Sintering Behavior and Machinability Properties of Sepiolite Based Glass-Ceramics

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Abstract

Glass-ceramics were produced by sintering method from sepiolite containing of SiO2 and other glass making oxides. The sintering behavior and machinability of glass-ceramic composition were investigated. Some additives were added to natural raw materials for increment machinability and sintering properties. All starting materials were mixed by ball milling for 2 h using alumina media. The mixed and milled powders were sieved to grain sizes smaller than 75 µm and pressed at 100 MPa. The pressed samples were sintered at 900 -1200 ºC for 1 h in an electric furnace using a heating rate of 5 ºC/min. Some characterization tests such as X-ray diffraction (XRD), scanning electron microscopy (SEM) and machinability tests were performed on sintered samples. The results indicated that all samples exhibits good sintering and machinability properties.

Key words: Sepiolite, glass-ceramic, sintering, machinability

1. Introduction

Ceramics are suitable for their some properties such as high hardness, heat resistance, corrosion resistivity and insulation in terms of heat and electricity. Thanks to these, they can be employed for many applications, for instance; coatings having high wear resistance, refractory industry, and electricity industries as electro-ceramics. But, these areas are restricted by some issues about ceramics. Ceramics are hard and durable where as they are prone against sudden impact due to lower toughness and impossibility of plastic deformation of ceramics compared with metals [1]. Some mechanisms have been developed to increase their toughness, for example; composite formation, crack propagation and branching. These application provide higher strength for ceramics, however, their plastic deformation possibilities do not improve, sufficiently. On the other hand, some phase formation in ceramic matrix exhibit positive results against to mechanical effects as cutting and drilling. The phases called micas provide plastic deformation and machinability for ceramics. Mica phases have been obtained from glass-ceramic systems and their controlled crystallizations. Glass-ceramics are solid materials with one or more crystal line phases embedded in amorphous matrix[2].Some modifications in chemical composition and production methods can provide better mechanical behavior as for these effects [3]. Mica glass-ceramics with their unique machinable property have become an interesting subject for many researchers. The machinability of these glass-ceramics is related to an interlocking microstructure and the cleavage of plate-like mica makes them suitable for machining with high precision by ordinary tools [4]. Mica based glass-ceramics are transformed to commercial products as MACOR and DICOR brands. Excepted for prepared commercial glass-ceramic systems, some raw materials and wastes have been used for scientific studies about this.

In the current study, Sepiolite as raw materials was used for machinable glass-ceramic production. Sepiolite is a type of clay mineral including magnesium silicate, and its typical formula is$Mg_4Si_6O_{15}(OH)_2{\cdot}6H_2O$. Owing to Mg and Si content of mica glass-ceramic phases, it thought that Sepiolite can be used for machinable glass-ceramic production.The possibility of machinable glass ceramics production without using pure ceramic oxides was investigated in the current study. We aimed that the possibility of using sepiolite for machinable glass-ceramic production was investigated.

2. Materials and Method

The chemical composition of Sepiolite obtained from Beylikova-Eskişehir/TURKEY was given in Table 1. A homogeneous batch mixture including Sepiolite 50%, Al_2O_3 12%,B_2O_3 5%, K_2O 10%, SiO_2 15% and MgF_2 8% was prepared by weight. These ratios were determined to form mica phases.The powders were ground and mixed thoroughly by using ball mill with alumina ball for 2 h. The mixture was sieved to 75 µm and then uniaxial pressing was employed to shape the samples. Cylindrical samples (∅ 20 mm) were shaped under the 100 MPaload and then sintered at 900-1200°C the heating rate of 5°/min for 1 h. For the crystalline phase's observation, X-ray diffraction (XRD, Rigaku D/MAX, Cu-Kα radiation) was used. The microstructural examinations by using scanning electron microscopy (SEM, Joel 6060) were performed. Furthermore, machinability tests were applied to the disc shaped specimens using 5 mm diamond drills with 200 rpm drilling rate under uncontrolled load.

