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Effects of Sintering Temperature on the Flexural Strength and Shrinkage of Feldspar Reinforced Clay Composite for the Vitrified Clay Pipe Application

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Abstract

Clay is a common material that was used to manufacture the green sewerage pipe, vitrified clay pipe (VCP). As known, clay is a ceramic material that behaves in a brittle manner. In the current study, addition of appropriate feldspar reinforcement in clay material and heat treatment by sintering method were focused to improve the shrinkage and flexural strength of ceramic clay composite for future material selection in VCP manufacturing at JPC-Intan Sdn. Bhd. Investigation on five different composition ratios between feldspar and clay was conducted. The ratio of clay/feldspar consists of 75/25, 80/20, 85/15, 90/10 and 95/5 (wt%). Subsequently, five different sintering temperatures were subjected to the ceramic clay composite, from 950, 1000, 1050, 1100 and 1120 °C. Shrinkage analysis, flexural test and Scanning Electron Microscopy (SEM) towards fracture surface were performed to the clay and ceramic clay composites. Thermogravimetric Analysis (TGA) and Energy-Dispersive Spectroscopy (EDS) analysis were carried out to the dry clay and fine feldspar powders. Shrinkage of ceramic clay composite apparently temperature dependent. Shrinkage was increased by increasing the sintering temperature for all ceramic clay composites. Flexural strength at sintering temperatures of 1000 °C and above was significantly higher compared to the flexural strength at low sintering temperature of 950 °C for each composition clay/feldspar composite. The highest flexural strength was indicated at ratio of 85/15 at 1100 °C. Plus, all samples were significantly behaved in brittle manner. Pores were obviously seen in fracture surface of clay/feldspar at 950 °C. Meanwhile, pores were reduced at the fracture surface of clay/feldspar composite when sintered at 1100 °C. Less porosity on fracture surface was tended to increase the percentage of shrinkage at high temperature and, also affects the flexural strength with the maximum value achieved.

1. Introduction

Commonly, clay is a material used to manufacture the green product of vitrified clay pipe (VCP). VCP has good chemical and abrasive resistances suitable for sewerage application, which is applied under a corrosive sewerage environment. VCP is manufactured from clay material, and it has been subjected to a high firing temperature in the kiln, to improve the strength and quality of VCP. VCP is a low pressure for large diameter applications with the advantages of low thermal expansion, long life cycle, raw materials availability, and good corrosion resistance [1]. The significant elements composition in clay started with silicon (Si), aluminium (Al), iron (Fe), titanium (Ti) and potassium (K). These elemental contents were detected from clay at several locations of North-West Peninsular Malaysia [2]. Meanwhile, Bakil et. al. (2017) and Natrah et. al. (2019) reported the chemical composition of Malaysia's stoneware clay consists of silicon oxide in major, aluminium oxide and others [3-4]. At feldspar, silicon oxide was also detected as a higher element composition and followed by the element of aluminium oxide including other elements [5]. In ceramic product manufacturing, feldspar is generally used as a flux, which lowers the melting temperature of another material.

