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Ceramics International 36 (2010) 1675–1680
CERAMICS INTERNATIONAL
www.elsevier.com/locate/ceramint

Optimisation of sintered glass–ceramics from an industrial waste glass

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Received 10 January 2010; received in revised form 25 January 2010; accepted 28 February 2010
Available online 2 May 2010

Abstract

Industrial plasma melting of municipal solid waste (MSW) incinerator fly ashes leads to a glass that may be easily crystallised to gehlenite glass–ceramics, by the sintering of fine glass powders. However, since the glass composition is not optimised for glass–ceramic manufacturing, the viscous flow is much hindered by a very significant surface crystallisation and dense glass–ceramics are feasible only by sintering above 1000 °C. This paper reports a new strategy for obtaining dense and strong glass–ceramics at 950 °C, with a holding time of only 30 min, consisting of the mixing of waste glass with a secondary recycled glass, such as soda-lime–silica glass or borosilicate glass. For an optimum balance between the two types of glass also the addition of kaolin clay, in order to favour the shaping, was found to be feasible. The approach had a positive effect, besides on the mechanical properties (e.g. bending strength exceeding 100 MPa), on the chemical stability.

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

1. Introduction

Recent papers have shown that sinter-crystallisation is a very effective process for obtaining strong and dense glass–ceramics from waste glasses, i.e. glasses from the melting of several kinds of industrial waste [1–7]. The technology relies on the viscous flow sintering of fine glass powders with concurrent crystallisation, in turn due to a surface mechanism [8,9]. Sinter-crystallisation allows very short processing cycles, on the basis of a short single step firing at the glass crystallisation temperature accompanied by rapid heating and cooling; in some cases even direct heating, i.e. the putting of pressed glass powders directly in the furnace at the crystallisation temperature, was possible [5–7]. Secondly, sinter-crystallisation may lead to rather unusual crystal phases, such as feldspar and feldspathoids [1–3].

A critical point in sinter-crystallisation, evidenced by the latest papers, may be the poor cooperation between the two phenomena of viscous flow sintering and surface crystallisation: if the glass is particularly prone to surface devitrification, crystallisation may be even excessive, determining a number of rigid crystal inclusions, which greatly delay the viscous flow and cause the obtained products to be very porous [7]. The delay in sintering is expected when the glass sintering range, approximately 100–150 °C above the dilatometric softening temperature [10], overlaps with the crystallisation range, i.e. temperatures close to the exothermic crystallisation peak in DTA plots. This fact has been experimentally demonstrated [7] for a glass from plasma melting of municipal solid waste incinerator fly ashes, operated by Europlasma (Bordeaux, France), a company dealing with the plasma vitrification of several wastes [11]. Since the glass composition depends only on the composition of the starting residue and it is not specifically designed for any specific engineering application, the obtainment of dense sintered glass–ceramics was possible only for rapid heating cycles at 1050 °C, far higher than the glass crystallisation temperature, being about 850 °C.

The present paper reports a further development in the valorisation of Europlasma's glass for the manufacturing of dense and strong sintered glass–ceramics, based on the introduction of a secondary glass as sintering aid. The addition of secondary glass led to the development of strong glass–ceramics even with a firing temperature limited to 950 °C, by complex interactions with both viscous flow sintering and crystallisation, and positively influenced the chemical stability.

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0272-8842/$36.00
doi:10.1016/j.ceramint.2010.02.047

Table 1
Chemical composition and characteristic temperatures of P (“Plasmalit”), S (soda-lime–silica) and B (borosilicate) glasses.