Table 1. Chemical composition of Sepiolite

Oxide	SiO_2	Al_2O_3	TiO_2	Fe_2O_3	CaO	MgO	K_2O	L.O.I.*
%	54.2	4.43	0.2	1.7	8.15	28.52	0.5	2.3

*lost on ignition

3. Results and Discussion

Phlogopite, Leucite and Forsterite phases crystallize depending on temperature were determined by XRD results given in Figure 1. Apart from these, Diopside peaks were determined for the samples heated at 900°C and 1000°C. The structure was dominated by Phlogopite and Leucite phases atat 900°C and 1000°C. Phlogopite[$KMg_3AlSi_3O_{10}(F,OH)_2$]is major phase for machinable glass-ceramic systems [5]. Leucite is potassium and aluminiumtectosilicate$K[AlSi_2O_6]$ form and it occurs above 650°C for suitable compositions, generally. Leucite based glass-ceramics have been used for dental applications, widely [6]. Leucite phase provides higher flexural strength and toughness for glass-ceramics. Cesar et. al. presented that the higher the amount of leucite means the higher resistance to crack propagation [7].Leucite together with Phlogopite provides better characterization for machinability. When the temperature raised 1100°C, Forsterite crystallization became effective. Forsterite, which is magnesium silicate form of ceramic phase (Mg_2SiO_4), has usually utilized in applicationswith high coefficient of thermal expansion. Typicalapplications are making substrates for high frequencyelectronics, ceramic–metal seals and high temperaturebonding agent [8].Forsterite phase crystallizes above 1000°Cin the literature, effectively [9]. In the current study, Forsterite forms upper temperatures such as 1100°C and 1200°C.

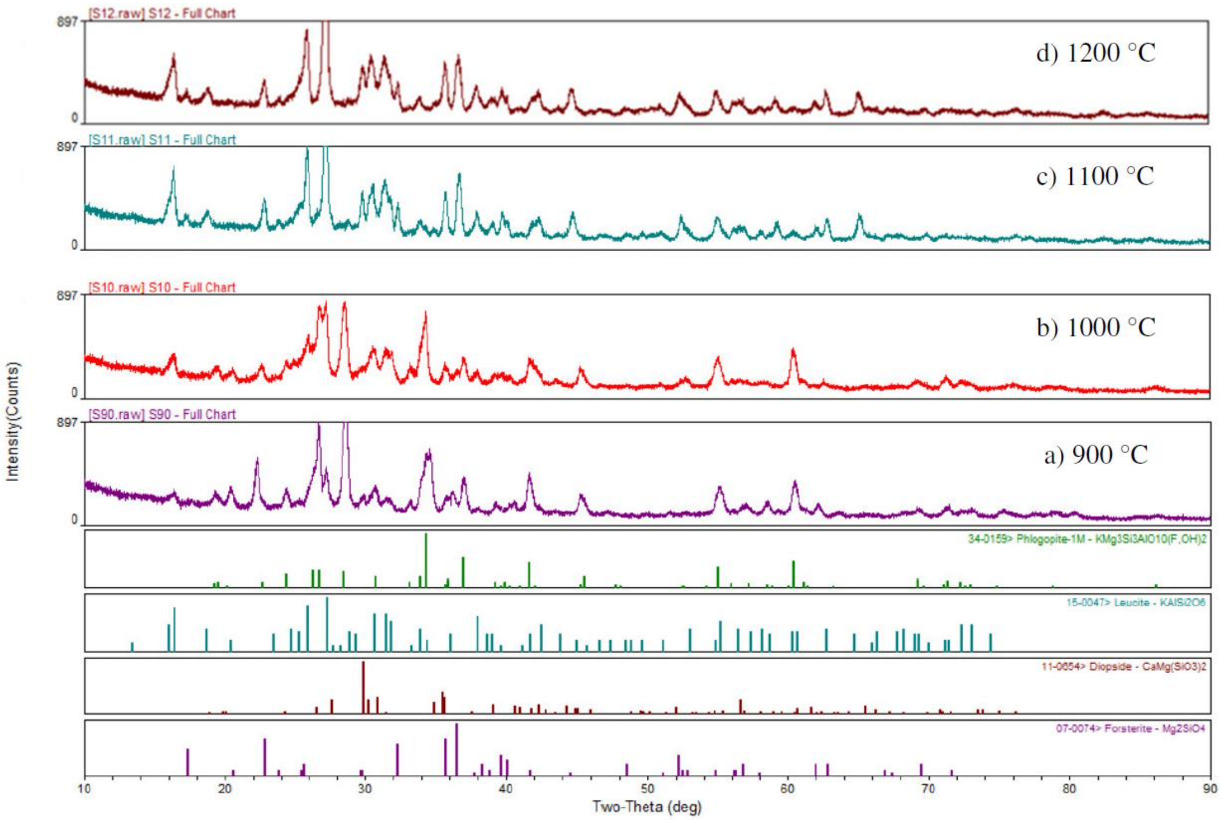
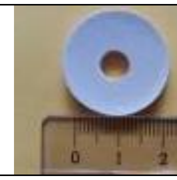
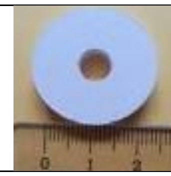
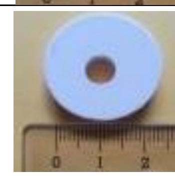