El-Shimy et. al. (2014) investigated the addition of cullet (ground glass waste) into clay and grog (waste of defect and broken VCP) for the application of VCP. They found that the highest modulus of rupture (MOR) was reported at 10% glass in clay and grog under a firing temperature of 1050 °C [6]. Abbey et. al. (2020) studied the mechanical properties of the fiber reinforced clay blended with cement, lime, ground granulated blast slag and micro silica. The unconfined compressive strength of the clay sample was significantly lower compared to samples of Portland cement, polypropylene and glass fibers reinforced clay [7]. El Sherbiny et. al. (2004) determined that the MOR of unfired clay was linearly decreased at increasing the addition of grog. Also, the addition of grog at more than 20% indicated low drying shrinkage. Apparently, adding feldspar in 10% grog sample resulted in higher drying shrinkage compared to the cement dust in 10% grog sample. Meanwhile, MOR of feldspar in 10% grog-clay sample indicated lower than that of cement dust in 10% grog-clay sample [8]. Al Hoseny et. al. (2018) studied the effect of sludge and cullet addition on the MOR for the application of clay pipe. The MOR was indicated lower at increasing the percentage of sludge at every percentage of cullet addition in clay. However, the addition of 10% cullet was found higher at all percentages of sludge addition compared to the other percentages of cullet [9]. Shrinkage and flexural strength were reported higher at high temperature for stoneware clay without reinforcement [3,10]. Bakil et. al. (2017) found that the higher flexural strength was at 5 wt% of soda lime silica reinforced stoneware clay under the sintering temperature of 1100 °C [3]. Meanwhile, Natrah et. al. (2019) reported the high flexural strength was at 10 wt% of borax decahydrate reinforced stoneware clay under the sintering temperature of 1050 °C. However, the highest flexural strength was at 5 wt% of soda lime silica reinforcement under the sintering temperature of 1100 °C compared to the 10 wt% of borax decahydrate reinforcement under the sintering temperature of 1050 °C. Thus, the addition of soda lime silica and borax decahydrate reinforcements on stoneware were acceptable [4].

According to the VCP industry, JPC-Intan Sdn. Bhd., production of green VCP is mainly manufactured using clay from Johor, Malaysia with the addition of grog. VCP is fired at high temperature to improve its quality and durability. For the research interest, the effect of feldspar reinforcement and sintering temperature on the shrinkage and flexural strength of ceramic clay composites was focused on the current study. As stated, feldspar is an expensive material to reinforce in VCP material [6, 8]. Currently, high energy consumption has led to the high cost in the VCP manufacturing process, especially in the firing stage. Due to this problem, by the best combination of feldspar reinforced clay, an investigation on the minimum feldspar reinforcement in appropriate firing temperature was conducted. These findings are potentially to decrease the cost of reinforcement material for the manufacturing process of green VCP in the future. Furthermore, the best composition of clay/feldspar composite with good flexural strength at low sintering temperature also aimed to reduce the cost of energy consumption at the firing stage during the VCP manufacturing process.

2. Materials and Methods

The JPC-Intan Sdn. Bhd. supplied the red clay and light brown feldspar for this research. The origin of clay was taken from Johor, Malaysia. This clay was then added with 17 wt% of grog, where grog was known as a recycled waste grain of VCP and clay products [8]. Meanwhile, the source of fine feldspar powder was from Pahang, Malaysia. The experimental works of TGA and EDS analyses, shrinkage and flexural test are shown in Figure 1. Thermogravimetric Analysis (TGA) was carried out to dry clay powder and fine feldspar powder with a setting temperature of 1300 °C. In addition, elemental analysis was conducted using Energy-Dispersive Spectroscopy (EDS) on the dry clay powder and fine feldspar powder. Five different composition ratios between clay and reinforcement of feldspar were 75/25, 80/20, 85/15, 90/10 and 95/5 wt%. At first, clay was dried in the oven at 120 °C within 24 hours. Next, it was crushed to become dry clay powder. For each ratio, the dry clay powder and fine feldspar powder were crushed and mixed using the rotary ball milling and glaze mill machines. The 10g of powder mixture was uniaxially pressed at 40MPa to form the rectangular bar sample with length, width and height of 65, 12 and 7 mm, respectively. Subsequently, five different sintering temperatures were subjected to the ceramic clay composites, from 950, 1000, 1050, 1100 and 1120 °C for 2 hours in the conventional furnace. Meanwhile, heating and cooling rates were set at 120 °C/hr. Shrinkage was analyzed using the measurement of sample sizes of rectangular bar at before and after heat treatment.

A Universal Testing Machine (UTM) with a capacity of 10kN load cell was used to perform the flexural test under a three-point bending test configuration with a crosshead speed of 0.05 mm/min to ceramic clay composites. The standard of ASTM C1161 was referred to conduct the flexural strength of ceramic clay composites at ambient temperature [11]. At last, all the samples were sputtered and coated with gold prior to scanning electron microscope (SEM) analysis to examine the fracture surface after the flexural test.

