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The effect of investment materials on the surface of cast fluorcanasite glasses and glass–ceramics

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Abstract Modified fluorcanasite glass–ceramics were produced by controlled two stage heat-treatment of as-cast glasses. Castability was determined using a spiral castability test and the lost-wax method. Specimens were cast into moulds formed from gypsum and phosphate bonded investments to observe their effect on the casting process, surface roughness, surface composition and biocompatibility. Both gypsum and phosphate bonded investments could be successfully used for the lost-wax casting of fluorcanasite glasses. Although the stoichiometric glass composition had the highest castability, all modified compositions showed good relative castability. X-ray diffraction showed similar bulk crystallisation for each glass, irrespective of the investment material. However, differences in surface crystallisation were detected when different investment materials were used. Gypsum bonded investment discs showed slightly improved in vitro biocompatibility than equivalent phosphate bonded investment discs under the conditions used.

Introduction

Glass–ceramics are polycrystalline solids obtained by controlled devitrification of glasses. Devitrification or crystallisation is accomplished by subjecting suitable glasses to a carefully regulated heat-treatment schedule that results in the nucleation and growth of crystal phases within the glass. Castable glass–ceramic materials have been developed for both dental and medical use, for example as crowns, inlays and as bone substitutes. These materials have shown considerable potential in restorative dentistry [1, 2]. An ideal load bearing bioactive glass–ceramic should have good mechanical properties, have the ability to form complex shapes, should bulk nucleate and be biocompatible [3–7]. An interesting group of castable glass–ceramics are chain silicates. Chain silicates are polymeric crystals in which single or multiple chains of silica tetrahedral form the structural backbone. One promising multiple chain silicate is fluorcanasite [8, 9]. Fluorcanasite can be formed from glasses close to its stoichiometry. Internal nucleation is achieved through precipitation of CaF_2 crystallites and spherulitic growth of fluorcanasite upon these nuclei. Fluorcanasite ($Ca_5Na_4K_2Si_{12}O_{30}F_4$) has a highly crystalline microstructure composed of interpenetrating blades that give rise to a relatively high flexural strength (>300 MPa) and fracture toughness (>5 MPa $m^{1/2}$) [10]. Miller et al. [8] showed that the addition of excess CaO and P_2O_5 to the stoichiometric fluorcanasite glass–ceramic composition induced the early formation of an apatite layer in simulated body fluid, and commented that these materials might be useful for use as bone substitute. It was also suggested that these materials could be cast to shape via the lost-wax casting process (due to their relatively low liquidus temperatures) [9].

Gypsum-bonded and phosphate-bonded investments are used primarily as investment materials for lost-wax casting. Two main criteria governing the selection of investment materials for casting are, the casting temperature of the material being used; and the thermal expansion from the

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investment which compensates for casting shrinkage, to ensure a satisfactory fit. Different glass–ceramic formulations require different lost-wax casting procedural factors to optimise the casting process and the biaxial flexural strength (BFS) [1]. It is therefore essential that effects and variations in procedural factors involved in the lost-wax casting production of glass–ceramic restorations are identified and optimised. The surface roughness of castable glass–ceramics is also dependent on the choice of investment material and the type of castable glass–ceramic used [11]. Walsh et al. [12, 13] assessed the relative castability of apatite–mullite glasses using a spiral test and was found to be linked to composition and therefore cross-link density. The spiral test piece is based on a method for casting alloys and was designed *in house*, at the University of Sheffield, UK. However, no quantitative data has been published to date on castability of fluorcanasite glasses. The aim of this study was therefore to determine the relative castability of modified fluorcanasite glasses and to evaluate the effect of investment materials on the cast surfaces. The effect of two different investment materials on the in vitro biocompatibility of parent glasses and glass–ceramics were also evaluated.

Materials and methods

Glass preparation

Stoichiometric fluorcanasite glass (Glass 1) [10] was used as a reference for the other three modified compositions (Glass 2–4), Table 1, designed to enhance biocompatibility. The glasses were melted in an uncovered platinum-rhodium (2%) crucible at 1,450 °C for 3 h in an electric furnace and stirred for the final 2 h with a platinum stirrer (60 rpm) to encourage homogeneity. To reduce internal stresses the glasses, after casting onto preheated steel plates, were annealed in a muffle furnace for 1 h at 460 °C then cooled at 1 °C min^{-1} to room temperature. Glass frit was prepared by quenching of the molten glass into cold water.

