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Sintered glass-ceramics from mixtures of wastes

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Abstract

Panel glass from dismantled cathode ray tubes, mining residues from feldspar excavation and lime from fume abatement systems of the glass industry have been employed as raw materials for several glass compositions. The prepared glasses were ground into fine powders and subjected to sintering treatments at low temperatures (880–930 °C), with concurrent crystallization, thus obtaining sintered glass-ceramics. The mechanical properties (for example, bending strength exceeding 100 MPa) and the aesthetic appearance of the materials, together with the simplicity of the manufacturing method, are promising for applications in the building industry.

Keywords: A. Sintering; C. Mechanical properties; D. Glass ceramics; E. Structural applications

1. Introduction

Glass-ceramics, intended for building applications, constitute an undoubtedly well-developed and widespread way to absorb glasses obtained from the treatment of several wastes. In addition to the environmental advantage of immobilizing wastes into materials with a generally high chemical resistance (like glasses), a certain economic benefit may be found in entering the large market of construction materials. The first and most important example was certainly that of Russian Slagsitalls, developed as early as the 1960s, by employing several slags of ferrous and non-ferrous metallurgy, ashes and wastes from mining and chemical industries [1,2]. Lately, several combinations of wastes have been proposed [3–5], sometimes leading to materials with excellent properties (Öveçoglu [6] obtained glass-ceramics with a bending strength exceeding 300 MPa).

Conventional glass-ceramics start from a two-step thermal treatment of nucleation and crystal growth. Although undoubtedly widespread, this route presents some disadvantages: the treatment is quite difficult and expensive, and sometimes particularly slow, so that catalysts (TiO_2, Cr_2O_3, etc.) are needed [1,2]. In addition, the removal of defects (like gas bubbles) in the parent glass is essential for the achievement of strong glass-ceramics, so that long fining times are required.

A secondary glass-ceramics manufacturing route, that of sintered glass-ceramics, has been established since the 1960s. Finely powdered glass is generally pressed and sintered, the crystallisation occurring together with densification [7–9]. Since free glass surfaces are preferred sites for devitrification, crystallisation may occur without catalysts, especially for small grains [7], thus configuring a ‘‘surface mechanism’’. The sintering approach may lead to a pleasant ‘‘marble-like’’ appearance of the glass-ceramics, much superior to that of Slagsitalls. Commercial examples of this approach are the wollastonite-based ‘‘Neoparies’’, developed in Japan since 1970s [1,2]. Sintered pyroxene-based glass-ceramics from cheaper and more accessible raw materials has been developed, in Bulgaria [10] and in Italy [11–13], since the early 1990s. The parent glass being provided in powdered form, long fining times are not needed, thus drastically reducing the costs of preliminary glass-making; in addition, very limited processing times may be advantageous in avoiding the volatilisation of dangerous oxides (like those of heavy metals) which may be dissolved in glasses when obtained from wastes.

In this paper, we describe the production of sintered glass-ceramics based on glass obtained by the treatment of various wastes, consisting of panel glass from dismantled cathode ray tubes (CRTs), mining residues from feldspar excavation and lime from fume abatement systems of the glass industry. Panel

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glass from dismantled CRTs (corresponding to the front part) is actually a waste material, being employed in the manufacturing of new CRTs only in low percentages (15 wt.%) [14] due to the fact that only high-quality pristine glass, from selected raw materials and with no lead contaminations, is desirable [15,16]. It has been estimated that the amount of end-of-life CRTs in Western Europe is in the range of 300 kton/year [17,18], so that an enormous quantity of panel glass is available and new applications are expected for its reutilization. Residues from feldspar excavation correspond to fractions of feldspar minerals which cannot be employed in the ceramic industry due to their high silica content. Lime consists of calcium hydroxide employed to react with acidic fumes coming from vitrification plants: besides $Ca(OH)_2$, the composition features several pollutants, like nitrates, sulphates, fluorides etc, or traces of heavy metals so that this waste is hardly used as a source of CaO in conventional glass-making.

The achieved mechanical properties of sintered glass-ceramics (the bending strength, in some cases, approaching or exceeding 100 MPa) and the very simple and cost effective manufacturing process (owing to the particular glass compositions) are promising for applications in the building industry. The great dimensions of this field of application should be profitable for a remarkable absorption of the reported wastes.