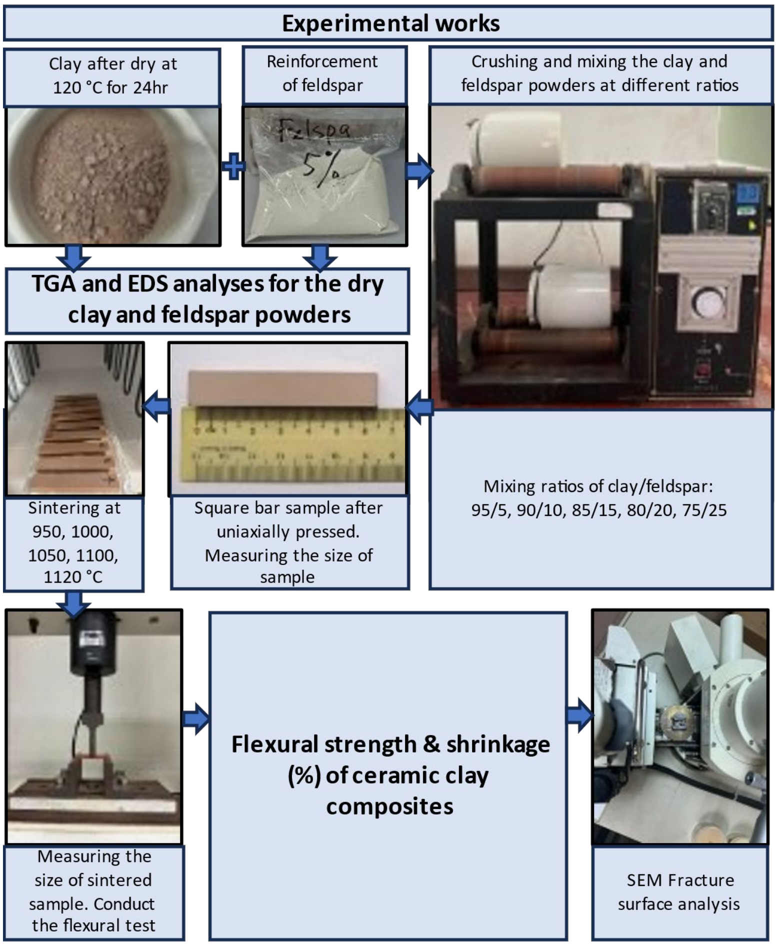


Fig. 1 *Experimental works*

3. Results and Discussion

3.1 Thermogravimetric Analysis (TGA) and Elemental Analysis of Dry Clay Powder and Fine Feldspar Powder

TGA analysis for dry clay powder and fine feldspar powder is shown in Figure 2. Thermal analysis of clay powder shows the release of water at around 500 to 600 °C. The decomposition of carbon is shown at around 700 to 800 °C. The percentage of weight loss in clay when heated until 1300 °C is 10.1%. Fine feldspar powder shows that the water release occurred at around 100 to 400 °C. The graph shows that the feldspar powder is unstable when heated until 1300 °C where the weight loss is 3.3%. However, Abd Bakil et. al (2017) reported the percentage of weight loss for stoneware clay was 4%. The addition of borax decahydrate in stoneware clay at 5 and 10 wt% was 4.43 and 4.47 % of weight loss, respectively [10]. The highest weight loss for porcelain clay and alkali-resistant glass-reinforced porcelain clays was between 450 °C to 800 °C with 61% to 75% [12].

Figure 3 shows the element composition of dry clay powder and fine feldspar powder. The main weight percentage of these two materials was oxygen in major. Then, silicon, aluminium and other elements were traced in both clay and feldspar powders.

