ARTICLE

Microstructural analysis of clay ceramics by SEM

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Abstract

Clay is a raw material interesting to industry because it exhibits properties like plasticity, easy processing and mechanical resistance after sintering, besides of being abundant and having low cost. In this paper, clay was additived with feldspar or glass fines, waste of glass industry, to compare the physical and mechanical properties of sintered body and verify the behavior of these additives as a function of the sintering temperature. Compacts were sintered at temperatures of 900, 1000, 1100 and 1200°C, and fracture surface was examined through microstructural characterization in a Leo 1450 VP Scanning Electron Microscope in secondary electrons mode. Relative density, linear contraction, water absorption and flexure rupture modulus were determinated, and the obtained materials can be used as ceramic cove tile. Microstructural analysis shows that the glass is a better flux than the feldspar to the same sintering conditions and that the physical and mechanical properties of materials depend mainly on the porosity present in its structure.

Keywords: Clay, glass, feldspar, microstructure and properties.

Introduction

Clay is a very old raw material, used by primitive people to make figures and manufactured articles (2). It is used in different industrial chemical processes and in the fabrication of ceramic artefacts like sanitary wares, porcelain, roof-tiles, wall-tiles and floor-tiles, among others (2,8,10).

Clays are fine-grained inorganic material (grain size smaller than 2 μm) and consist on aluminum and iron hydrated silicates with alkaline earths and alkaline elements (2,8).

Clays exhibit interesting properties like plasticity when wet, easy processing and mechanical resistance after sintering. Besides, they are abundant and have low cost, leading to scientific and industrial interest (8,10).

However to get ceramic articles with characteristics to carry out a function it is necessary to use an additive (flux), responsible to the formation of the liquid phase during sintering. It promotes a best rearrangement of the particles and fills the present porous in the material, making it dense (4).

The feldspar is the most used flux in ceramic industry. However its world consumption is increasing and at middle term will be difficult to buy it with accessible cost and quality (1).

Nowadays to incorporate industrial wastes in ceramic masses is a good alternative to the destination of these rejects, since they contribute to the final properties of the ceramics (5,6,9).

The glass fines, which are a waste of glass industry, is a new alternative to replace the feldspar in the production of ceramic cove tile. To produce these materials it is necessary to get physical and mechanical properties that attend to the standard specifications (10). So the microstructural characterization is an important tool because these properties depend on factors like porosity, defects, grain size and shape. (3,5,6,9,11).

In this paper it was examined the microstructure of clay ceramics additived with feldspar or glass fines as a function of the sintering temperature and correlating the microstructure with physical and mechanical properties of clay ceramics. Also the formation and diffusion of the liquid phase were studied.

Materials and Methods

Materials

The materials used were: clay from São Simão Company, sodic feldspar from Prominex Mineração and glass fines wastes from Pilkington Ltda. The chemical composition of clay, feldspar and glass fines is reported in Table 1.

Table 1. Chemical composition of raw materials (%).

composition	clay	fesdspar	Glass fines
SiO_2	47,38	69,0-72,0	72,4
Al_2O_3	30,83	16,5-19,5	0,7
Fe_2O_3	5,59	0,05-0,25	0,11
CaO	0,15	<0,42	8,6
MgO	0,28	<0,01	4,0
Na_2O	0,083	7,6-8,5	13,6
K_2O	0,54	1,0-2,0	0,3
TiO_2	1,21	-	0.02
LF	14,0	0,40-0,55	-
SO_2	-	-	0,2

Methods

Two mixtures were prepared: clay plus 10 wt% feldspar and clay plus 6.9 wt% glass. The raw materials were mixed in a steel attritor mill clothed of aluminum oxide in watery environment during 60 minutes. The powder was dried and compacted in uniaxial pressing at 14 MPa in a dye measuring 115x25x14 mm^3. The compacts were measured with caliper square, weighed and sintered in air, at a heating rate of 5°C/minute, up to 900, 1000, 1100 and 1200°C, staying 1 hour at these temperatures. It was used an isothermal at 600°C for 0.5 hour. Relative density, linear contraction, water absorption and flexure rupture modulus in three points of the sintered body were calculated and the fractured surface of the samples was observed using a Leo 1450 VP Scanning Electron Microscope in secondary electrons mode.