2. Experimental

The chemical composition of the employed wastes is available in Table 1 (the composition of lime is that after calcination). The wastes were mixed in the proportion panel-lime-mining residues 28%–25%–47% by weight. The particular combination was chosen in order to approach the composition of wollastonite-based "Neoparies" [1]; the high content of alkali, with respect to ordinary wollastonite-based glass-ceramics was compensated by a remarkable amount of alumina (about 10%), in order to determine alumino-silicate secondary phases. Since several compositions of panel glass and lime were available, three different glass compositions were achieved, by vitrification of the mixtures at about 1300 °C for 1.5 h. The glasses were not extensively refined and drastically cooled by pouring them on a steel plate, thus producing large fragments. A chemical analysis was conducted on each glass by means of X-ray fluorescence analyser (Philips XRF Sequential Spectrometer PW 2400, Eindhoven, The Netherlands). The glasses were dry ball-milled and sized to grains <88 µm, for "coarser powders", and <37 µm, for "finer powders".

Table 1
Chemical and physical properties of the starting wastes and of the investigated glasses

	Panel glass	Lime	Mining residues	A glass	B glass	C glass
SiO_2	60.7–64.6	10.7–16.8	67.3	53.49	54.59	51.85
Al_2O_3	2.1–2.8	1.1–1.4	18.3	9.74	10.93	9.46
Na_2O	8.0–9.4	3.0–3.2	8.7	7.51	7.41	7.14
K_2O	7.2–7.5	0.4–2.6	1.1	2.66	2.23	3.24
CaO	0.5–1.0	59.9–65.9	1.9	17.87	17.29	16.18
MgO	0.3	0.1–2.4	1.1	1.24	1.21	0.78
BaO	9.1–12.7	0.0–0.2		3.51	2.69	3.07
SrO	1.0–8.4	0.0–0.1		0.27	0.24	1.29
Fe_2O_3	0.1	0.0–0.1	1.5	0.79	0.88	0.74
TiO_2	0.4		0.1	0.17	0.17	0.19
ZrO_2	0.1–0.9	0.0–0.6		0.03	0.05	0.24
PbO	0.1–0.3	0.0–1.1		0.03	0.04	0.37
ZnO	0.1–0.4	0.0–2.6		0.02	0.02	0.13
As_2O_3		0.1–0.9		0.03	0.03	0.23
Sb_2O_3	0.4–0.5	0.9–1.3		0.43	0.41	0.62
P_2O_5		0.2		0.05	0.06	0.05
B_2O_3		0.8–5.9		0.30	0.10	1.48
F		6.4–9.5		1.69	1.50	2.37
Cl		0.4–0.8		0.11	0.11	0.12
SO_3		0.2–1.7		0.03	0.05	0.43
Transition temperature (°C)				560	575	595
Thermal expansion coefficient (10^{-6} °C^{-1})				9.4	9.1	9.5

The glasses were subjected to dilatometric and differential thermal analysis (DSC 404, Netzsch Gerätebau GmbH, Selb, Germany, 10 °C/min heating rate). The DTA allowed to identify the glass crystallisation temperatures, as illustrated by Fig. 1. This analysis constituted an important reference point for sintering since, like in previous experiences [16,19], the processing temperature for achieving glass-ceramics was coincident with the crystallisation temperature. This choice was suggested by the expected high nucleating activity of fine-grained glass (maximum size always <88 µm), and may represent a desirable feature from an economic point of view, due to the lower processing temperature than in other experiences of simultaneous sintering and crystallisation of glass [10–13].

The powders of each composition (both coarser and finer) were gently pressed in a rectangular die, then sintered in air at the crystallisation temperature, for various times, with a heating rate of 10 °C min. In the case of a holding time of 0 h, the samples were cooled down just at the reaching of the sintering temperature.

The density of the sintered compacts was measured by the Archimedes' principle. Beam samples of about 3 mm × 2 mm × 30 mm, for bending strength determinations were cut from larger sintered tiles. All samples were carefully

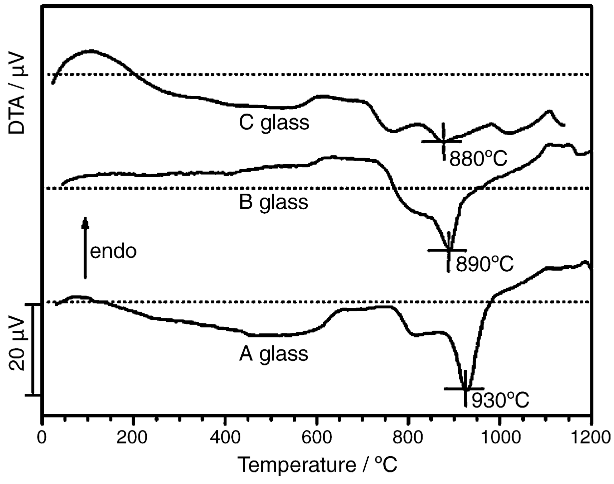

Fig. 1. DTA plots of the investigated glasses.