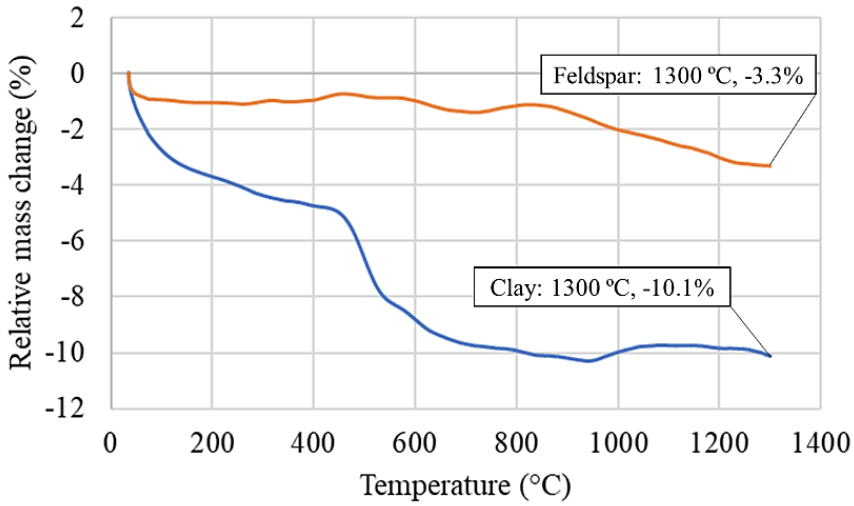
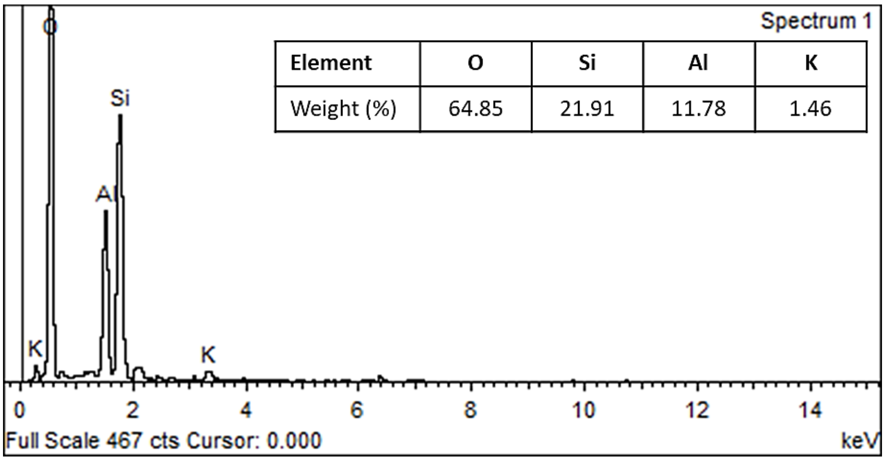
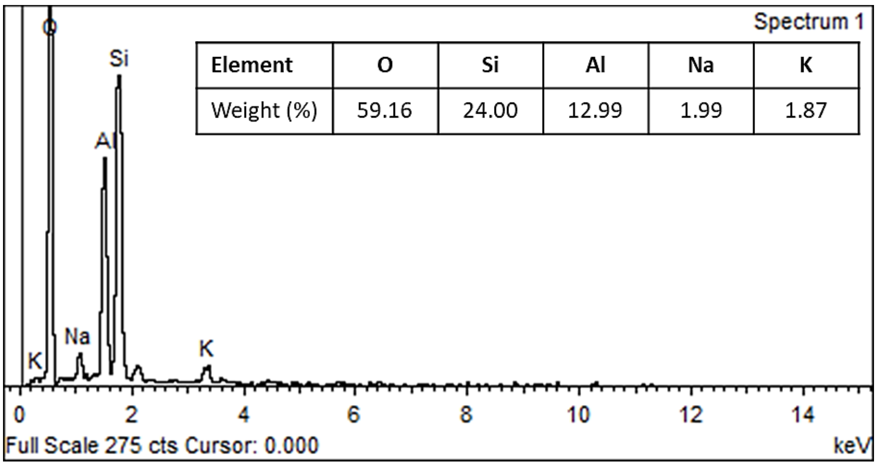