Results and Discussion

Figure 1 shows the relative density (RD) as a function of the sintering temperature. The sintered body with glass has greater densification when compared to those with feldspar, at the same temperature.

Figure 2 shows the sandstoning diagram of sintered body, that is the linear contraction (LC) and water absorption (WA) as a function of the sintering temperature. It is observed that up to 1100°C the sintered bodies exhibit accepted LC values (ca. up to 9%), but at 1200°C the sintered bodies with glass exhibits a higher value (ca. 15%). The values of WA to porous ceramic cove tile (10%<WA<20%) (10) are obtained at 1100°C for sintered body with glass and at 1200°C with feldspar. For semi- porous ceramic cove tile (6%< WA<10%) (10) values of 4% WA are obtained only for sintered body with glass at 1200°C.

Figures 1 and 2 show the expected dependence among RD, LC and WA. When the RD increases, the LC increases to and the WA decreases.

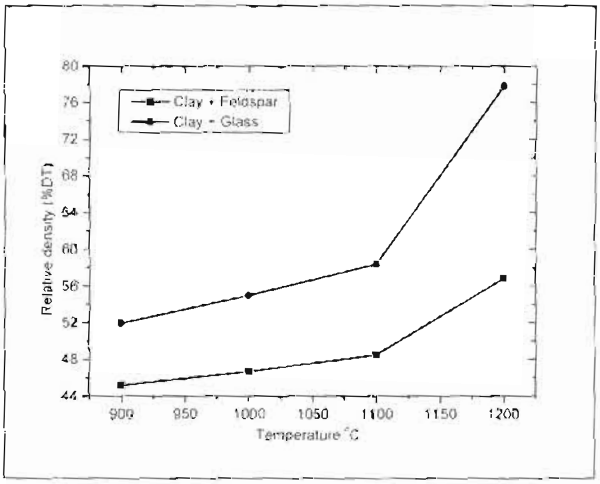

Fig 1. Sintered relative density as a function of sintering temperature.

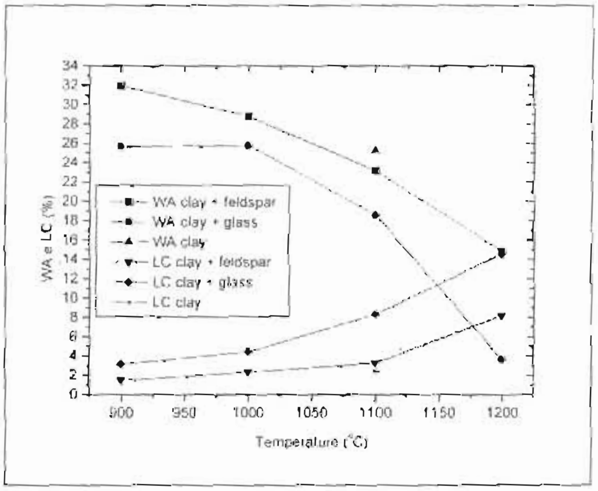

Fig. 2. Sandstoning diagram of ceramic masses.

Figure 3 shows the flexure rupture modulus (FRM) as a function of the sintering temperature. The values of FRM for sintered body with glass are higher than the values with feldspar for all sintering temperatures studied. This can be explained because the RD (Figure 1) is larger and the porosity is smaller for sintered body with glass, and as the porosity reduces the FRM, the larger as RD, the larger will be the FRM. It was observed that the values to produce wall ceramic cove tile (> 12 MPa) (10) are obtained at 1100°C to sintered body with glass and only at 1200°C with feldspar.

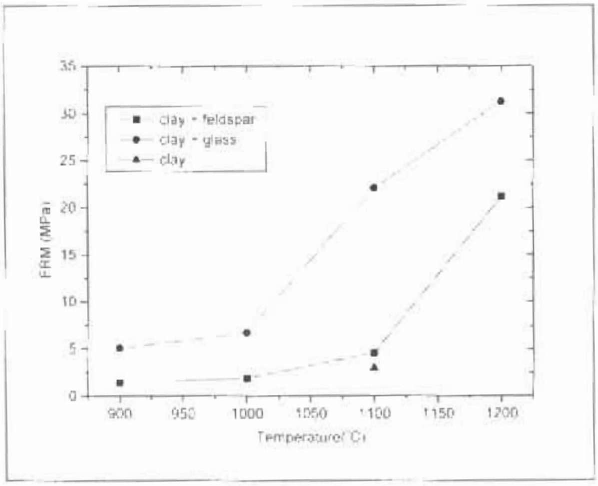

Fig. 3. Flexure Rupture Modulus (FRM) as a function of sintering temperature.