polished to a 6 μm finish, by using abrasive papers and diamond paste. The edges of the bars were bevelled by using fine abrasive papers and diamond paste. The Young's modulus was measured by means of non-destructive resonance frequency testing (GrindoSonic Mk5, Leuven, Belgium). Four point bending tests (24 mm outer span, 8 mm inner span) were performed by using an Instron 1121 UTS (Instron, Danvers, MA), with a crosshead speed of 0.2 mm/min. The reported results are the average of about 10 flexural tests for each specimen. The glass-ceramics were subjected to SEM observation (Philips XL 30 ESEM, Eindhoven, The Netherlands) and X-ray diffraction analysis (in powdered form) (XRD, Philips PW3710, Eindhoven, The Netherlands).

3. Results and discussion

The sintering treatments at the crystallisation temperature were effective, for all glasses, to produce glass-ceramics. The employed temperatures were particularly low (ranging between 880 and 930 °C) and the treatments were much shorter than those required for conventional nucleation/crystal growth treatments.

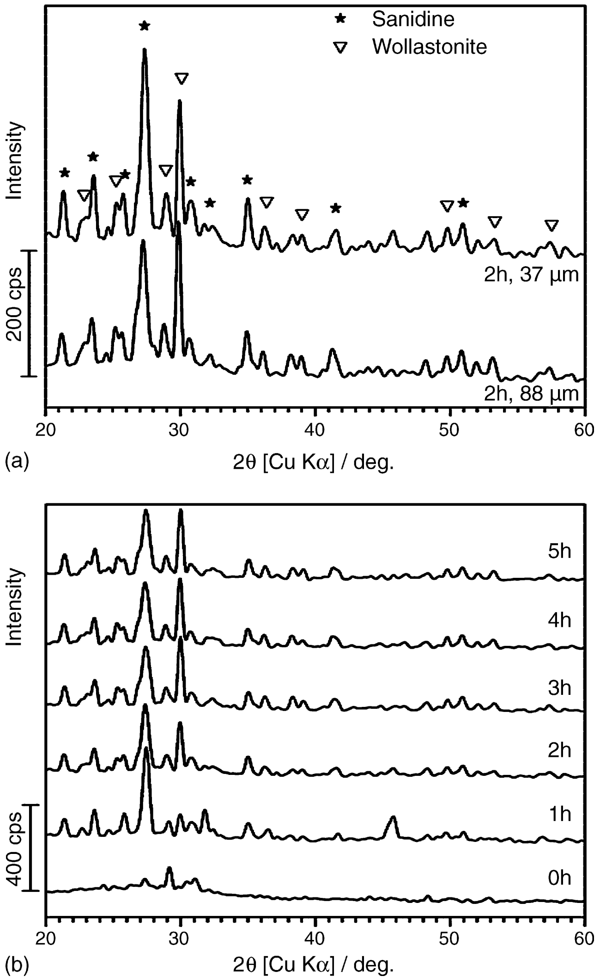

Fig. 2. XRD spectra of glass-ceramics from A glass: (a) comparison between coarser and finer powders and (b) phase evolution as a function of sintering time (finer powders).

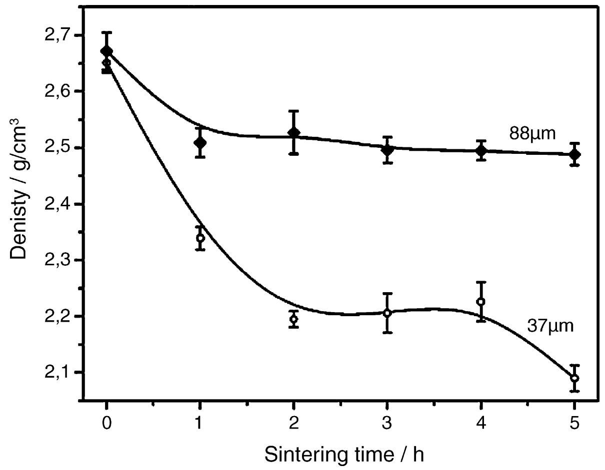

Fig. 3. Sintered density of glass-ceramics from A glass (coarser and finer powders).