Measurement of castability

Glass frits were used as starting materials. A cm graduated metal cone was used as a standard [12, 13] for measurement of spiral castability (Fig. 1a). Frits of Glass 3 (CaO rich) and Glass 4 (~2% P_2O_5) were used to make complex jaw castings using the conventional lost-wax casting route (Fig. 2a–d).

Table 1 Compositions of glasses in mole percent

Type	SiO_2	CaF_2	Na_2O	K_2O	CaO	P_2O_5
Glass 1	60.0	10.0	10.0	5.0	15.0	0
Glass 2	63.5	10.6	4.8	5.3	15.9	0
Glass 3	61.6	10.3	3.8	5.1	19.2	0
Glass 4	62.7	8.4	3.9	5.2	17.8	2.1

Cast preparation

The lost-wax casting process was used to measure castability. About 3 mm sprue wax, spiral patterns were formed around a graduated metal cone (Fig. 1b). A thin film of cleanser (Tensilab, alcohol based, Zhermack, Italy) was sprayed and dried on the pattern to reduce the surface tension of the wax and to permit better wetting of the investment. The wax pattern was then attached to the sprue former inside a casting ring lined with a ceramic liner (Whip-Mix Ring Liner, Whip-Mix Corp., USA), (Fig. 1c), and then invested with phosphate-bonded investment (Fujivest Super FS, GC Belgium). The investment was mechanically mixed under vacuum (Mulitvac 4, Degussa, Germany) for 40 s and then poured into the casting ring and around the wax pattern. The invested ring was then allowed to bench set for 1 h. Three castings per glass composition were produced.

Furnace and casting procedure

After the investment had set, the sprue former was removed from the casting ring and the ring placed into a burn out furnace for wax elimination. The invested ring was heated to 800 °C to eliminate the wax spiral pattern then, heated at this temperature for 1½ h. The temperature was then reduced to 460 °C prior to casting, to prevent surface nucleation of the glass on contact with the mould. An electric resistance furnace casting machine with centrifugal casting pressure was used (Degussa TS3, Degussa AG Hanau, Germany) to melt and cast each material. About 16 g of molten glass frits (1,450 °C) were cast into the mould. The amount of glass required was calculated by weighing the wax spiral and multiplying this by the glass density. The castings were then annealed at 460 °C for 1 h. After cooling to room temperature the castings were devested and the investment removed from around the casting using 50 μm aluminium oxide blasting at 6 bars of pressure. The casting length was measured against the metal cone and confirmed using a flexible ruler (Fig. 1d).

Measurement of casting shrinkage

Fluorcanasite glass frit of each material (Glass 1–4) was used to produce identical MOD inlays using the lost-wax casting process with gypsum bonded investment (Whip-Mix Cristobalite, Whip-Mix Corp., Louisville, USA) to determine if any shrinkage took place during the controlled

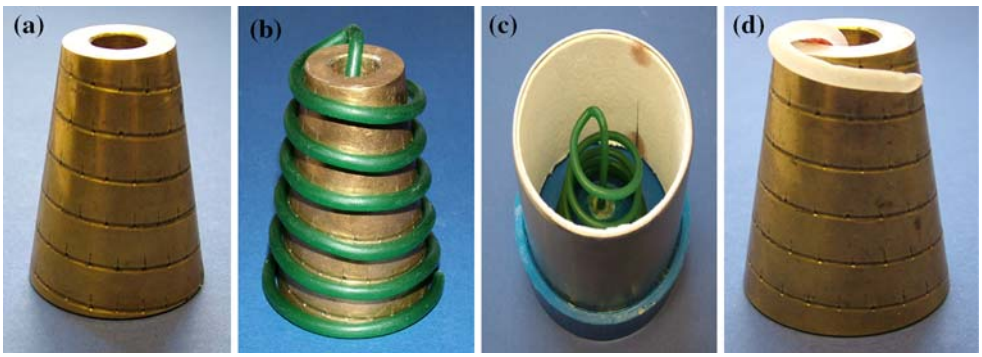

Fig. 1 (**a**–**d**) Measurement of castability using graduated spiral cast: (**a**) Graduated metal cone; (**b**) Metal cone with wax pattern in place; (**c**) Wax pattern ready for investing; (**d**) Metal cone with spiral casting repositioned ready for measuring