Glass	P	S	B
Chemical composition (wt.%)			
SiO_2	40.7	71.6	72.0
Al_2O_3	12.6	1.0	7.0
CaO	32.2	9.0	1.0
MgO	3.3	3.9	
Na_2O	4.6	13.5	6.0
K_2O	0.3	0.4	2.0
Fe_2O_3	1.0	0.1	
Cr_2O_3	0.7		
TiO_2	1.5		
P_2O_5	2.2		
B_2O_3			12.0
Other	0.9	0.5	
Characteristic temperatures (°C)			
Transformation point (T_g)	710	570	590
Dilatometric softening point (T_d)	750	640	645
Crystallisation temperature (T_{cryst})	850 [powders <37 μm]		

2. Experimental procedure

The chemical composition of the starting glass [12], produced from the melting of MSW fly ashes (“Plasmalit” glass, later referred as “P” glass), is reported in Table 1; the same table also reports some characteristic temperatures of the glass. The material, directly available from the plasma melter in the form of flakes (with dimensions of about 25 mm × 15 mm × 1.5 mm), obtained by lamination through cold steel rollers, was dry ball milled and sieved to a dimension <37 μm. Powders of the same size were obtained by the milling of common soda-lime–silica glass (“S” glass), whose composition [13] and characteristic temperatures are also reported in Table 1.

Sintering experiments were first performed on disc samples, with a diameter of about 30 mm and a height of 2 mm, obtained by uni-axial pressing at 40 MPa of fine powders in a cylindrical steel die at room temperature, without any binder. The powders consisted of pure P glass (P100) and mixtures of P glass with soda-lime–silica glass, which replaced P glass in a quantity of 10 wt.% (P90S), 20 wt.% (P80S) and 30 wt.% (P70S). Further samples were obtained from aqueous slips, in which kaolin clay was added to the glass powders (P80S-C; P70S-C) in a fixed amount of 10 wt.%, together with 40 wt.% distilled water. The slips were homogenised in a ceramic ball mill (200 rpm/min) for 30 min, then dried at 110 °C overnight and sieved to granules with a dimension of about 100 μm, employed for uni-axial pressing at 40 MPa. Further samples (P80B-C; P70B-C) were prepared from analogous aqueous slips based on P glass, recycled glass and kaolin clay, made by considering pharmaceutical borosilicate glass (“B” glass) [14], again presented in Table 1, instead of soda-lime–silica glass.

All the samples were sintered at 950 °C, for 30 min, with a heating rate of 40 °C/min and with natural cooling after the holding stage. The colour of the samples varied from bronze green (P mixed with S glass) to ecru (P mixed with clay and B glass). For selected compositions sintering was performed also on larger samples, with dimensions of about 40 mm × 30 mm × 3 mm, obtained by uni-axial pressing at 40 MPa of fine powders in a rectangular steel die at room temperature. These samples were later cut in small beams of approximately 3 mm × 2 mm × 30 mm, for bending strength (σ_{BS}) determinations. All beams were carefully polished to a 6 μm finish and chamfered at the edges, using abrasive papers and diamond paste. The Young’s modulus (E) was measured by non-destructive resonance frequency testing (GrindoSonic Mk5, Leuven, Belgium). Four point bending tests (24 mm outer span, 8 mm inner span) were performed using an Instron 1121 UTS (Instron, Danvers, MA), with a crosshead speed of 1 mm/min. Each data point represents the average of at least 10 individual tests. Selected polished samples were employed for Vickers indentation tests at low load (10N), which yielded the micro-hardness (H_V).

The bulk density of the sintered glass–ceramics was measured by the Archimedes’ principle. At least 10 fragments were analysed for each sample. The true density of the glass–ceramics was evaluated by means of a gas pycnometer (Micromeritics AccuPyc 1330, Norcross, GA).

Polished samples were characterised by scanning electron microscopy (ESEM Quanta 200, FEI Company, Eindhoven, The Netherlands). Powdered glass–ceramics were investigated by X-ray diffraction (Bruker D8 Advance, Karlsruhe, Germany), employing CuKα radiation (0.15418 nm). The identification of crystal phases from diffraction patterns was performed by means of the Match! software package (Crystal Impact GbR, Bonn, Germany), supported by data from PDF-2 database (ICDD-International Centre for Diffraction Data, Newtown Square, PA); the software also provided a semi-quantitative analysis.