The XRD spectra of glass-ceramics from A glass are reported in Fig. 2. In Fig. 2a, it may be seen that finer powders, with the same treatment (2 h at 930 °C), produced a more pronounced crystallisation than coarser powders, thus confirming the surface mechanism of crystallisation. It can be observed also that the crystallisation is almost complete for short treatments (3 h, see Fig. 2b). The precipitated phases consisted of wollastonite ($CaO \cdot SiO_2$) and sanidine ($(K, Na)(AlSi_3)O_8$. Wollastonite is due to the high content of CaO in the composition of A glass, and it could be favoured by the sintering temperature, close to the temperature of maximum crystallisation rate for the same phase (950 °C [1]). The formation of sanidine is rather surprising, since this phase corresponds to an alkali feldspar, a mineral which is known to be easily vitrified but hardly converted to a glass-ceramic: glasses from feldspars are known to be unable to crystallise in practical times [1]. The particular phase was likely due to the high Al_2O_3 content of A glass, accompanied by a remarkable content of alkali ($\sim$10%), which caused the achievement of the optimum composition for sanidine, and to the mechanism of surface crystallisation, which allows very short crystal precipitation times, like in [16,19]. In Fig. 2b, it can be seen that sanidine was formed very rapidly (relevant peaks are visible after 1 h), while the completion of wollastonite precipitation took a longer time (3 h).

Table 2
Elastic moduli of the investigated glass-ceramics

Sintering time (h)	Young's modulus (GPa)					
	A glass		B glass		C glass	
	88 μm	37 μm	88 μm	37 μm	88 μm	37 μm
0	82.9	80.4	82.1	81.0	32.6	22.6
1	99.3	57.1	76.1	71.2	70.9	74.0
2	88.3	47.6	63.8	61.1	67.5	70.7
3	85.2	50.1	63.9		69.6	68.9
4	81.6	48.8	62.2			
5	76.6	41.7	61.8			

Table 3
Bending strength values of the investigated glass-ceramics

Sintering time (h)	Bending strength (MPa)					
	A glass		B glass		C glass	
	88 μm	37 μm	88 μm	37 μm	88 μm	37 μm
0	77.9 ± 12.9	62.0 ± 8.9	58.6 ± 11.9	63.6 ± 10.5	30.0 ± 1.6	22.7 ± 7.1
1	77.7 ± 8.6	54.2 ± 12.1	57.4 ± 12.5	73.2 ± 10.5	73.3 ± 17.9	85.3 ± 19.4
2	75.3 ± 11.6	45.7 ± 5.7	60.8 ± 7.2	74.2 ± 7.8	91.8 ± 13.1	109.2 ± 11.5
3	73.5 ± 8.5	52.4 ± 4.5	59.6 ± 5.9		92.1 ± 16.3	93.3 ± 14.8
4	73.3 ± 7.2	50.7 ± 7.1	65.7 ± 6.3			
5	78.6 ± 6.1	49.6 ± 3.8	67.6 ± 2.5			

Although the precipitation of crystal phase was stronger for finer powders, the glass-ceramics from A glass resulted in an anomalous behaviour, since the density decreased with increasing sintering time (see Fig. 3), and the samples from finer powders revealed poor mechanical properties. The elastic modulus (see Table 2) exceeded 80 GPa (a typical value for aluminosilicate glasses [20,21]) for samples from coarser and finer powders sintered with 0 h holding time, but with increasing sintering time it remained almost stationary for coarser powders and strongly decreased for finer powders, approaching 50 GPa, in spite of the increase of crystal precipitation. The bending strength (see Table 3) confirmed this trend, being ~80 and ~50 MPa for samples from coarser and finer powders, respectively.