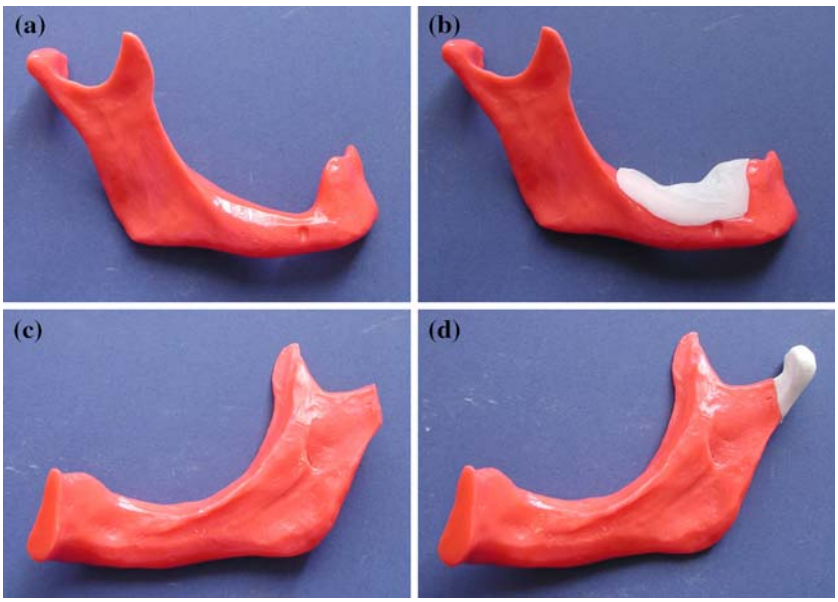

Fig. 2 (**a**, **b**) Photographs showing jaw without and with cast alveolar bone substitute (Glass 3). (**c**, **d**) Photographs showing jaw without and with a replacement condyle casting (Glass 4)

two stage heat-treatment process to convert the as-cast glass to a glass–ceramic. The fit of the crown to the master die at four sites at the interproximal margins were measured using a travelling microscope (±1 μm). Heat treatment was carried out identically for all four glasses by controlled heating from room temperature to 520 °C at 5 °C min^{-1} and then holding for 2 h, followed by a ramp at 3 °C min^{-1} to 780 °C for 2 h. The sample was then cooled at 5 °C min^{-1} to room temperature. The inlays were then de-vested, as previously described for castability testing, and had the sprues removed. The heat-treated ceramic inlays then had the fit remeasured against the master die using the travelling microscope.

Effect of investment materials on cast surfaces

Frit of Glass 2 (reduced Na_2O content), Glass 3 (CaO rich) and Glass 4 (~2% P_2O_5) were used to make lost-wax casting discs (12 mm diameter × 2 mm thickness). Gypsum-bonded investment (Whip-Mix Cristobalite, Whip-Mix Corp., Louisville, USA) and phosphate-bonded investment (Fujivest Super FS, GC Belgium) were used as investment materials to produce discs to assess the effect of investment materials on the cast surfaces of the various compositions. The investment casting rings were heated up to 700 °C and held for 1½ h. The temperature was then reduced to 460 °C prior to casting. After casting the discs were annealed at 460 °C for 1 h.

Measurement of surface roughness

Wax discs 12 mm diameter and 2 mm thick were identically produced from a silicone mould. The discs were sprued and invested using both gypsum and phosphate bonded investment and cast using glasses 2, 3 and 4. After de-vesting with 50 μm aluminium oxide blasting at 6 bars of pressure, the surface roughness was measured using a surface roughness tester (Surftest 301 Mitutoyo Corp Kawasaki, Japan) which is a stylus, surface contact instrument. The total length measured was 4 mm. Each disc was measured twice, in orthogonal directions.

Investment effects on cast surfaces: X-ray diffraction

X-ray diffraction (Siemens D500) was used to characterise both surface and bulk crystallisation of fluorcanasite casting discs of as-cast glass and glass–ceramic materials made using both gypsum and phosphate bonded investment materials by Cu radiation (λ = 1.5406 Å) with an angle range of 5–75°2θ in 0.02°2θ intervals with a speed of 2°2θ min^{-1}. Peaks were analysed using STOE WinXPOW software and JCPDS cards. These XRD traces were compared with the XRD trace obtained from as-cast glass without any investments.