Selected samples were subjected to a toxicity evaluation, by application of TCLP leaching test, in analogy with recent researches on glass–ceramics from waste-derived glasses [15]: small fragments from bending strength determinations (<9 mm) were placed in a extraction solution, consisting of acetic acid diluted in distilled water, with a pH value of about 5, for a liquid to solid ratio of 20, and stored at 25 °C for 18 h. The resulting solutions were filtered through a 0.6 μm filter and analysed using inductively coupled plasma (ICP, SPECTRO Analytical Instruments GmbH, Kleve, Germany).

3. Results and discussion

When sintered without additives (P100) at 950 °C, P glass led to a glass–ceramic with about 8% total porosity, consistent with the findings of the above cited previous research [7]. The main crystal phase, as shown by the diffraction patterns in Fig. 1, is a solid solution of the melilite group, which features gehlenite ($Ca_2Al_2SiO_7$) and akermanite ($Ca_2MgSi_2O_7$) as main end members [16]; the most probable phases, recognised by the semi-automatic phase identification provided by the Match! software package, were actually closer to gehlenite end

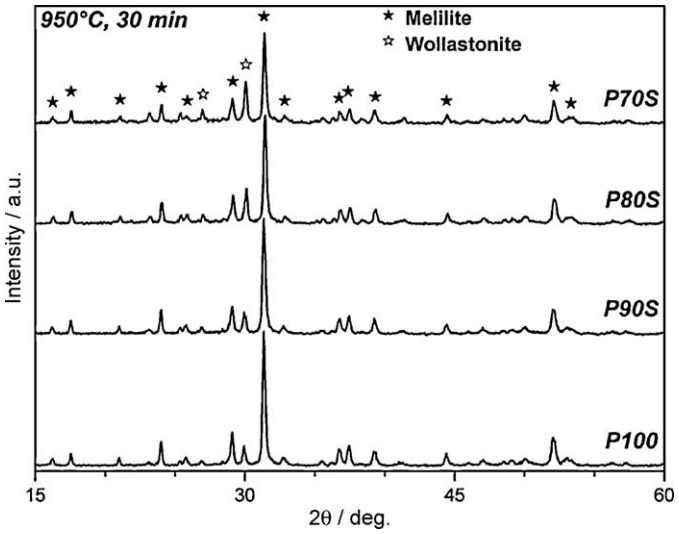

Fig. 1. Phase evolution of mixtures of P glass and S glass, fired at 950 °C, with increasing S glass content.

Table 2
Quantity of crystal phases and porosity of samples from mixtures of P glass and S glass.

Sample	wt.% of crystal phases		Bulk density (g/cm^3)	Residual porosity (%)
	Melilite	Wollastonite		
P100	50	6	2.67 ± 0.02	7.8
P90S	45	8	2.76 ± 0.02	6.3
P80S	42	11	2.71 ± 0.01	5.8
P70S	37	13	2.67 ± 0.01	2.8

member, being $Ca_2(Mg_{0.25}Al_{0.75})(Si_{1.25}Al_{0.75}O_7)$ (corresponding to ICDD PDF#792422) and $(Ca_{1.96}Na_{0.05})(Mg_{0.24}Al_{0.64}Fe_{0.12})(Si_{1.39}Al_{0.61}O_7)$ (corresponding to ICDD PDF#722128). In Fig. 1 also minor traces of wollastonite ($CaSiO_3$, ICDD PDF#731110) can be recognised. From the semi-quantitative analysis also, provided by Match! and reported in Table 2, the total quantity of melilite solid solution corresponds to about 50 wt.%, while wollastonite content is about 6 wt.%.

