The Effect of Sintering Temperature on Machinability Properties of Vermiculite Based Glass-Ceramics

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ABSTRACT

Glass-ceramic was produced by sintering method from vermiculite, K₂CO₃ and MgF₂. The sintering behavior and machinability of glass-ceramic composition was investigated. The starting materials were mixed together and milled in an alumina mill for 2 h. Then the powders were sieved to grain sizes smaller than 75 μm. For the sintering route, disc samples were prepared by pressing at 100 MPa. The pressed discs were sintered at 1100 - 1300 °C for 2 h in an electric furnace using a heating rate of 5 °C/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.

1. INTRODUCTION

Glass–ceramics (GC) are attractive materials for engineering purpose including electronic, semiconductor, laser, high vacuum, aerospace and space industry and also bio-medical purpose including bone, dental, and tissue engineering applications. They are normally obtained by a controlled crystallization process of suitable glasses. It can be produced from different waste or natural materials such as fly ash or natural volcanic rocks. Initially, glass base structures are transformed to glass-ceramics via controlled crystallization [1-4]. Machinable glass-ceramics rely on mica crystals in their microstructure. They can be machined to complicated shapes and precision parts with ordinary metalworking tools, quickly and inexpensively. Machinable glass-ceramics require no post firing after machining. This means that specifications can be met without having to resort to costly machining with diamond or other cutting tools [4,5]. Most of the commercial machinable glass–ceramics are based on potassium fluorophlogopite and other types are based mica group. The mica containing glass–ceramics received wide application due to their high machinability, excellent esthetics, low thermal conductivity, high strength, durability, biocompatibility, ease of manufacture and high wear resistance [1].

Vermiculite is a member of clay minerals, produced by the decompositions of micas and occurs as large crystals of mica-like habits [6]. Vermiculite mineral has a mica- like lamellar structure that quickly expands on heating to produce a lightweight material. Vermiculite is formed by weathering or hydrothermal alteration of hydrobiotite or phlogopite (KMg₃Al₃Si₃O₁₀(F,OH)₂) mineral phases [7,8]. In Turkey, totally 5.2 million tons reserve of vermiculite is reported by Mineral Research&Exploration General Directorate. The most important deposits of vermiculite in Turkey are Karakoç mines (in Yildizeli, Turkey) because of its expansion ratios [9].

The aim of our investigation is to synthesize machinable glass-ceramics by using the expanded vermiculite minerals. When it comes to machinable ceramics, glass and glass-ceramic materials produced from vermiculite by sintering method have not been evaluated in the literature.

2. EXPERIMENTAL

In Table 1 the chemical compositions of the vermiculite used in this study were given. The vermiculites were provided from Organik Madencilik company in the Yıldızeli region of SIVAS, TÜRKİYE. K₂O % 5 and MgF₂ 15 % (wt. %) was added to vermiculite for obtaining machinability property. The raw materials and other additives were mixed by using an alumina ball mill at 250 rpm for 60 minutes in an alumina media. The mixture was sieved to a particle size fraction of -75 μm and then uniaxial pressing was employed to shape the samples. Cylindrical samples (Ø 25 mm) were shaped under the 100 MPa load and then sintered at several temperatures as 1100, 1200 and 1300 °C the heating rate of 5 °C/min for 2 h. XRD analysis (Rigaku D/MAX, CuKα radiation) was used for the crystalline phases determining.

The micro-structural examinations were realized by using SEM (JEOL 6060) to polished and etched surfaces of samples in the solution of 5 vol. % HF for 1-2 min was used for etching process. Furthermore, machinability tests were applied to the disc shaped
specimens using 5 mm diamond drills with 440 rpm drilling rate under uncontrolled load.

**TABLE 1.** Chemical composition of the vermiculite

<table>
<thead>
<tr>
<th>% wt.</th>
<th>Vermiculite</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>36-40</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>12-17</td>
</tr>
<tr>
<td>TiO₂</td>
<td>1-3</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>6-12</td>
</tr>
<tr>
<td>CaO</td>
<td>2-13</td>
</tr>
<tr>
<td>MgO</td>
<td>8-18</td>
</tr>
<tr>
<td>K₂O</td>
<td>1-6</td>
</tr>
<tr>
<td>L.O.I.</td>
<td>0-10</td>
</tr>
</tbody>
</table>

3. RESULT AND DISCUSSION

Table 2 exhibits detected crystalline phases and the macro images as to machinability test results. As seen from Table, machinability test was performed for all samples, successfully. As can be seen in the macro images, firing shrinkage and also darkening of color was observed clearly depending on increase in temperature.