Fig. 2 *TGA analysis of dry clay powder and fine feldspar powder*



Element	O	Si	Al	K
Weight (%)	64.85	21.91	11.78	1.46

(a)



Element	O	Si	Al	Na	K
Weight (%)	59.16	24.00	12.99	1.99	1.87

(b)

Fig. 3 *Elemental analysis (a) Dry clay powder; (b) Fine feldspar powder*

3.2 Shrinkage of Feldspar Reinforced Ceramic Clay Composites

The effect of composition ratio and sintering temperature toward shrinkage of clay/feldspar composites is shown in Figure 4. The shrinkage of clay/feldspar composites was significantly temperature dependent. Shrinkage was increased at increasing the sintering temperature for all clay/feldspar composites. On average, shrinkage was found higher in clay without feldspar reinforcement, while shrinkage was lower at 75/25 ratio of all clay/feldspar composites. El Sherbiny et. al. (2004) reported that the addition of feldspar at 2 to 10 wt% in unfired clay with

10% grog was slightly decreased from around 4.9 to 5.8 % of drying shrinkage. However, addition of cement dust in unfired clay of 10% grog significantly decreased the percentage of drying shrinkage [8]. Bakil et. al (2017) reported the percentages of linear shrinkage were between 7.5 to 11.5% for soda lime silica reinforced stoneware clays, while around 8.3% for stoneware clay without reinforcement under sintering temperature of 1100 °C [3]. Next, linear shrinkage of borax decahydrate reinforced stoneware clays were at 7 to 10.2%, and 10% shrinkage at stoneware clay under sintering temperature of 1150 °C [10]. Drying shrinkage was reported to decrease at 25 and 30% of glass powder added, where glass powder addition is known tend to reduce the shrinkage for ceramic bodies [6]. Subsequently, it found that the percentage of volume shrinkage was high at alkali resistant glass reinforced porcelain clays for all sintering temperatures compared to the percentage of volume shrinkage for clay porcelain without reinforcement at all temperatures. The higher volume shrinkage was indicated at 1200°C with a range of 28 to 32% for alkali resistant glass reinforced porcelain clays [12].

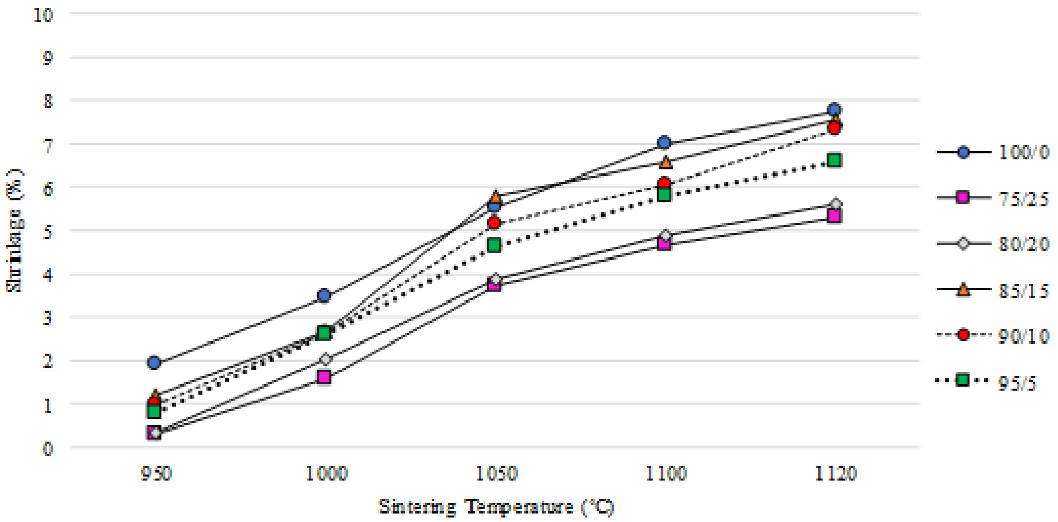


Fig. 4 *Shrinkage of clay and clay/feldspar composites in various weight percentage ratios at various sintering temperatures*