The addition of soda-lime–silica glass had a substantial influence on the balance between densification and crystallisation of the investigated waste glass. Fig. 1 shows that the height of melilite peaks, and also the quantity of that phase, as reported in Table 2, significantly decreased with increasing S glass content, from about 60 to 40 wt.%; on the contrary, the intensity of wollastonite peaks increased almost linearly with S glass content. The change in the melilite/wollastonite balance, i.e. in the balance between alumino-silicates and silicates (approximately from 8:1 to 3:1), was first attributed to the decrease of the alumina/silica ratio with increasing S content (the alumina content in common soda-lime–silica glass is practically negligible). As shown by Table 2, the residual porosity had a significant reduction with increasing S glass content; the porosity is particularly low for the addition of S glass in the amount of 30 wt.% (P70S): this formulation was therefore used as a reference for further experiments, together with that with a slightly lower content of recycled glass (P80S).

It must be noted that the addition of soda-lime–silica glass has a complex impact on the sinter-crystallisation of P glass: the secondary glass does not improve the densification by simply providing more liquid phase upon sintering (i.e. by ''diluting'' the glass which is subjected to an excessive devitrification), but it affects the overall crystallisation sequence. This fact has an important consequence on the valorisation of waste glasses: dense (and reasonably strong) glass–ceramics from waste glasses are not feasible only by the optimisation of the composition of wastes *before* vitrification, as usually found in the literature [9,17,18] but also by the optimisation of the content of secondary glass *after* vitrification.

The addition of kaolin clay led to further changes. The evolution of water, due to the conversion of kaolin into metakaolinite, at about 550 °C, did not determine a significant increase in porosity in the obtained glass–ceramics, as shown by Table 3. The most interesting effect of kaolin clay concerns the crystallisation: as an example, for the mixture with 70 wt.% P glass and 30 wt.% S glass, shown in Fig. 2, the addition decreased the peaks in the diffraction patterns of both melilite solid solution (M) and wollastonite (W); traces of anorthite–labradorite (L), i.e. calcium–sodium feldspar ($Ca_{0.65}Na_{0.32}Al_{1.62}Si_{2.38}O_8$, ICDD PDF#831367) are also visible. As found in previous papers [6,19] the formation of a feldspar is likely promoted by a direct glass/clay reaction, i.e. an interaction between CaO and Na_2O, dissolved in the glass, and metakaolinite, from the dehydration of kaolin clay. From Table 3 it can be observed a decrease of the overall crystallinity (from more than 50%, without clay, to about 40%): this fact justifies the observed good densification, despite water evolution, on the basis of an enhanced viscous flow of the residual glass.

The mechanical properties of samples from a mixture with 70 wt.% P glass and 30 wt.% S, added with clay (P70S-C), are particularly interesting. The values of elastic modulus, bending strength and Vickers micro-hardness are comparable to those exhibited by the best traditional ceramics for applications in the

Table 3
Quantity of crystal phases, porosity and mechanical properties of samples from P glass/recycled glass/clay mixtures.

Sample	wt.% of crystal phases			Residual porosity (%)	Mechanical properties
	Melilite	Wollastonite	Anorthite–Labradorite		
P80S-C	31	5	5	3.9	Not determined
P70S-C	27	6	6	4.2	$E = 88.5 \pm 3.1$ GPa, $\sigma_{BS} = 99.2 \pm 5.2$ MPa, and $H_V = 6.1 \pm 0.4$ GPa
P80B-C	21	12	16	5.9	$E = 88.9 \pm 2.8$ GPa, $\sigma_{BS} = 101.6 \pm 8.2$ MPa, and $H_V = 6.5 \pm 0.5$ GPa
P70B-C	16	11	19	6.0	$E = 86.2 \pm 1.4$ GPa, $\sigma_{BS} = 113.7 \pm 10.1$ MPa, and $H_V = 6.6 \pm 0.2$ GPa