Analyzing SEM micrographs (Figures 4-6), the clay non- additived (Figure 4) is composed by lamellas with plates of different sizes, similar to the red smectite clay (7).

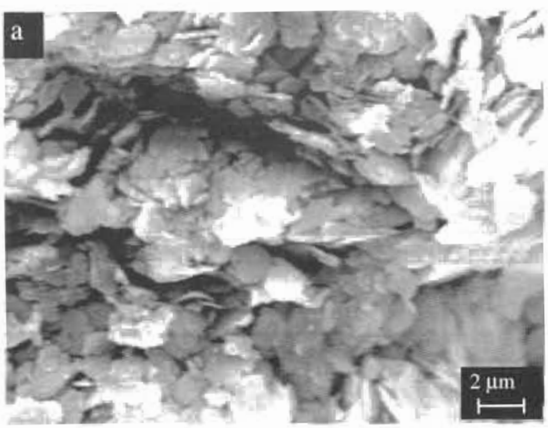

Fig. 4. SEM by SE of clay sintered at 1100°C.

Figure 5a shows that the glass forms veins of liquid phase at 900°C which increase in volume with the time and temperature (Figure 5b), and diffuses among the particles. This phenomenon is called "wetting", better observed in Figure 5c, and occurs when the contact angle between liquid and solid is low. The capillary pressure generates enables the rearrangement of the particles reducing the porosity of the material (Figure 5d), making it denser, as shown in Figure 1. Figures 6a and 6b show that the feldspar does not melt up to 1000°C, forming veins of liquid phase only at 1100°C (Figure 6c) and partially diffuses on the particles at 1200°C (Figure 6d).

Relating these microstructures with the properties of sintered bodies it is shows that when the additive (flux) forms a liquid phase the opening porosity is reduced and occurs a higher densification of material after its cooling.