Investment effects on cast surfaces: In vitro biocompatibility

Casting discs (12 mm diameter × 2 mm thickness) were prepared from glass frit using the lost-wax casting technique previously described. The glass discs were then heat-treated in a two stage heat-treatment process to form fluorcanasite glass–ceramics in the hot zone of a calibrated Lenton tube furnace (room temperature to 520 °C, nucleation temperature at 5 °C min^{-1} and held for 2 h, followed by a ramp at 3 °C min^{-1} to 780 °C, crystal growth temperature. Samples were held at this temperature for 2 h and then cooled at 5 °C min^{-1} to room temperature). The as-cast discs and heat-treated glass–ceramic discs were then cleaned in an ultra-sonic cleaner with distilled water. The discs were produced using both gypsum and phosphate bonded investment materials. These discs were then sterilised by autoclaving (15 min at 121 °C/15 psi). Biocompatibility was investigated using rat osteosarcoma (ROS 17/2.8, Merck Inc.) cells seeded into wells of a 24 well plate containing test samples (seeding density of 1.25 × 10^4 cells/ml) with a total well volume of 1 ml. The materials and cells were incubated at 37 °C in a 5% CO_2 atmosphere for 72 h. ROS cells were grown on tissue culture plastic as a control. These methods have been reported previously for the evaluation of in vitro biocompatibility [14, 15].

Cellular response was assessed qualitatively using JEOL 6400 scanning electron microscopy which gives an idea of the morphology of desiccated cells. MTT (3-(4, 5-dimethylthiazol-2-yl)-2, 5-diphenyltetrazolium bromide) assay was also carried out to measure quantitatively the respiratory rate of the cells cultured on samples of both parent glasses and glass–ceramics produced using both gypsum and phosphate bonded investments.

Results and discussion

Glass compositions

Glass 1 is a stoichiometric fluorcanasite composition ($Na_4K_2Ca_5Si_{12}O_{30}F_4$) [10] whereas Glass 2, 3 and 4 are modified in a manner anticipated to induce bioactivity. Osteoconductive materials often contain CaO and P_2O_5 as both these oxides are constituents of human bone. Incorporation of P_2O_5 and/or excess CaO in the starting composition might induce bioactivity in the resulting glass–ceramics. Hence, Glass 2 was modified by reducing Na_2O content as the leaching of Na ions was one of the major problems in the original fluorcanasite compositions [8]. Glass 3 was modified by increasing CaO content and Glass 4 was modified by incorporating P_2O_5 [8]. Excess CaO in Glass 3 and addition of P_2O_5 in Glass 4 were compensated by reducing the Na_2O content in these compositions [8].

Measurement of castability and casting complexity assessment

The castability results are shown in Table 2. Glass 1 (stoichiometric), Glass 3 (CaO rich) and Glass 4 (~2% P_2O_5) have higher castability than Glass 2 (reduced Na_2O content). Glass 1 had the highest castability while increasing calcium concentration relative to sodium did not significantly alter castability. However, castability has varied with glass composition. It is thought that fluoride and sodium ions disrupted the glassy network, lowering the glass transition temperature and making the material less viscous at casting temperature [16]. Glasses with the lowest concentrations of these ions were too viscous to cast using this spiral model [13]. Castability is therefore composition dependent. Several attempts have been made to model the relationship between glass composition and a number of material properties. One such model is the cross-link density, first described by Strnad [17] and modified by Hill [18]. The cross-link density (CLD) is calculated using the formula [18]. CLD = (Number of bridging oxygens/number of network forming species)-2. Castability was linked to composition and to the theoretical cross-link density of each glass formulations. Glasses with highest values for cross-link density were the least castable [13]. In general, modified fluorcanasite glasses have shown good castability and are suitable for the production of custom made prostheses.

