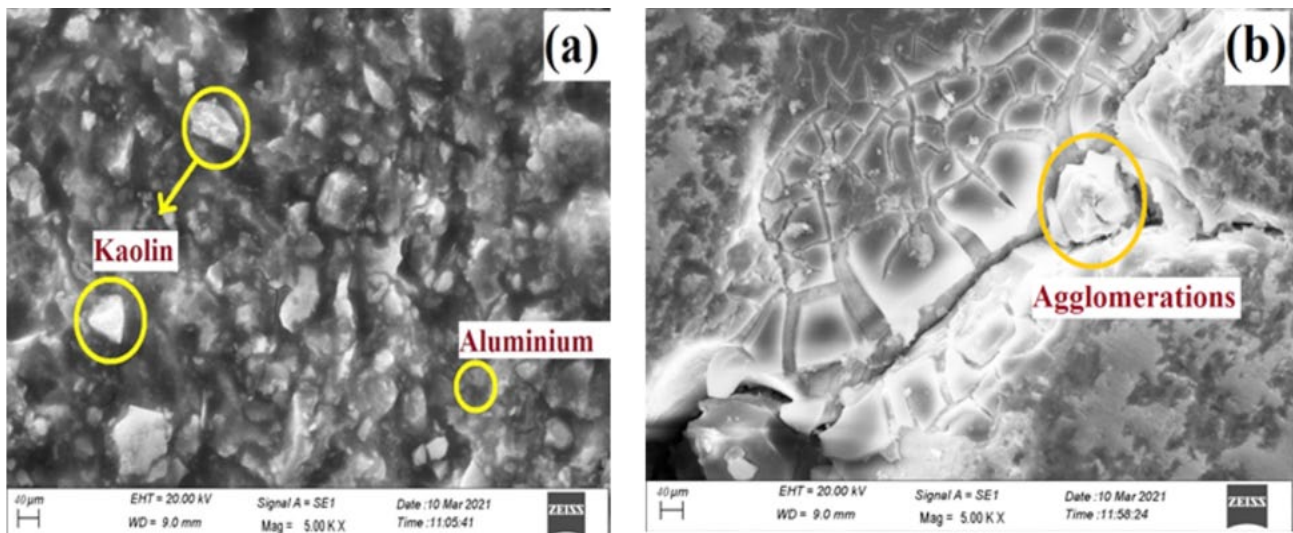


Fig. 5. SEM images for single-mode cavity MAS composite at (a) 550°C and (b) 600°C

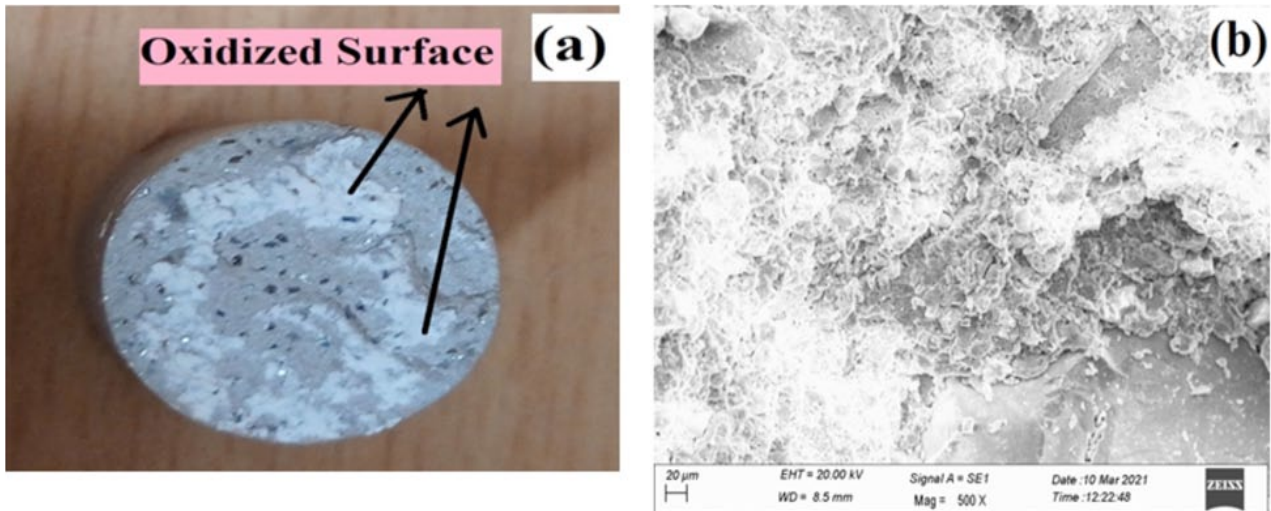


Fig. 6. (a) Oxidized surface of the composite sintered at 600°C, (b) SEM image for the oxidized surface