One likely reason of the observed anomaly may be illustrated by Fig. 4. The glass-ceramics sintered at 930 °C with a 0 h holding time showed a compact microstructure, with small spherical pores (see Fig. 4a). A relatively thick grain boundary between several glass grains is evident, probably due to the beginning of crystallisation. The glass-ceramics sintered for 5 h (see Fig. 4b) showed large fibrous wollastonite crystals together with several irregular pores, much larger than those visible for 0 h, which consequently cannot be attributed to the sintering process. One likely reason of the observed pores is the formation of wollastonite, which contrasted the contraction,

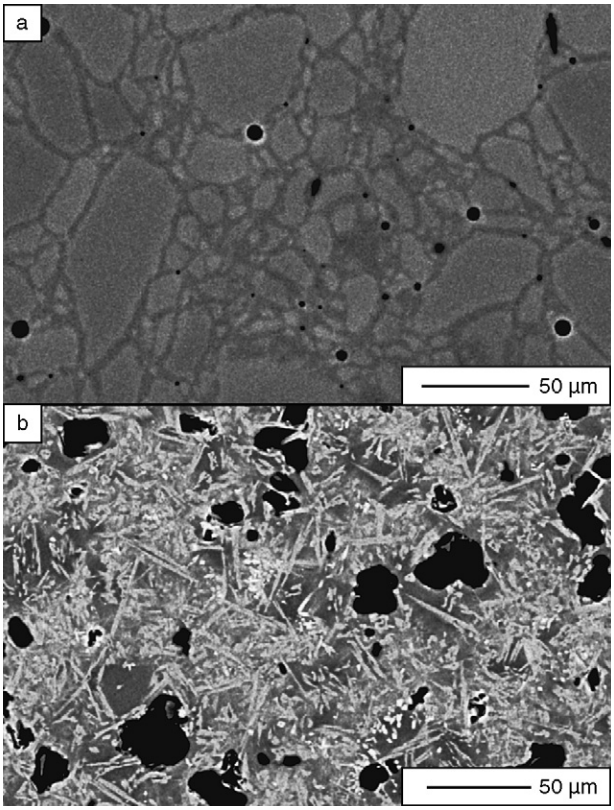

Fig. 4. SEM micrographs of glass-ceramics from A glass (finer powders): (a) 0 h and (b) 5 h at 930 °C.

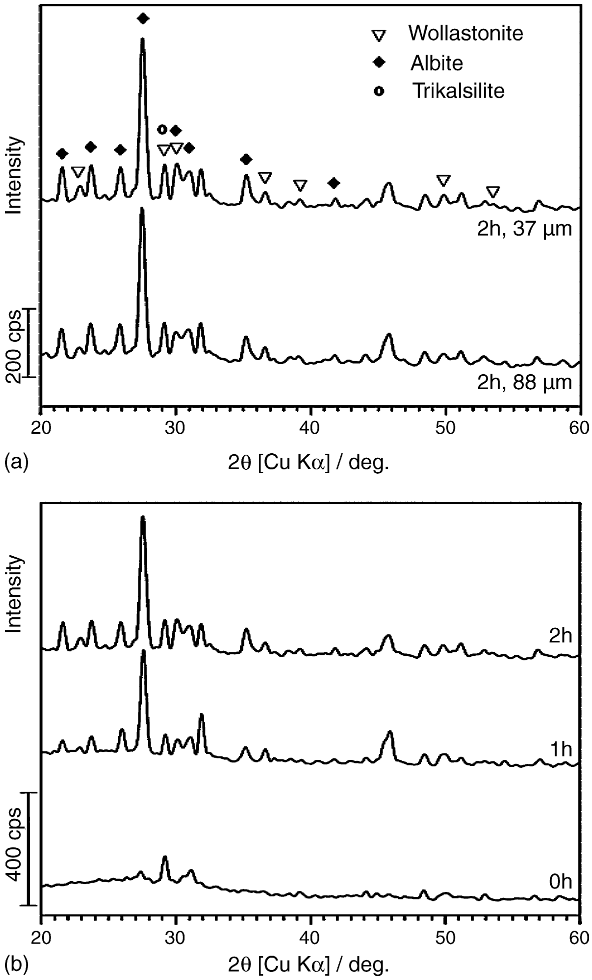

Fig. 5. XRD spectra of glass-ceramics from B glass: (a) comparison between coarser and finer powders and (b) phase evolution as a function of sintering time (finer powders).