Figures 2a,c show a model jaw with defects and glass 3 (Fig. 2b), and glass 4 (Fig. 2d) used to recontour/replace

Table 2 Measurement of castability

Glass	Casting 1 (mm)	Casting 2 (mm)	Casting 3 (mm)	Mean (mm)	S.D. (mm)
Glass 1	30	28	30	29.33	1.15
Glass 2	17	17	14	16.00	1.73
Glass 3	25	20	21	22.00	2.64
Glass 4	21	19	22	20.60	1.53

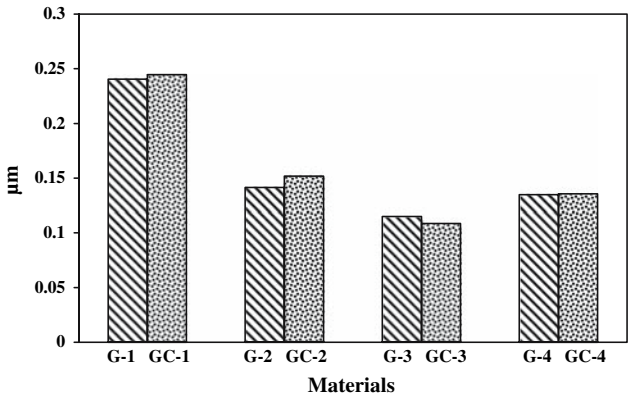

Fig. 3 Measurement of casting shrinkage during two stage heat-treatment process, for glasses 2, 3 and 4; as-cast glass (G) and glass–ceramic (GC)

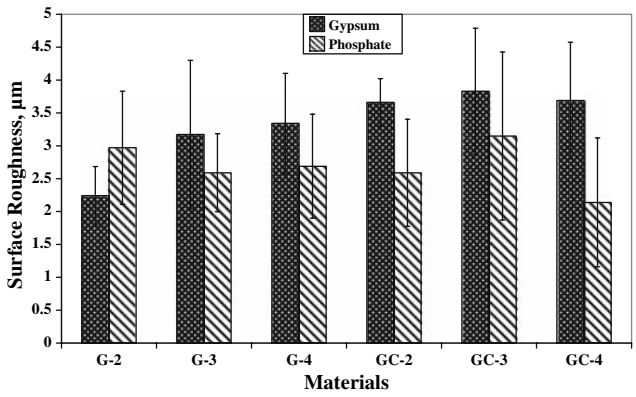

Fig. 5 Determination of surface roughness for Glasses (G) and Glass–Ceramics (GC)

the defects. Both these initial castings reveal that fluorcanasite glasses could be cast to form complex shapes using the lost-wax casting route.

Measurement of casting shrinkage

Figure 3 shows the extent of shrinkage that took place during controlled two stage heat-treatment process. No significant shrinkage was observed when glasses were converted to glass–ceramics. The gap measured between the fit and the crown was visually identical when comparing before (Fig. 4a) and after heat-treatment (Fig. 4b). The slight differences that have been seen in Fig. 3 might come from experimental error during measurement of the fit. All castings seemed to be clinically acceptable.

Effect of investment materials on cast surfaces

Surface roughness

No significant differences in the surface roughness were observed in the glassy and crystallised states for any of the materials tested (Fig. 5). However, as-cast glasses and glass–ceramics with the gypsum bonded investment showed slightly higher surface roughness values than with the phosphate bonded investment, with the exception of Glass 2. For gypsum bonded investments, surface roughness increased from glass to glass–ceramics. This trend was not seen for the phosphate bonded investments.

X-ray diffraction

Figure 6 shows the XRD traces from the surface of castings produced using both gypsum-bonded and phosphate-bonded investments. As-cast casting discs with investment materials as well as as-cast glasses without investments were studied. Glass 2 (reduced Na_2O content) showed a completely amorphous trace irrespective of investment materials. Glass 3 (CaO rich) produced with a gypsum bonded investment revealed some unknown peaks but as-cast Glass 3 with and without phosphate investments was completely amorphous. It is thought that the unknown peaks are due to the presence of some gypsum contamination on the surface. Glass 4 (~2% P_2O_5) in its as-cast state contains fluorite and fluorapatite crystals. Phosphate bonded as-cast Glass 4 shows the same crystalline phases. However, gypsum bonded as-cast Glass 4 shows unidentified peaks along with fluorite and fluorapatite crystals. Figure 7 shows the XRD traces obtained from the bulk of the samples. Glasses 2 and 3 show a completely amorphous nature irrespective of the investment materials. Glass 4 has