### 3.3. Mechanical properties

The plastic strain between the kaolin and aluminium particles affects the hardness and interface strength of the synthesized composite. The U.T.S of the MAS Al-Kaolin composite was shown in Fig. 7. Results reveal that with the incorporation of kaolin particles from 2 wt % to 4 wt %, the U.T.S was improved from 102 MPa to 136 MPa due to the availability of harder  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  compounds in the kaolin reinforcement [25,32]. Beyond the incorporation of 4 wt % kaolin particles leads to decrement in U.T.S due to the formation of agglomerations at the interfacial regions. The composite corresponding to optimal U.T.S (Al-4% Kaolin) was subjected to single-mode microwave sintering at the sintering temperatures of 500°C, 550°C and 600°C and the obtained compression strength and U.T.S results were depicted in Fig. 8. From the results, it was clear that an 11.76% enhancement in U.T.S of the single-mode cavity MAS composite at 550°C than the MAS sintered Al-4%kaolin composite. The decrease in trend for the U.T.S was obtained when the sintering temperature was raised beyond 550°C as shown in Fig. 8. This was attributed due to the accelerated chemical interactions at the kaolin and aluminium grain boundaries which in turn generates the brittle agglomerations of  $\text{Al}_2\text{Cu}$ . These  $\text{Al}_2\text{Cu}$  particles were brittle characteristics and processed a lower thermal expansion coefficient compared to the aluminium matrix which leads to uneven expansions during the MAS process [33]. During the application of load crack initiation occurs along the boundaries of  $\text{Al}_2\text{Cu}$  agglomerations and the aluminium particles and makes the composite fail at the lower intensity of externally applied load [20].

The variation of microhardness of the single-mode cavity MAS composite with sintering temperatures was depicted in Fig. 9. Results reveal that the hardness of the Al-4%Kaoline was improved from 83 Hv to 97 Hv with a rise in the sintering temperature from 500°C to 550°C. i.e an enhancement of 16.86% hardness was obtained at 550°C than the composite sintered at 500°C. This enhancement in hardness was due to the reinforc-