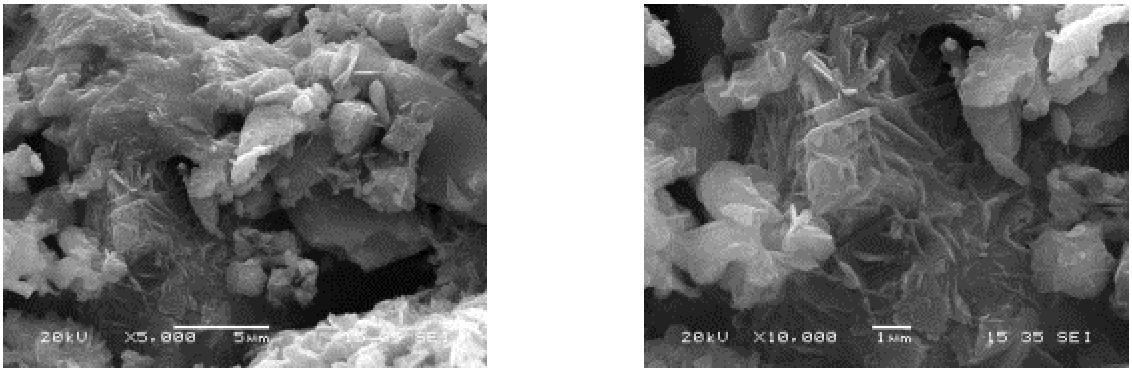
Figure 1.XRD patterns of the samples depending on sintering temperature.
a) 900 °C, b) 1000 °C, c) 1100 °C, d) 1200 °C

The machining test results have good agreement with XRD results. Table 2 exhibits detected crystalline phases and the macro images as to machinability test results. All samples were drilled, successfully. Any mechanical damage such as fracture or crack was not observed during to the machinability tests.

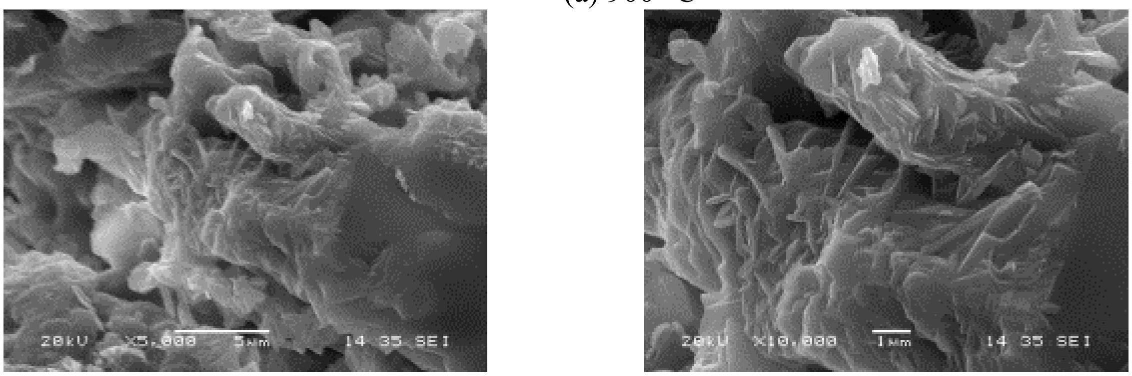
SEM images of the samples can be seen in Figure 2. The first point of these is that the fibrous and rod like structures observe in the sample heated at 900°C and 1000°C, clearly. These temperatures are effective conditions for Phlogopite crystallization. These structures indicate Phlogoite crystals, probably. It is possible that spherical structures surrounding fibrous and rod like structures is Leucite crystals. When the temperatures raise 1100°C and 1200°C, the structures change. More smooth, sharp and bigger grains occur depending on increasing in temperature. It is possible that increasing in temperature provides better crystallization conditions, grain coarsening and lesser residual glass phase in these temperatures compared with the samples heated lower temperatures.

Table 2. The macro images and crystalline phases versus sintering temperature.