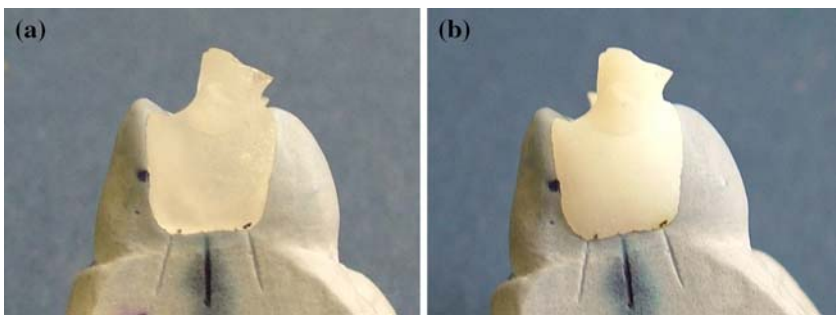

Fig. 4 Measurement of casting shrinkage during two stage heat-treatment process; (**a**) Glass B and (**b**) glass–ceramic B

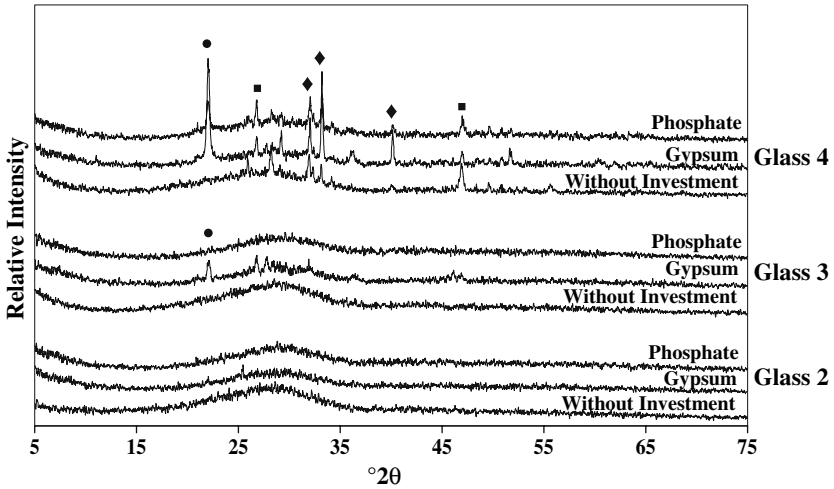

Fig. 6 XRD traces obtained from as-cast surfaces of gypsum and phosphate bonded investment casting discs and pure as-cast glasses: (■) Fluorite, (◆) Fluorapatite and (●) Unknown

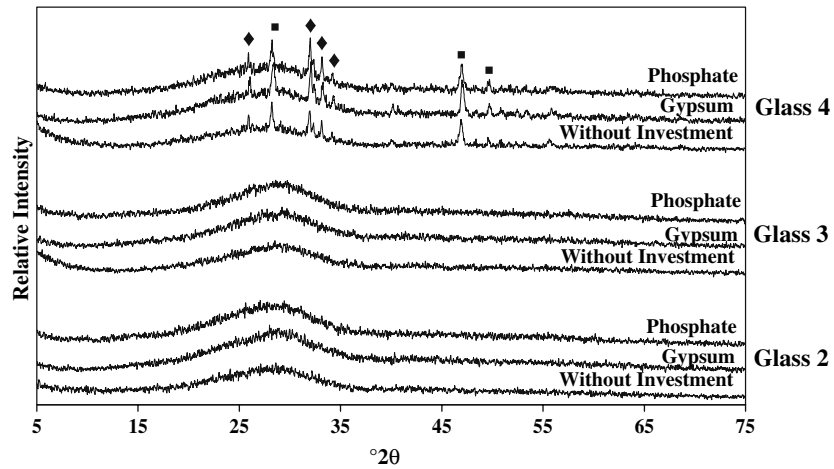

Fig. 7 XRD traces obtained from as-cast bulk of gypsum and phosphate bonded investment casting discs and pure as-cast glasses: (■) Fluorite and (◆) Fluorapatite

only fluorite and fluorapatite crystals in the as-cast state both with and without investments. Further details of the nucleation and crystallisation of these glasses can be found in references [19] and [20].

