CHAPTER 5: CONCLUSIONS AND SUGGESTIONS FOR FUTURE WORK

5.1 Conclusions

The optimized forsterite synthesis method was developed by tweaking the parameters of the common process steps to form forsterite after analyzing methods and results from previous studies. The main differences were applying ultrasonic bath during powder mixing process, set a fixed ball milling duration of 3 hours, and no heat treatment prior final sintering phase. Four sintering temperature, 1200°C, 1300°C, 1400°C and 1500°C were applied for sintering the forsterite compacts with three different holding times for comparison. The three holding time consists of 2 hours (Group 1), 1 hour (Group 2) and 1 minute (Group 3) with 10°C/min ramp rate. The following points are concluded from this study:

1. The additional 22 minutes ultrasonic bath increased the homogenization effect on the mixture contributed to the formation of forsterite without any secondary phase with all four sintering temperature range (1200°C, 1300°C, 1400°C and 1500°C) for Group 1 and Group 2. These were energy efficient results because the fixed 3 hours ball milling process was effective in eliminating secondary phase instead of long duration ball milling up to 60 hours were applied in previous studies.

2. Secondary phases were found present in sintering temperature 1200°C (MgO and Talc), 1300°C (Enstatite and talc) and 1400°C (Enstatite) for Group 3. However the secondary phases were gradually eliminated as diffusion process took place following increasing sintering temperature. MgO, talc and enstatite were eliminated at 1300°C, 1400°C and 1500°C respectively. This result was confirmed it is possible

to produce forsterite with no secondary phase at 1500°C with short holding time of 1 minute.

3. Forsterite with crystallite size below 80 nm was achieved in this study. This result is more feasible than previous result which applied 60 hours ball milling to obtain crystallite size of 33 nm. Holding time also has an effect on the crystallite size. Group 1, 2 and 3 produced the highest crystallite size of 75.6 nm at 1400°C, 73.3 nm at 1300°C and 74.8 nm at 1400°C respectively. Increasing sintering temperature and holding time increased the crystallite size due to grain growth.

4. Volume shrinkage for Group 1 and 2 were linearly proportionate with increasing sintering temperature. The short holding time of 1 minute in Group 3 has caused grain growth due to incomplete thermal reaction to form forsterite resulting in low volume shrinkage of 3.2% at 1300°C resulting in inconsistent trend. Group 1 recorded the highest volume shrinkage followed by Group 2 and Group 3 at 1500°C with 14.85%, 13.15% and 3.35% respectively. Group 2 at 1400°C was the optimal sintering temperature and holding time because it was more efficient to produce similar volume shrinkage as Group 1 which required longer holding time.

5. At 1500°C, Group 1 recorded the highest relative bulk density of 84.5% followed by Group 3 at 78.8% and lastly Group 2 at 78.2%. Group 3 recorded lower value at 1200°C and 1300°C due to the formation of secondary phase. The longer holding time in Group 1 has improved densification and shrinkage. Overall, relative bulk density increased together with sintering temperature resulting in reduced porosity.

6. Group 2 recorded the highest Vickers hardness of 5.01 GPa at 1500°C followed by Group 3 and Group 1 with 4.79 GPa and 4.4 GPa respectively at 1500°C. These results were agreeable with the theory suggesting Vickers hardness is inversely proportional to the square of average diagonal length. However longer holding time in Group 1 may have resulted in over firing which reduced the toughness property. Hardness is also governed by the relative density as shown in the result for Group 1 at 1300°C which recorded the biggest jump in Vickers hardness from 0.55 GPa to 3.73 GPa due to increasing relative density from 59.3% to 71.9%. Group 2 has the optimal holding time to produce highest Vickers hardness even though the highest relative density of 85.2% was recorded in Group 1 at 1400°C.

7. The highest fracture toughness was recorded at 1500°C with 3.75 $MPam^{1/2}$ for Group 2 followed by 3.63 $MPam^{1/2}$ and 3.4 $MPam^{1/2}$ for Group 3 and Group 1 respectively. The highest fracture toughness, 3.75 $MPam^{1/2}$ was recorded at relative density of 78.2%. The lowest fracture toughness of 3.4 $MPam^{1/2}$ was recorded at highest relative density of 84.5%. This result concluded that holding time was an important factor other than the relative density to improve hardness and fracture toughness properties.

8. The Young's modulus was proportional with increasing sintering temperature for Group 1 and Group 2. Group 1 and Group 2 recorded highest stiffness of 73.84 GPa and 90.46 GPa at 1300°and 1400°C respectively. The decreasing trend after maximum stiffness was due to the formation of large grain size which resulted in deterioration of Young's modulus. The presence of secondary phase (MgO and talc) for Group 3 resulted in low Young's modulus values of 37 GPa and 30.2 GPa at 1200°C and 1300°C respectively. However, the highest Young's modulus value for

Group 3 was recorded at 1400°C with 83.4 GPa as secondary phase was eliminated. Group 2 was the optimal holding time to produce highest Young's modulus at 1400°C.