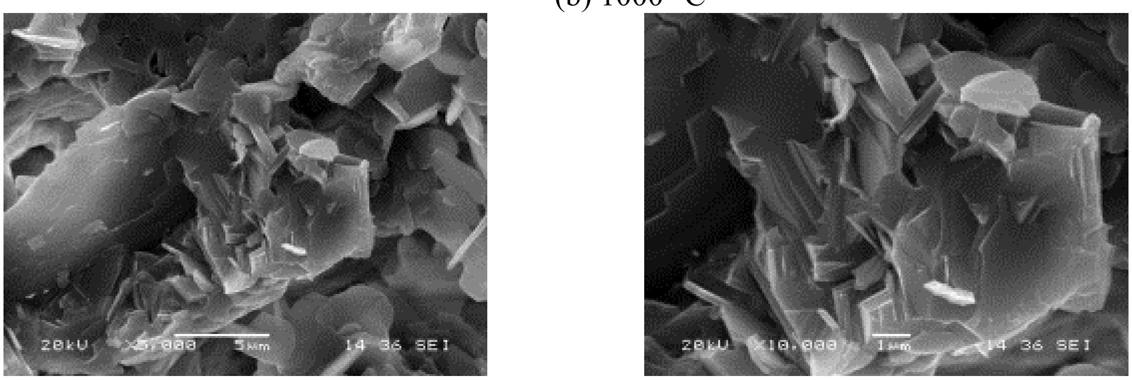
Sintering Temp. (°C)	Detected phases	Before machinability test	After machinability test
900	Phlogopite (Major) Leucite (Major) Diopside		
1000	Phlogopite (Major) Leucite (Major) Diopside		
1100	Phlogopite Leucite Forsterite (Major)		
1200	Phlogopite Leucite Forsterite (Major)		



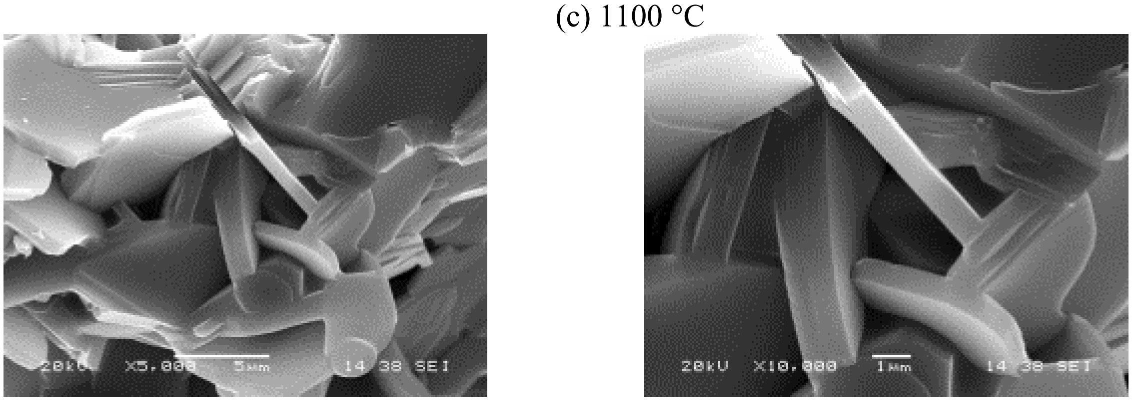
(a) 900 °C



(b) 1000 °C



(c) 1100 °C



(d) 1200 °C

Figure 2.SEM microstructure of themachinable glass-ceramics versus sintering temperature

Conclusions

The main conclusions of the study were summarized below;

1. In this study, possibility of Sepiolite utilization for machinable glass-ceramic production was investigated. According to commercial mica glass-ceramic compositions, the mixture was prepared by using Sepiolite. The theory about preference of Sepiolite is high Si and Mg content of this and these elements are common components for mica phases.

2. Phlogopite, Leucite and Forsterite phases crystallize depending on temperatures were determined by XRD. Especially, Phlogopite and Leucite phases provide extra properties on glass-ceramic body in terms of mechanical behavior. Forsterite is prominent thanks to thermal properties such as high thermal expansion coefficient. When Phlogopite and Leucite phases are dominant at 900°C and 1000°C, Forsterite crystallizes at 1100°C and 1200°C.

3. The SEM images exhibited good agreement with XRD results. Fibrous and rod like structures as Phlogopite observed in the samples heated at 900°C and 1000°C. The structure transformed to view including more sharp, coarse and smooth grains at 1100°C and 1200°C.

4. The drilling test results showed that all specimens were drilled, successfully. There is no significant difference among machining performances.

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