3.3 Flexural Strength of Feldspar Reinforced Ceramic Clay Composites

The effect of composition ratio and sintering temperature toward flexural strength of clay/feldspar clay composites is indicated in Figure 5. Based on the current result, flexural strength at sintering temperatures of 1000 °C and above were significantly higher compared to the flexural strength at low sintering temperature of 950 °C for each composition clay/feldspar composite. The highest flexural strength is indicated at a ratio of 85/15 under sintering temperature of 1100 °C with 52 MPa. The following is a ratio of 95/5 under sintering temperatures of 1100 and 1120 °C shows the second highest of flexural strength with 46 MPa. In overall, sintering temperatures at 1050 °C and above indicate the flexural strength was more than 30 MPa. Moreover, all clay and clay composites were significantly behaved in brittle characteristic, where all samples started from elastic deformation to brittle at increasing load and displacement as shown in Figure 6. Similar behavior was also reported by Xiao et. al (2023) for the perfect and defective kaolinite clay under tension and compression conditions. Failure deformation of kaolinite clay crystals under external forces was due to the breakage of bonds between atom [13]. El Sherbiny et. al. (2004) reported that addition of feldspar at 2 to 10 wt% for unfired samples with 10% grog was around 690 to 740 kPa of MOR. Meanwhile, addition of cement dust as reinforcement for unfired samples was from 4.1 to 5.4 kPa [8]. The highest MOR of glass addition in unfired clay samples was reported at 5% of glass addition with 2.1 MPa [6]. Sintered sample from this study was found higher in flexural strength compared to the unfired clay composites. Natrah et. al. (2019) reported that the MOR of stoneware clay was increased with increasing the sintering temperature. However, the highest MOR was indicated at 5 wt% of soda lime silica reinforced stoneware clay [4]. Abbey et. al. (2020) reported that elevated curing temperatures on polypropylene-reinforced clay-binder and glass fibre-reinforced clay-binder mixtures improved the unconfined compressive strength. Appropriate composition of polypropylene and glass fibre contents also enhanced the unconfined compressive strength of fibre-reinforced cement-clay mixture [7].

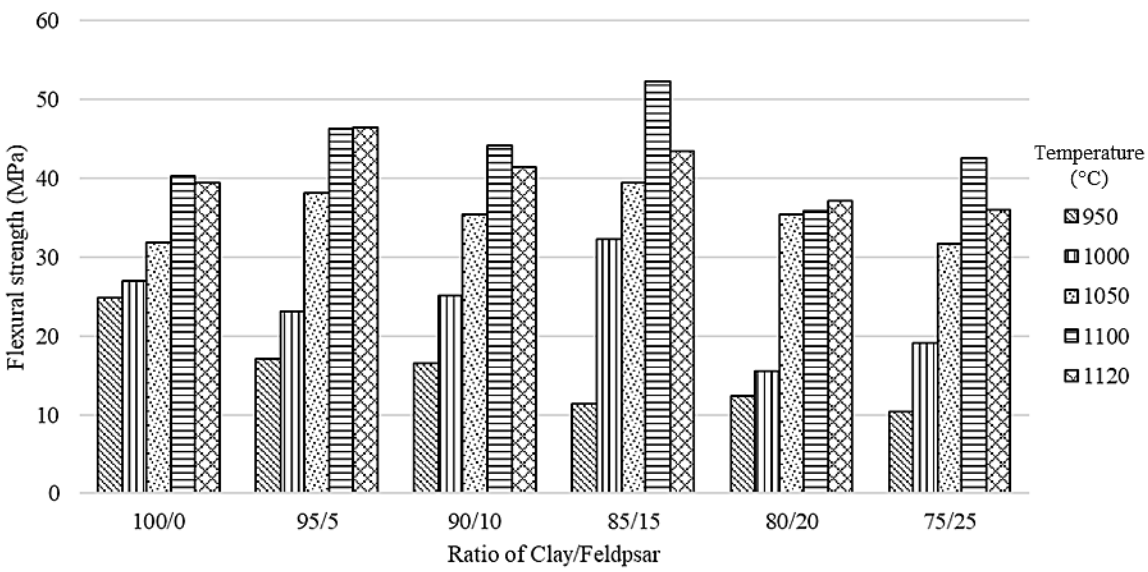


Fig. 5 *Flexural strength of clay and clay/feldspar composites in various weight percentage ratios at various sintering temperatures*