**TABLE 2.** The macro images and detected crystalline phases of samples according to sintering temperature

<table>
<thead>
<tr>
<th>Sintering Temperature</th>
<th>Detected Phases After</th>
<th>Machining Test</th>
<th>Before</th>
<th>After</th>
</tr>
</thead>
<tbody>
<tr>
<td>1100 °C</td>
<td>Forsterite, Phlogopite, Jadeite-diopside, Augite</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1200 °C</td>
<td>Forsterite, Phlogopite, Jadeite-diopside, Spinel</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1300 °C</td>
<td>Forsterite, Phlogopite, Spinel</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The XRD patterns of the samples sintered at temperatures in the range from 1100 to 1300 °C are seen in Fig. 1. (Mg,Fe,Ti,Al)(Ca,Na,Fe,Mg)(Si,Al)₂O₆ [Augite (ASTM card no:01-088-0859)], Mg₂SiO₄ [Forsterite (ASTM card no:01-071-1080)], (Ca₀.₄₂Na₀.₅₈)(Mg₀.₃₇Fe₀.₀₃Al₀.₆₀)(Si₁.₉₂Al₀.₀₈O₆) [Jadeite-diopside (ASTM card no: 01-080-1869)], MgAl₂O₄ [Spinel (ASTM card no: 98-000-0098)] and Al₁.₇₁F₀.₀₆S₀.₁₂H₁.₉₃K₀.₈₂Mg₂.₂₅Na₀.₁₁₃O₁.₁₁₂Si₂.₇₈₅ [Phlogopite (ASTM card no: 01-089-6513)] phases were determined by XRD in the samples.

As seen in the XRD results, Forsterite, and Phlogopite phases were detected for all samples. When jadeite diopside and augite phases were determined for the sample sintered at 1100 °C 1200 °C, spinel phase was detected at 1200 °C and 1300 °C. Similarly to fluor mica phases, Diopside is common phase for machinable glass-ceramics [10]. Alizadeh et al. have reported Forsterite, Phlogopite, Jadeite-diopside phases at 1140 °C for similar glass-ceramic system [11]. Furthermore, the peak intensities of forsterite phase detected at 1100 °C decreases with increasing in temperature, it is possible that the phase transform to spinel. 1000 °C is considered as the onset point for spinelisation. MgAl₂O₄ spinel has crystallized above
1000 °C, generally [12]. Zheng et al. have presented that Diopside is major phase from 950°C to 1110 °C, Spinel is minor in related temperatures [13]. In the current study, Augite and Diopside phases possibly started decomposition after 1200°C, and then Spinel phase had dominant role at 1300 °C. Strong spinel phase signals in the XRD pattern for 1300 °C have good agreement to this.

FIGURE 2. SEM images of the sintered samples at a-) 1100 °C, b-) 1200 °C and c-) 1300 °C

Fig. 2 shows SEM images of the samples, rod-like and crystals are observed in the samples sintered at 1100 °C and 1200 °C. Diopside crystals are presented as this kind of rod like structures in the literature [14]. These structures are Diopside phases, possibly. When the temperatures reached at 1300 °C, these crystals have disappeared. This result supported to the XRD results, the grains and crystals are seem to be more oval compared to the samples sintered at 1100 °C and 1200 °C.

4. CONCLUSION

The main object of this study was to produce machinable glass-ceramic materials from vermiculite by sintering method. Phlogopite, Augite, Forsterite, Jadeite-diopside, and MgAl₂O₄ spinel phases were detected by XRD. Diopside and Phlogopite phases are common phases for machinable glass-ceramic systems. Phlogopite, Augite and Diopside phase intensities decreased with increasing temperature. When temperature reached to 1300 °C, Spinel phase is major phase. This phase transformation was approved by SEM images. Sharp igneous Diopside crystals disappeared for SEM image of the sample sintered at 1300 °C. It has not negative effect on machinability. All samples were machined, successfully. When macro images of samples were scrutinized, firing shrinkage and also darkening of color was observed clearly depending on increase in sintering temperature.

5. REFERENCES