9. Characteristics of the starting powders, MgO and talc were observed from the FESEM micrographs of forsterite powder due to the abstinence of heat treatment process. Group 1 showed highest porosity reduction rate at 1500°C because of the longer holding time. Partial densification was proven occurred for Group 3 with the presence of flaky like MgO trace in the FESEM micrographs. This result was agreeable with the XRD result which detected the presence of secondary phases (MgO, talc and enstatite). However the elimination of the secondary phases at 1500°C showed improvement in Vickers hardness and fracture toughness values which were 8.1% and 6.3% better than Group 1 results.

10. Group 2 has lower bulk density than Group 1 by 14.2% and 8.1% at 1400°C and 1500° respectively which were reflected in the FESEM micrographs indicating Group 1 with less porosity than Group 2. However, the Vickers hardness and fracture toughness for Group 2 were higher than Group 1 by 4.9% and 4.7% at 1400°C and 12.2% and 9.3% at 1500°C respectively. These results concluded Group 2 was the optimal holding time that resulted in highest Vickers hardness and fracture toughness properties. Over firing temperature due to longer holding time in Group 1 was the probable cause that resulted in lower toughness properties.

11. No unusual microstructure was observed on the cracked surface of forsterite compacts which were quenched in ice cold water. The flaky-like structure on the cracked surface of forsterite compact quenched at 1000°C indicate 1200°C was the

optimum temperature for densification to take place. Maximum densification and minimum porosity was observed on the cracked surface quenched at 1500°C. The grain boundary diffusivity was enhanced at 1500°C, hence promoting particles consolidation that formed rounded pore with rounded grain boundary. However there was no sign of liquid phase sintering.

12. Group 2 recorded the largest grain size at 1500°C followed by Group 1 and Group 3 with 4.21 µm, 3.05 µm and 3.01 µm respectively. The highest relative density recorded at 85.2% for Group 1 did not guarantee the largest grain size with 2.98 µm. However the highest grain size 4.21 µm was recorded together with highest Vickers hardness and fracture toughness of 5.01 GPa and 3.75 $MPam^{1/2}$ respectively at 1500°C. The presence of secondary phases talc and enstatite for Group 3 at 1300°C limit the Vickers hadness and fracture toughness enhancement but improved as the increasing sintering temperature to 1500°C eliminated the secondary phases. Grain size was observed decreasing at 1300°C for Group 1 only, even though the Vickers hardness and fracture toughness were increasing.

13. Grain size results for Group 1 were agreeable with Hall-Petch relationship indicating hardness and fracture toughness are inversely proportional to grain size. However results in Group 2 and Group 3 are agreeable with most results from previous studies indicating the inverse of Hall-Petch relationship which means hardness and fracture toughness increase with increasing grain size.

14. It can be concluded that 1 hour holding time with sintering temperature 1400°C and 1500°C is the ideal sintering profile to produce forsterite with improved mechanical properties. Other contributing factors include the optimized forsterite synthesis steps

which include the fixed 3 hours ball milling and abstinence of heat treatment which are more energy efficient than previous methods used.

15. The highest Vickers hardness and fracture toughness recorded for the forsterite compacts in this study were of 5.01 GPa and 3.75 $MPam^{1/2}$ respectively which were higher than the cortical bone lower limits of 0.2 GPa and 2.0 $MPam^{1/2}$. These results proved that forsterite has the potential to be used in biomedical application.

16. The optimized synthesis method via solid state route to produce single phase forsterite ceramic powder from this study will be beneficial for further research work related to forsterite ceramic. The optimized forsterite powder processing parameters include, fixed 3 hours ball milling, ultrasonic bath for 22 minutes and abstinence of heat treatment on the forsterite powder before final phase sintering via conventional pressureless sintering.

5.2 Suggestions for Future Work

The following are some suggestions which can be used to extend the scope of study on forsterite ceramic:

1. In the current research, 1 hour holding time recorded the highest Vickers hardness and fracture toughness values followed by 1 minute holding time and the lowest by the 2 hour holding time. The shorter holding time proved to be more energy efficient. Therefore microwave sintering method is suggested which could be energy efficient.

2. Current ramp rate of 10°C/min was used in all Group 1, 2 and 3 with Group 2 excelling in Vickers hardness and fracture toughness properties. It is suggested to add in 2 more ramp rates, 5°C/min and 15°C/min respectively using microwave sintering method with similar holding time of 2 hours, 1 hour and 1 minute to observe the effect on the mechanical properties of forsterite.

3. The good results of Vickers hardness and fracture toughness for forsterite over cortical bone lower limit indicate good prospect for biomedical application. It is suggested to use the forsterite produced using current optimized method and the forsterite produced using the suggested microwave sintering method subjected to in vivo and in vitro studies.

4. It is also recommended to use other sintering method such as hot isostatic pressing and spark plasma sintering to evaluate the densification behavior of forsterite ceramic.