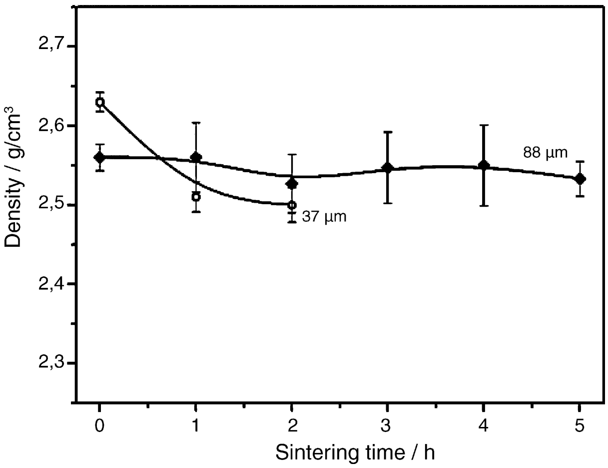

Fig. 6. Sintered density of glass-ceramics from B glass (coarser and finer powders).

upon cooling, of the residual glass matrix, due to lower thermal expansion coefficient (6.5×10^{-6} °C^{-1} against 9.4×10^{-6} °C^{-1} for A glass); the tensile stresses consequently developed in the matrix promoted the formation of pores upon holding at 930 °C. Such effect is more evident with finer powders, which enhanced the crystallisation phenomena, while for coarser powders a sort of equilibrium between the formation of pores and the strengthening effect of crystal precipitation may be found. As an evidence of the role of wollastonite, glass-ceramics from A glass may be satisfactory only for a holding time of 1 h (elastic modulus of about 100 GPa, bending strength of about 80 MPa), for a very limited amount of CaO.SiO$_2$ crystals, from coarser powders.

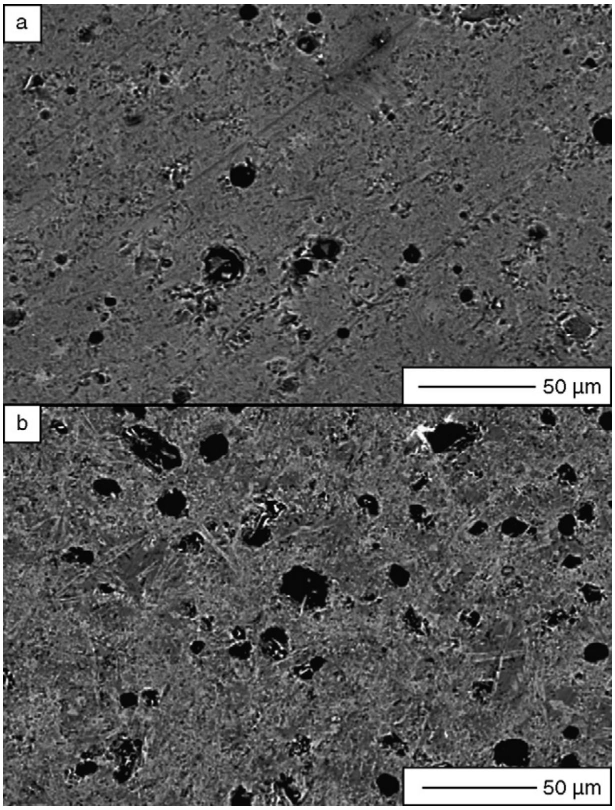

Fig. 7. SEM micrographs of glass-ceramics from B glass (coarser powders): (a) 0 h and (b) 5 h at 890 °C.

B glass showed an analogous behaviour to A glass. In addition to wollastonite, aluminosilicate phases consisting of albite (NaAlSi$_3$O$_8$) and traces of trikalsilite (KNaAlSiO$_4$) were formed (see Fig. 5). Albite is a sodium feldspar, similar to sanidine; even in this case the sintering approach led to the formation of crystals hardly found in glass-ceramics. The formation of pores, for B glass, was less substantial than in the previous one, so that the density showed a decrease only for glass-ceramics from finer powders (see Fig. 6) and the bending strength increased with increasing sintering time, reaching almost 75 MPa, for finer powders (see Table 3). The Young's modulus of the glass-ceramics from B glass reached an almost stationary level of about 60 GPa (see Table 2).

The reduction of pore formation (see Fig. 7) is attributable to the decrease of wollastonite content compared to glass-ceramics from A glass, due to the lower sintering temperature (890 °C), far from the temperature of maximum wollastonite formation. The limited wollastonite content in glass-ceramics from B glass may also be due to the reduction of CaO content,