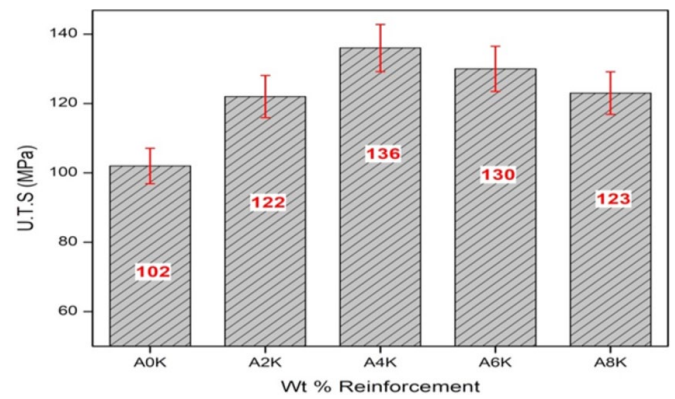


Fig. 7. Variation of U.T.S of MAS composite with wt% of kaolin particles

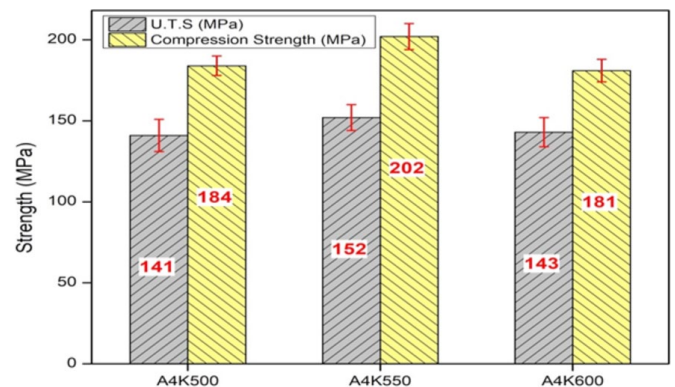


Fig. 8. Variation of Compression strength and U.T.S of the Al-Kaolin composite

ing the stiffer kaolin particles (comprises of harder  $\text{SiO}_2$ ,  $\text{TiO}_2$  and  $\text{CaO}$ ) into the ductile aluminium matrix. Despite this, the added kaolin particles pile at the grain boundaries and obstruct the movement of dislocations from one grain to another grain during the indentation test which improves the hardness of the composite [34]. However, increasing the sintering temperature beyond 550°C is prone to decrement in hardness from 97 Hv

to 85 Hv. The presence of difference in linear thermal expansion coefficient among the generated agglomerations and aluminium matrix leads to uneven expansions of the interface particles and leads to void generation along the grain boundaries and reduces the hardness of the composite at the higher sintering temperatures [35,36].

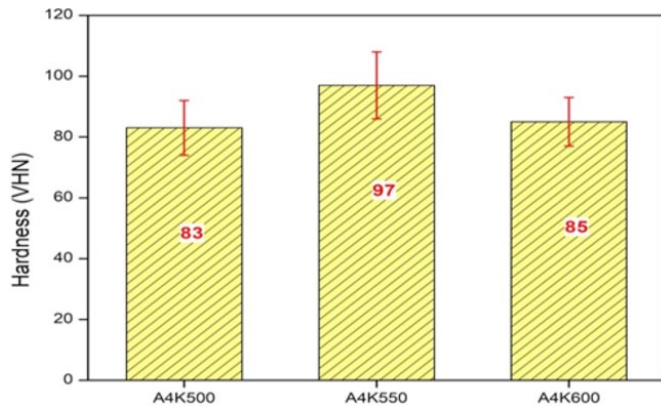


Fig. 9. Variation of Microhardness of the composite with different sintering temperatures

### 3.4. Toughness of the synthesized composites

The energy absorbing capacity of the synthesized composite was assessed by performing the Charpy test and the obtained results were shown in Fig. 10. There was a 45.7% increment in Toughness of the composite sintered at 550°C than the composite sintered at 500°C. The Magnesium constituent present in the kaolin clay enhances the wettability among the neighbouring particles and creates the strong interfacial bond between adjacent particles, thereby, the toughness of the composite was improved from 6.56 MPa.m<sup>1/2</sup> to 9.56 MPa.m<sup>1/2</sup> with increasing the MAS temperature from 500°C to 550°C. However, the generated intermetallics make the composite brittle and reduce the energy-absorbing capability by 31.4% at 550°C sintering temperature than the composite sintered at 500°C. For each wt% of kaolin

reinforcement, the experiment was performed on four samples to obtain accurate results. The error bars shown in Fig. 10 lie within the ±5% acceptable deviation range.

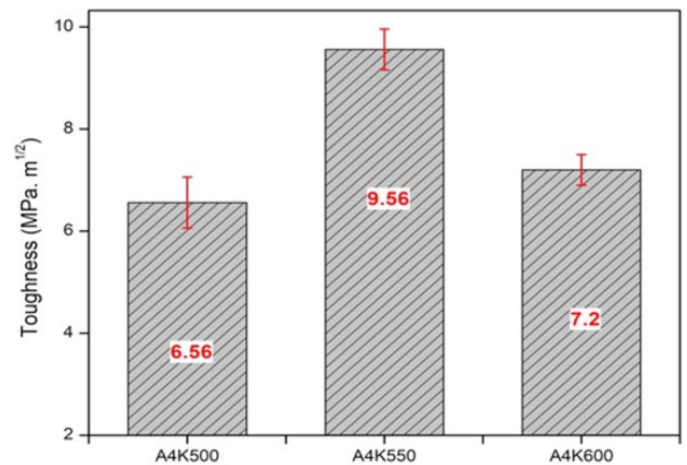


Fig. 10. Toughness of the single-mode cavity MAS sintered composite at different sintering temperatures

### 3.5. Fractography investigation

Fractography is a failure assessment technique used to examine the fracture surface of materials. In this study, Fractography was performed on the tensile fractured Al-Kaolin composites to identify the mode of fracture for the tensile specimens. Fig. 11 represents the fractography images for the composites sintered at 550°C and 600°C respectively. Dimples were identified on the composite sintered at 550°C which confirms the ductile fracture of the composite [37]. However, the presence of Al<sub>2</sub>Cu agglomerations in the composite sintered at 600°C leads to cleavage facts on the fractured surface. These cleavage facts initiate the intergranular crack initiation and make the brittle fracture in the sample [2]. The ductile fracture in the composite sintered at 550°C makes the better utilization of the composite in automobile and industrial applications.