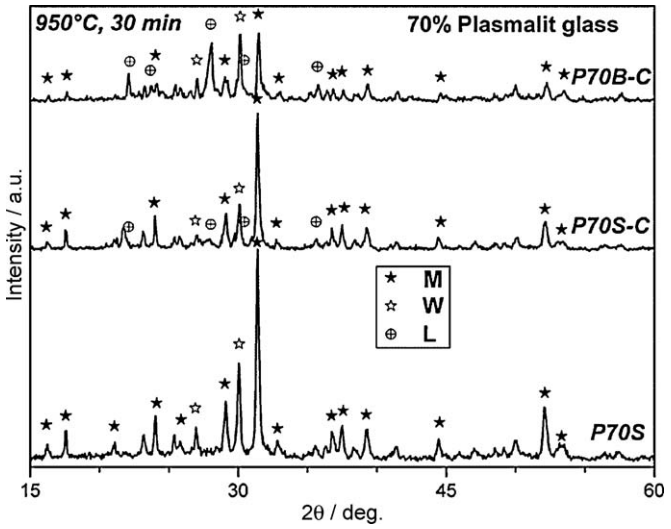

Fig. 2. Changes in the crystallisation of P glass/recycled glass mixture associated to the addition of clay and to the replacement of S glass with B glass (M, melilite; W, wollastonite; L, anorthite–labradorite).

building industry [20]. Mechanical properties were not determined on P80S-C samples, which were available only in the form of small discs; however, significant variations from the values for P70S-C are not expected, due to the similarity in physical properties (proportions between different phases and porosity).

The successful introduction of kaolin clay stimulated the testing of further mixtures, corresponding to the samples P80B-C and P70B-C, in which soda-lime–silica glass was replaced by pharmaceutical borosilicate glass. The replacement was mainly aimed at evaluating the impact of a glass with a much lower content of CaO. Fig. 2 and Table 3 show that the change in the secondary glass had an important impact on the crystallisation: borosilicate glass greatly promoted the formation of anorthite–labradorite, with a dramatic decrease of the peak associated to melilite.

In our opinion, the findings about borosilicate glass give a new light on the behaviour of P glass in the presence of any additive. In particular it is interesting to evaluate the improved formation of feldspar crystals with respect to the case of the introduction of soda-lime–silica glass. The interaction between metakaolinite with CaO, leading to anorthite–labradorite is likely influenced by the different diffusion conditions corresponding to the two types of recycled glass added. The sintering of powders of P glass with powders of another glass determined the joining of regions with different contents of Ca^{2+} ions, i.e. led to compositional gradients, obviously more significant with borosilicate glass. In other words, the secondary glass is useful for modifying the mobility of ions dissolved in P glass. Metakaolinite, present at the boundaries between former glass powders (due to the manufacturing process), reasonably captures the already extracted Ca^{2+} ions; Na^{+} ions are likely extracted from the secondary glasses (richer in sodium oxide than P glass). Also the formation of wollastonite could be interpreted on the basis of a chemical interaction of Ca^{2+} ions, extracted from P glass, with the secondary glass.

It must be noted that due to the much improved yield of feldspar crystals, as reported by Table 3, the overall crystallisation degree for samples P80B-C and P70B-C is much higher than that for samples P80S-C and P70S-C. This increase in turn limited the viscous flow, as evidenced by the relatively high residual porosity of P80B-C and P70B-C. However, it must be noted that the last samples, featuring the introduction of borosilicate glass, exhibit mechanical properties (elastic modulus, bending strength – exceeding 100 MPa – and Vickers micro-hardness) comparable or higher than those of the less porous P70S-C. These mechanical properties are particularly significant since they exceed the values exhibited by the best traditional ceramics, well known to be prepared at much higher temperatures (>1100 °C). In addition, it must be highlighted that all the samples from P glass/secondary glass/clay mixtures, like the best traditional ceramics, feature a very low water absorption, <0.1% (determined following standard procedures [21]). The residual porosity, as a consequence, is almost completely closed.