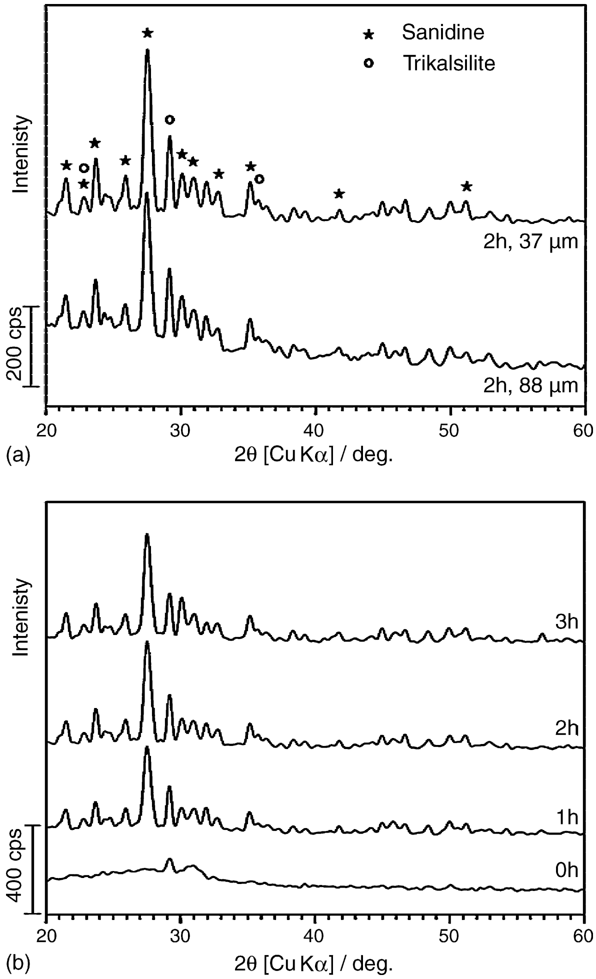

Fig. 8. XRD spectra of glass-ceramics from C glass: (a) comparison between coarser and finer powders and (b) phase evolution as a function of sintering time (finer powders).

and in the increase of Al_2O_3 content, favourable to the formation of aluminosilicates phases. It must be observed the formation of small crystals, likely attributable to trikalsilite (the first precipitated phase, see Fig. 5b), even with a 0 h treatment. However, the mechanical properties of the glass-ceramics cannot be satisfactory.

The formation of wollastonite, detrimental to the mechanical properties of glass-ceramics from A glass and B glass, was avoided for C glass, sintered at 880 °C and featuring the lowest CaO content. As illustrated by Fig. 8 the main crystal phases were sanidine and trikalsilite. The crystal formation is almost complete after 2 h (see Fig. 8b). The density did not reveal the anomalies of the previous cases, as illustrated by Fig. 9: the density increased with increasing sintering time and crystal formation. It should be noted that the densities from coarser and finer powders were similar, except for 0 h: the formation of crystals, more rapid for finer powders, probably retarded the initial viscous flow densification, due to the increase of the density of the glass provided by the crystal inclusions. The bending strength reached excellent values for both coarser and finer powders (the strength of samples from finer powders, sintered for 2 h, exceeded 100 MPa, reaching 109 ± 11 MPa, as shown by Table 3). The Young's modulus of all the glass-ceramics, sintered for more than 1 h, reached a stationary level of about 70 GPa (see Table 2).

The densification and mechanical behaviour were confirmed by the SEM observation, as illustrated by Fig. 10. Very small (3–4 μm), homogeneously distributed, fibre-like crystals of sanidine are visible, with no presence of large pores, likely for the thermoelastic compatibility with the residual glass. In addition to remarkable mechanical properties (preliminary Vickers' microhardness values of about 7–7.4 GPa were achieved for samples sintered for 2 or 3 h at 880 °C), the micron-sized crystals gave a pleasant white homogeneous appearance to the glass-ceramics. The C glass composition, as a consequence, may have a good potential for the production of economic strong sintered tiles, the processing temperature being far lower that of traditional ceramics (<900 °C). The completion of the characterisation (from both mechanical and chemical point of view) of the achieved glass-ceramics will constitute the focus of future work.

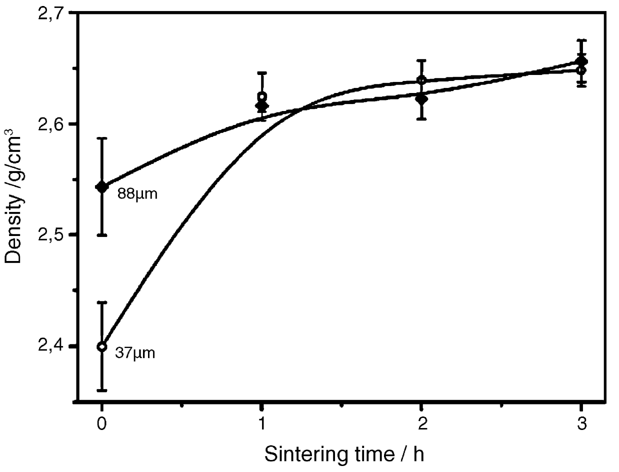

Fig. 9. Sintered density of glass-ceramics from C glass (coarser and finer powders).