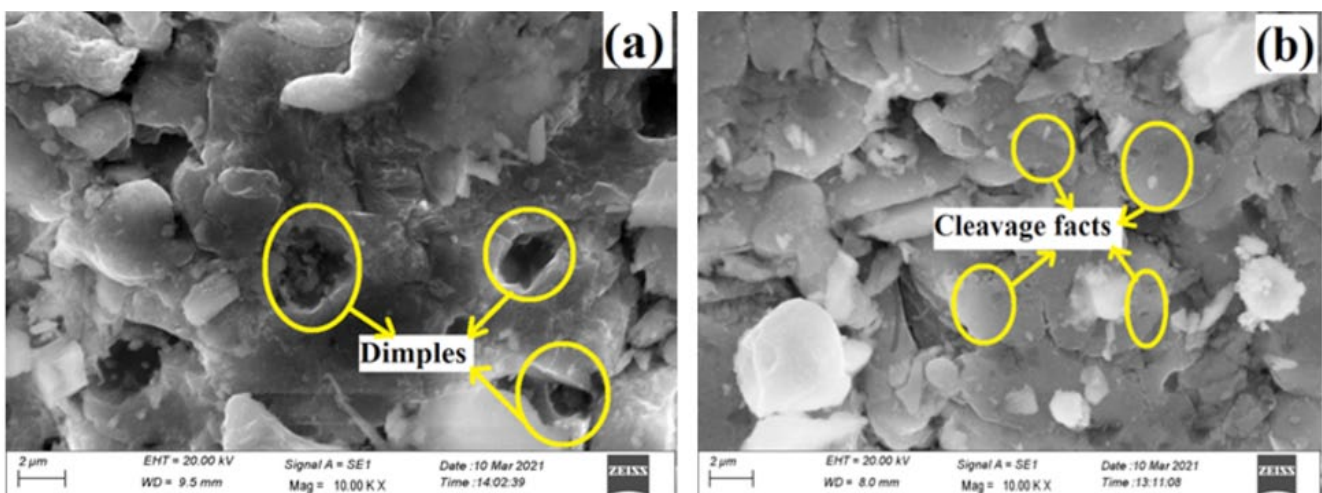


Fig. 11. SEM images for the tensile fractured Al-Kaolin composite at (a) 550°C and (b) 600°C

#### 4. Conclusions

In this research, Al-Kaolin composite was sintered through single-mode cavity microwave-assisted sintering operating at a microwave frequency of 2.45 GHz with 600 W input power. The obtained results were summarized below.

- The permittivity analysis results concluded that the incorporation of 4 wt% kaolin improved the dielectric loss factor of the composite powder (Al-4%Kaolin) which suggests that the composite can be microwave sintered at different sintering temperatures.
- The maximum U.T.S of 136 MPa has been obtained for the microwave sintered 4 wt% kaolin reinforced aluminium composite.
- With increasing the single-mode cavity sintered microwave sintering temperature from 500°C to 550°C, the compression strength and U.T.S was found to be 202 MPa and 152 MPa respectively due to the absorption of microwaves and the uniform kaolin particles dispersion in the aluminium matrix.
- The Al<sub>2</sub>Cu agglomerations generated at the grain boundaries of adjacent particles lead to decreases in the compression and U.T.S of the composite when the sintering temperature increases beyond 550°C.
- The fracture toughness of the composite sintered at 550°C was increased to 45.73% compared to the composite sintered at 500°C due to the absence of agglomerations at the grain boundaries.
- Finally, The Al-4%Kaolin composite sintered through single-mode cavity microwave sintering at 550°C exhibits superior properties compared to the composite sintered at 500°C and 600°C sintering temperatures.

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