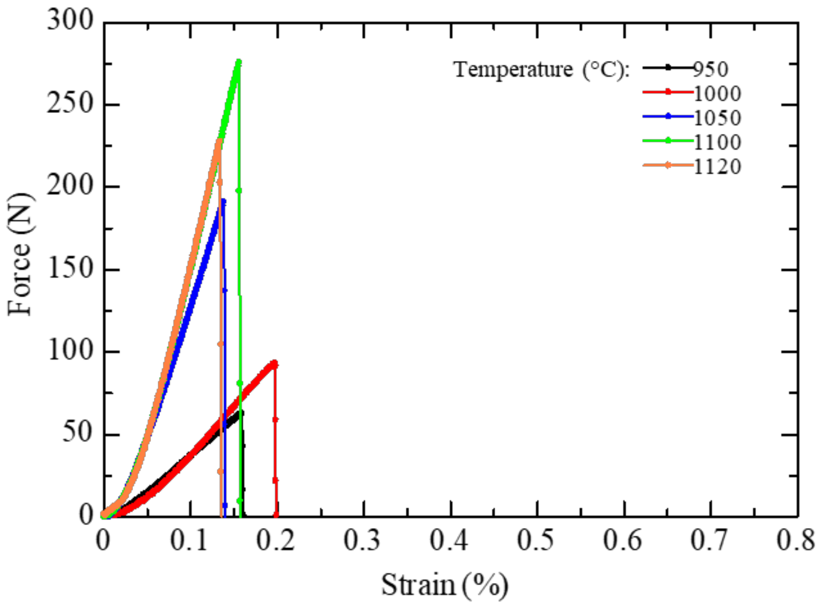


Fig. 6 *Force – strain curve of 85/15 clay/feldspar composite at different sintering temperatures*

3.4 Fracture Surface Micrograph

Fracture surface of 85/15 clay/feldspar composite under temperatures of 950 and 1100 °C is shown in Figure 7. It is proof that the fracture surface of ceramic clay composite was significantly temperature dependent. Pores are obviously seen in the fracture surface of clay/feldspar under temperature at 950 °C. Meanwhile, pores were reduced at fracture surface of clay/feldspar composite under temperature at 1100 °C. To relate, it was found that the less porosity on fracture surface tended to increase the percentage of shrinkage at high temperature. Consequently, flexural strength was increased at high temperatures. In the previous studies, the open porosity and large void formation were clearly seen in microstructure of alkali resistant glass reinforced porcelain clay under sintering temperature of 900 °C, while complete vitrification occurred at 1200 °C by elimination of porosity and void to generate a dense body for all composition samples. Thus, high shrinkage at high temperature was due to the complete densification in sample [12, 14]. Percentage of apparent porosity was also reported lower, while density was increased at high temperature of 1200 °C for all composition of alkali resistant glass reinforced porcelain clay [15].