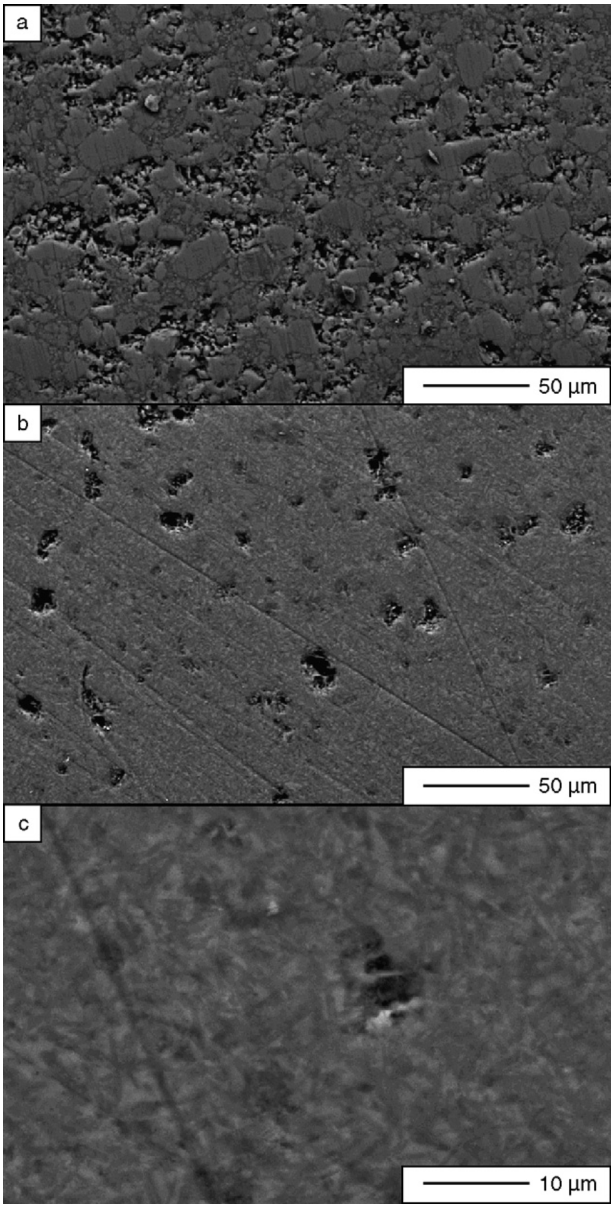

Fig. 10. SEM micrographs of glass-ceramics from C glass (finer powders): (a) 0 h, (b) and (c) 3 h at 880 °C.

4. Conclusions

Glasses from mixtures of wastes were converted into glass-ceramics by a simple and economic sintering treatment with concurrent crystallisation. The main features of the obtained glass-ceramics may be summarized as follows:

- The formation of aluminosilicate phases is more profitable for achieving compact and strong glass-ceramics than the formation of wollastonite, which is characterised by a large thermoelastic mismatch with the parent glass, causing the formation of pores upon sintering. Low sintering temperatures may have a positive effect in reducing the wollastonite formation.

- The relatively high alumina content of the glasses from wastes promotes the formation of alkali feldspar crystal phases, hardly found in glass-ceramics, favoured by the enhanced crystallisation through surface nucleation. Glass-ceramics with feldspars as the main crystal phases, to the authors' knowledge, have not been reported before. The study of the particular precipitation, owing to the investigated combinations of wastes, will need future efforts.
- The strength of the sintered glass-ceramics from C glass, featuring micron-sized alkali feldspar crystals, is remarkable (exceeding 100 MPa), so that the obtained product may be compared to commercial glass-ceramics like ‘‘Neoparies’’ and ‘‘Slagsitalls’’, for applications in the building industry.
- The processing temperatures are particularly low with respect to most types of traditional ceramics, and the duration of the sintering is far shorter than for most glass-ceramics, so that the proposed approach could be useful for the production of cheap building materials, which in turn could be highly profitable for a large absorption of the investigated wastes.

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