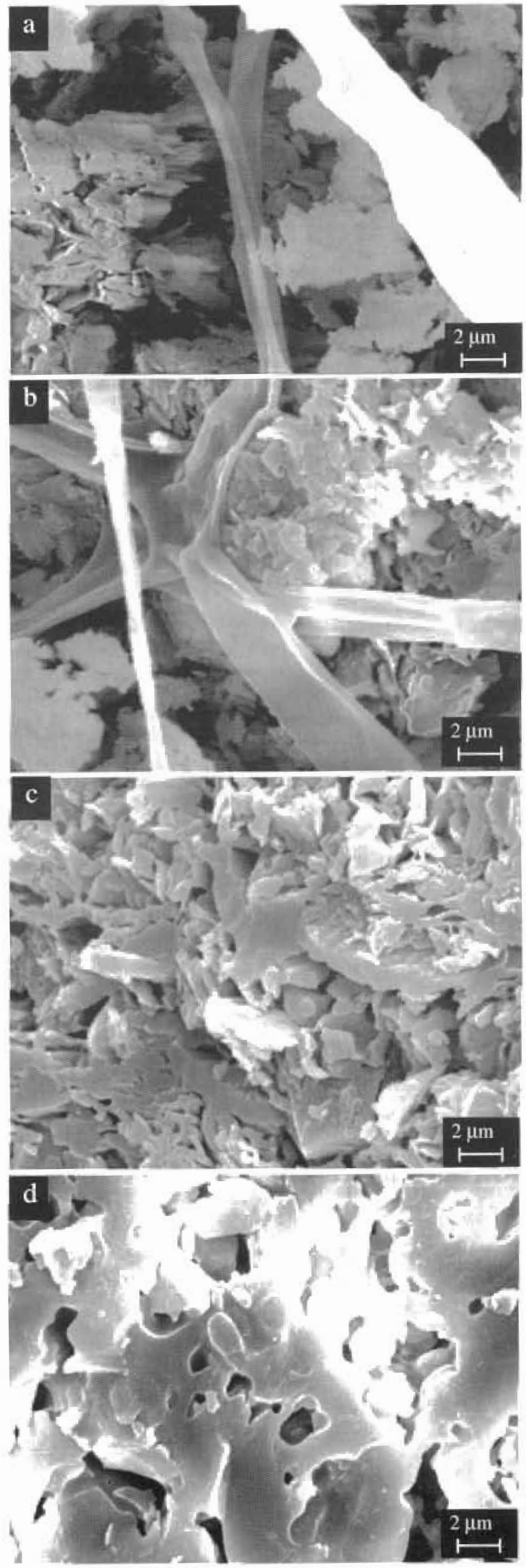

Fig. 5. SEM by SE of clay + glass sintered at 900, 100, 1100 and 1200°C, respectively.

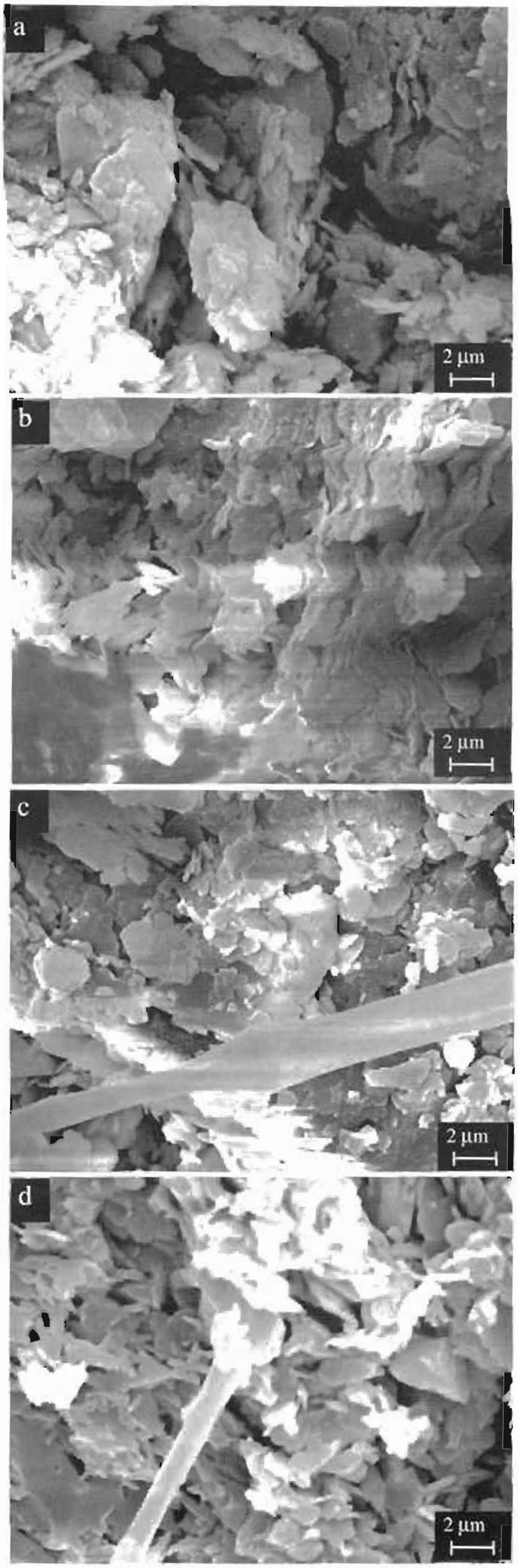

Fig. 6. SEM by SE of clay + feldspar sintered at 900, 100, 1100 and 1200°C, respectively

The final porosity damages mainly the water absorption and flexure resistance these ceramic materials.

Conclusion

Microstructural analysis of materials sintered was fundamental to understand and interpret the behavior of the feldspar and glass fines fluxes in the microstructure.

Acknowledgments

We wish to acknowledge the financial support from FAPESP (Process n° 00/11204-6 and n° 97/11020-8). We thank Pilkington for the donation of the glass powder.

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