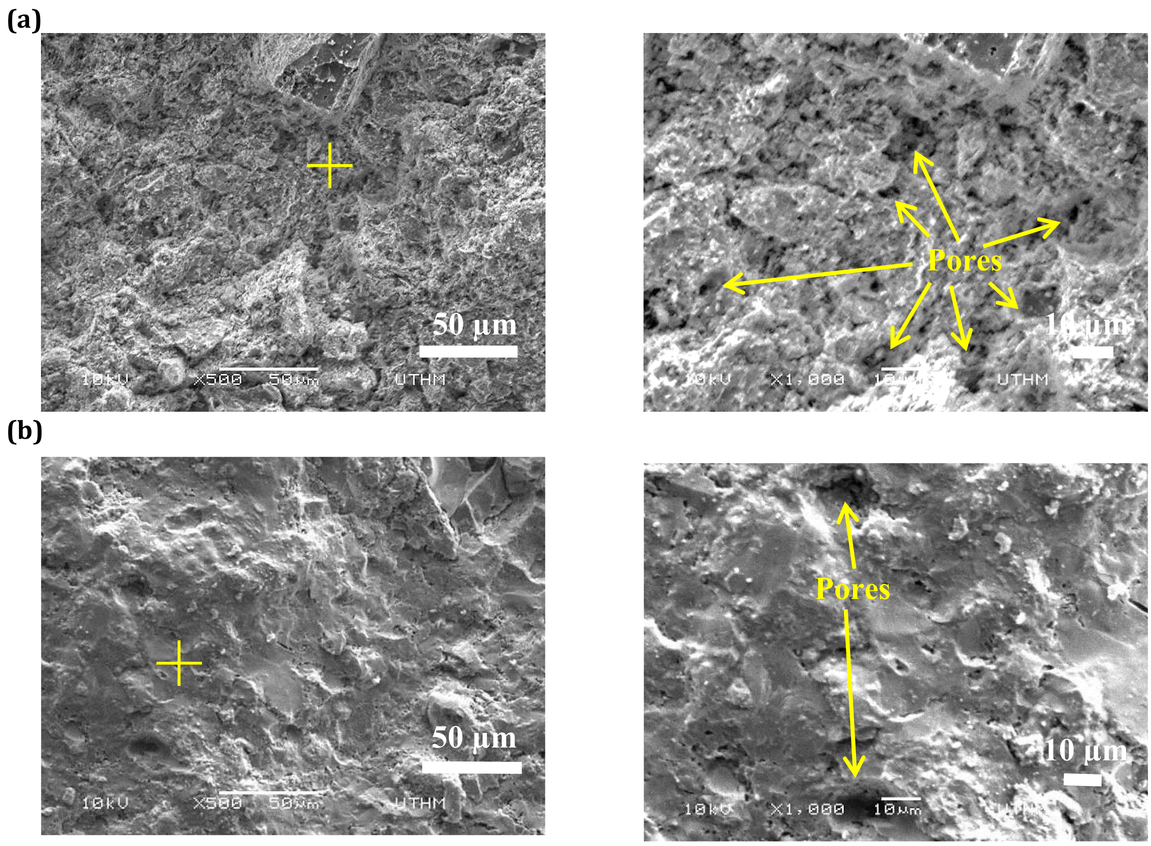


Fig. 7 *SEM images of (a) 85/15 composition of clay/feldspar under temperature of 950 °C at 500 and 1000 magnifications; and (b) 85/15 composition of clay/feldspar under temperature of 1100 °C at 500 and 1000 magnifications*

4. Conclusion

In summary, shrinkage of feldspar reinforced ceramic clay composites was significantly temperature dependent, where all the samples increased with increasing the sintering temperature. On average, the maximum shrinkage was indicated by clay samples without reinforcement, while shrinkage was found lower at 75/25 ratio of all ceramic clay composites. Flexural strength was significantly higher at sintering temperatures above 1000 °C than that of flexural strength at low sintering temperature of 950 °C for each composition clay/feldspar composite. The highest flexural strength was indicated by 85/15 ratio of clay/feldspar at 1100 °C with 52 MPa. All clay and ceramic clay composites were behaved in a brittle manner. The failure deformation occurred was due to the breakage of bonds between atom of clay and ceramic clay composites. Pores in fracture surface were shown reduced at fracture surface of clay/feldspar composite under temperature of 1100 °C. Hence, increasing the sintering temperature has affected the clay/feldspar composite by reducing the pores on the fracture surface, increasing the percentage of shrinkage, and the flexural strength.

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Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

The authors are responsible for the study conception, research design, data collection, data analysis, result interpretation and manuscript drafting.

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