In vitro biocompatibility

Figure 8a–l shows SEM images of cells cultured on discs, cast using both gypsum and phosphate bonded investments. Cells were able to grow on the Glass and Glass–Ceramic 2, 3 and 4. They formed confluent sheet of flattened cells with classical osteoblast-like morphology on the surfaces of the parent glasses and the glass–ceramics. Following the quantitative MTT assay, fluorcanasite discs showed a poorer cell response than was observed with control tissue culture plastic (Fig. 9). Tissue culture plastics which are considered in this study as a control material for in vitro biocompatibility assessment are undergone through a chemical treatment that increases its adhesiveness for cells and in turn could lead to the improved cell response compared to their glass and glass–ceramic counterparts. In general, glass–ceramics have higher relative absorbance and hence have improved biocompatibility than the parent glasses for both gypsum and phosphate bonded discs (Fig. 9). After controlled heat-treatment process, the parent glasses were converted to glass–ceramics with some crystalline phases mainly consisting of fluorcanasites, frankamenites and in some cases fluorapatites with an interpenetrating lath-like structure [21]. These fluorcanasite and fluorapatite crystals are mainly responsible for the improvement of in vitro biocompatibility of these glass–ceramics from their parent glasses. Bandyopadhyay-Ghosh et al. [21] showed that incorporation of excess CaO (Glass 3) and P_2O_5 (Glass 4) to the stoichiometric glass composition (Glass 1) improved in vitro biocompatibility, as did controlled crystallisation. They concluded that glass composition is a critical determinant of in vitro biocompatibility. Reduced ion release in combination with related pH effects appeared to be the principal mechanisms responsible for the improvement in the in vitro biocompatibility of modified compositions [21]. Glass–ceramics formed with gypsum bonded investment have higher relative