The complex crystallisation mechanism of samples from P glass/secondary glass/clay mixtures is illustrated by Fig. 3, which include SEM backscattered electron images. The lighter areas in Fig. 3a (P70S-C) and Fig. 3c (P80B-C) can be associated to P glass, possessing species with high atomic number (i.e. iron ions); the darker ones correspond, on the other hand, to the secondary glass. We can observe that there is no defined boundary between the two glasses, due to the viscous flow; in addition, both areas contain a number of different crystals. The lighter zones contain melilite solid solution, which develops in the form of ''clouds'' of very fine crystals (as observed in a previous research [7]), whereas the darker zones feature larger fibrous crystals. In Fig. 3b, corresponding to the introduction of soda-lime–silica glass, the high aspect ratio fibrous crystals are attributable to wollastonite (W; the morphology of wollastonite is consistent with that exhibited in the crystallisation of another waste glass [6]); in Fig. 3d the thicker fibrous crystals are likely associated to anorthite–labradorite. In both cases the crystal phases promoted by the additives, wollastonite and anorthite–labradorite (L), are effectively located at the interface between the P glass and the secondary glass, thus confirming the key role of the diffusion of Ca^{2+} ions.

The fact that the mechanical properties of more porous samples, from the introduction of borosilicate glass, are comparable or even higher than those of less porous samples, from the introduction of soda-lime–silica glass, is reasonably associated to the increased crystallisation degree; however, some effect of the different balance between different crystal phases, and consequent residual thermo-elastic stresses developed upon cooling, cannot be excluded. As an example, gehlenite features a much larger coefficient of thermal expansion (from the literature [16] the volumetric thermal expansion coefficient is reported to be about $28 \times 10^{-6}\ ^{\circ}C^{-1}$, so that the linear coefficients should be about $(9–10) \times 10^{-6}\ ^{\circ}C^{-1}$) than labradorite (about $5 \times 10^{-6}\ ^{\circ}C^{-1}$) [9] and wollastonite (about $6.5 \times 10^{-6}\ ^{\circ}C^{-1}$) [22]. As confirmed by other experiments with P glass and industrial clays [23], the best mechanical properties are