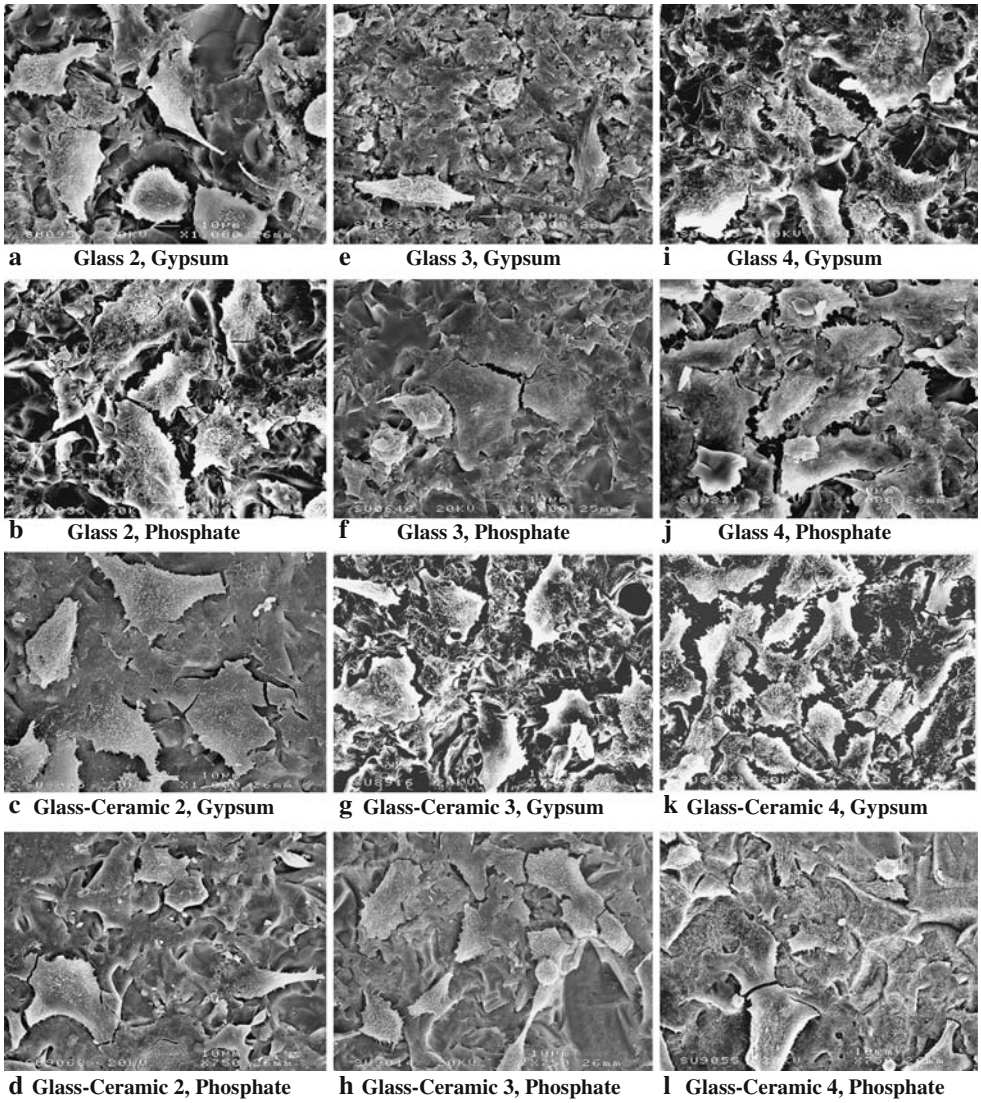

Fig. 8 SEM images of cell cultured casting discs with gypsum and phosphate bonded investments

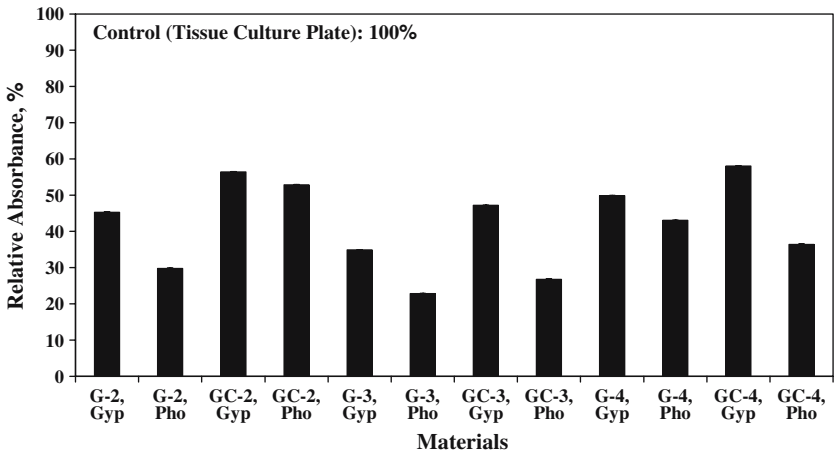

Fig. 9 Histogram showing the MTT assay of casting discs with gypsum (Gyp) and phosphate (Pho) investments which quantifies the biocompatibility for glasses (G) and glass–ceramics (GC)

absorbance than glass–ceramics formed with phosphate bonded investment. Gypsum bonded investments are better investment materials with improved biocompatibility compared to phosphate bonded investments for fluorcanasite glasses under the conditions used in this study. Slightly higher surface roughness for gypsum bonded investment

discs may help the cell attachments and hence the biocompatibility (this could also be an aid when cementing a restoration onto a tooth). Glass–ceramics showed higher absorbance and better biocompatibility than their parent glasses when using both gypsum and phosphate bonded investments.

Conclusions

Fluorcanasite glass–ceramics with modified compositions were produced by controlled two stage heat-treatment of as-cast glasses. These modified fluorcanasite glasses had good relative castability, confirming that they may be useful for the fabrication of custom prostheses via the lost-wax casting. No significant casting shrinkage was observed when glasses were converted to glass–ceramics via a controlled two stage heat-treatment process. The effects of investment material on the cast surfaces were also studied. X-ray diffraction showed similar bulk crystallisation for each glass irrespective of the investment materials. However, some differences in surface crystallisation in the presence of gypsum bonded investment were detected. For both gypsum and phosphate bonded investments, modified fluorcanasite glass–ceramics showed improved in vitro biocompatibility compared with their parent glasses. However, gypsum bonded investment produced glass and glass–ceramic discs showed improved in vitro biocompatibility under the conditions used in this study. Gypsum bonded investments produced slightly higher surface roughness than the phosphate bonded investments which may be considered an advantage for cementation of a dental restoration.

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