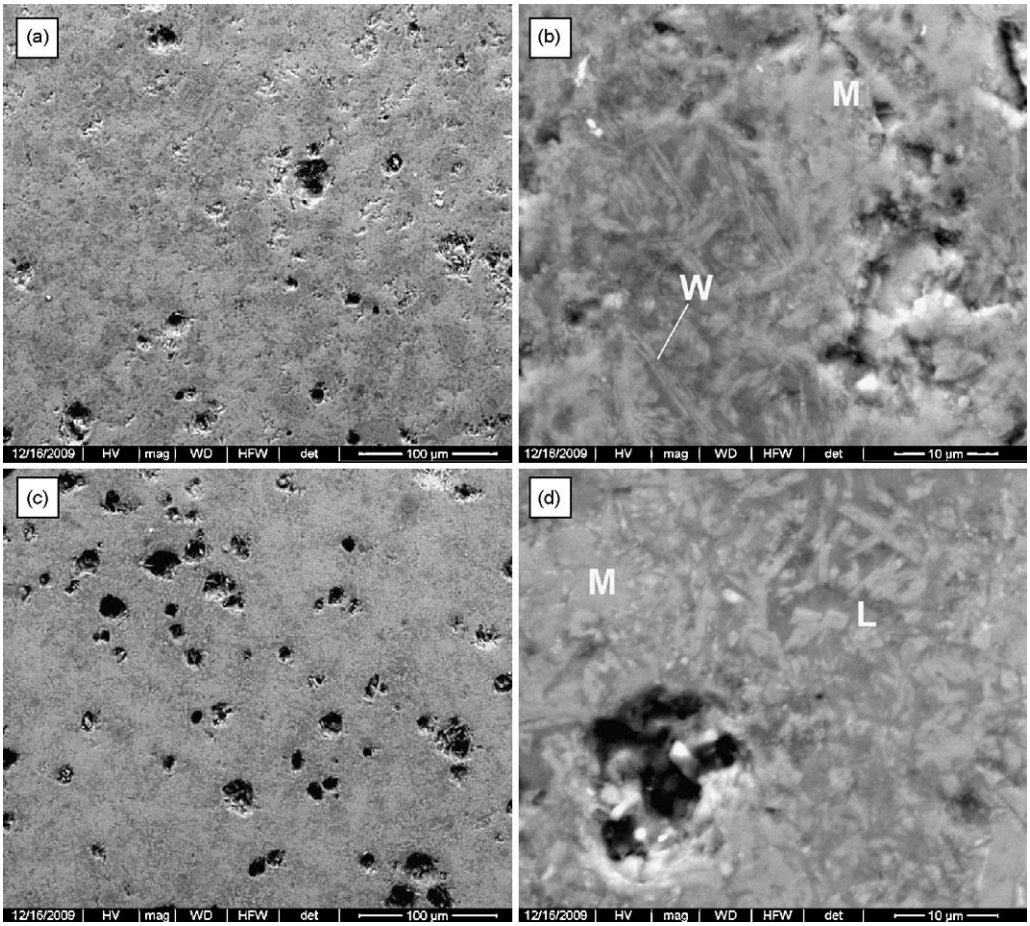

Fig. 3. Microstructural details of selected samples from the sintering of P glass/recycled glass/clay mixtures: (a and b) P70S-C; (c and d) P80B-C (M, melilite; W, wollastonite; L, anorthite–labradorite).

associated to a relatively high labradorite/melilite ratio, as found for P80B-C and P70B-C with respect to P80S-C and P70S-C (the increase of labradorite/melilite ratio, from P80S-C to P70S-C, is limited, passing from 0.16 to 0.22, but becomes very substantial with the change of secondary glass, passing to 0.76 for P80B-C and 1.19 for P70B-C).

The best products from both types of secondary glass, i.e. samples P70S-C and P70B-C, were subjected to a preliminary chemical test, i.e. TCLP leaching test. Table 4 shows that both products, after acid attach, do not release significant quantities of toxic ions, i.e. the releases are well below well recognised standards, like those provided by EPA and reported in the same Table 4 [24]; in particular, the release of chromium, that could be considered as the most dangerous metal dissolved in P glass, is negligible. This means that the glass–ceramic products of the present investigation practically maintain the chemical inertia of P glass vitrification in the as prepared state, already evaluated [11].

Table 4
Results from TCLP leaching test of selected samples.

Metal	Release from P70S-C (mg/l)	Release from P70B-C (mg/l)	EPA limits (mg/l)
Ag	<0.0004	0.0405	5.0
As	<0.0044	<0.0044	5.0
Ba	0.0231	0.0614	100.0
Cd	0.0010	0.0009	1.0
Cr	<0.0007	<0.0007	5.0
Hg	0.0009	0.0009	0.2
Pb	<0.0030	0.0119	5.0
Se	<0.0102	<0.0083	1.0

4. Conclusions

We may conclude that:

- the sinter-crystallisation process of a waste glass can be modified by the addition of a secondary glass; this glass does not simply affect the viscous flow, but it is active on the overall crystallisation sequence, in turn influenced by compositional gradients;
- significant changes in the sinter-crystallisation process are due to the addition of kaolin clay, that promotes the formation of feldspar crystals;
- the type of secondary glass introduced, by modifying the compositional gradients, affects the crystallisation sequence and the interaction with clay;

- strong glass–ceramics from waste glasses can be obtained even without the optimisation of the composition of wastes *before* vitrification, by the optimisation of waste glass/secondary glass/clay mixtures, *after* vitrification;
- the mechanical properties of sintered glass–ceramics from optimised glass/clay mixtures are equal or superior to those exhibited by traditional ceramics, although the sintering is performed at much lower temperatures (950 °C instead of >1100 °C).

Acknowledgements

The authors would like to thank Dr. Erica Edme, Dr. Ulysse Michon and Dr. Nicolas Planty at Europlasma for supplying Plasmalit glass. E.B. acknowledges Dr. Giulia Zanmarchi for the ICP analysis, Dr. Claudio Furlan for the ESEM aanlysis, and Prof. Giovanni Scarinci for a very fruitful and stimulating discussion.

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