

Dental castable glass ceramics: ceramming treatment and colour property

K. WAKASA, A. IKEDA, Y. YOSHIDA, M. YAMAKI

Hiroshima University, School of Dentistry, Department of Dental Materials, Kasumi 1 chome, Minamiku, Hiroshima city, 734 Japan

The magnitudes of colour change vector and lightness were examined for 20CaO/10P₂O₅/10MgO/10Al₂O₃/50SiO₂ glass ceramics (B₂O₃/CaF₂ = 0.01 wt% 0.01 wt%, mass fraction). The glass ceramics were thermally treated (cerammed) for crystallization at each selected temperature (800 to 980 °C for each ceramming period) within a steel ring including the investment mould. Cristobalite-quartz investment mould exhibited no adhesive mould interfaces, showing that the magnitudes of colour change vector and lightness ranged from 17.60 (890 °C) to 37.05 (980 °C) and 55.77 (890 °C) to 71.42 (980 °C) for glass ceramics, respectively, after thermal treatment for 2 h (sample thickness 2 mm). The thermal treatment controlled the colour property measured by colour analyser as compared the cerammed samples with bovine enamel and human tooth enamel.

1. Introduction

Castable glass ceramic is cast into the investment mould using a lost wax process [1, 2]. Adhesive oxides between the glass ceramic and the mould when cast into the phosphate-bonded investment mould affect the interface morphology [3-5], thus the interface is sandblasted mechanically to remove adhesive oxides. After casting or crystallizing, sandblasted samples with aluminum oxide particles have often been applied to the measurement of dimensional change [6, 7]. The appropriate value of mechanical strength as a cast dental crown is controlled by the types of crystals after thermal treatment of the sample [8], because such crystals as hydroxyapatite, diopside (CaO·MgO·2SiO₂) and beta-tricalcium phosphate (beta-3CaO·P₂O₅) occurred at ceramming temperatures of 890-980 °C [9, 10]. Therefore the ceramming condition after casting is important with respect to the removal of investment powder, the value of mechanical strength and formation of crystals. This study has examined the colour change vector and lightness of the glass ceramics after casting and ceramming for dental application to cast crown.

2. Materials and methods

Wax patterns approximating cast dental crowns were invested in a cristobalite-quartz (CQ) investment mould within a steel ring (32 mm diameter and 50 mm height) developed for glass ceramics. The sprue dimension for wax patterns (10 × 10 mm with 1.0-4.2 mm thickness) was of 4.0 mm diameter and 10 mm length (ready casting wax; GC Co., Tokyo). The CQ investment mould was made using a mixture of pH = 8.8 (A and B) (A; pH-adjusted silica sol liquid (pH = 3.5), B; ammonium aqueous solution (pH = 9.4)). The invest-

ment powders were silica powders as alpha-cristobalite (15 μm)/alpha-quartz (5 μm) used in the ratio 55/45. The liquid-to-powder ratio was 0.34 using a liquid mixture of A (16 ml) and B liquids (1 ml). The CQ mould was placed in a burnout furnace heated to 910 °C with a hold time of 30 min (Jelenko Accu-Therm II 500, J. Morita Co, Tokyo) immediately after setting to make the setting expansion 0% (the total expansion of the mould was considered to be the thermal expansion when heated to 910 °C; 2.1%) [11].

The experimental glass ceramic tested was 20CaO/10P₂O₅/10MgO/10Al₂O₃/50SiO₂ as a base composition [8, 12]. Other compounds, such as B₂O₃ and CaF₂, were added to it in the quantity 0.01 wt%. The ceramic was cast into the CQ mould using a vacuum-pressure casting machine (Prototype II, J. Morita Co., Tokyo). The samples were kept within the CQ investment mould which was heated to 500 °C for 2 h, and crystallized at 800 to 980 °C (Prototype I Ceramming Furnace; J. Morita Co., Tokyo). After removing the investment powders on the cast and cerammed surface with an ultrasonic cleanser, mechanically polished samples were used to examine the magnitudes of the colour change vector and lightness (Minolta Colour Analyser CR-121, Tokyo). Light source D65 was used for a measuring area of 3 mm² at CIE - L*a*b* coordinate [13], as indicated in Fig. 1:

$$\text{colour change vector} = [(L^* - L_0)^2 + (a^* - a_0)^2 + (b^* - b_0)^2]^{1/2}$$

where reference sample (MgO) = (L₀, a₀, b₀) = (75.21, 0.31, 0.33) and measuring sample = (L*, a*, b*). Control samples were bovine enamel and human tooth enamel (2 mm thickness).

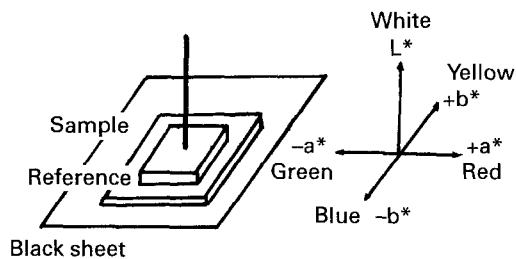


Figure 1 Schematic for colour property measurement and the CIE- $L^*a^*b^*$ coordinate.

As-cast and thermally-treated samples for EPMA observation (electron probe microanalyser; Shimadzu EPMA-8705, Kyoto) were mounted using unfilled resin. Very small amounts of investment powders remained to observe the interface between glass ceramic and mould. The 890 and 980 °C-treated samples were etched chemically with 47% concentrated hydrogen fluoride (HF) solution.

3. Results and discussion

Fig. 2(a) and (b), respectively, show micrographs of as-cast and 800 °C-treated samples with attached investment powders at the interface. All samples were polished mechanically, because initial observations showed that the interface was not smooth. The sample surface was polished with emery papers (#120, 240, 800 and 1500) and finally polished with selenium powder (0.05 µm particle size). Table I indicates the values of colour change vector and lightness of the 2 mm thick as-cast and thermally treated (800 to 980 °C for ceramming period of 2 h) samples. Control samples of bovine enamel and human tooth enamel had, respectively, 31.56 (colour change vector) and 67.82 (lightness) and 34.52 and 64.22. The values of the glass ceramic samples ranged from 17.60 to 37.05 for colour change vector and 55.77 to 71.42 for lightness. Table II indicates the values of colour change vector and lightness of the samples ceramming at 890 °C for 1 to 3 h (sample thickness 2 mm), showing that the colour change vector was reduced at ceramming periods of 2.5 and 3 h. Table III indicates the colour change vector and lightness values of the samples of

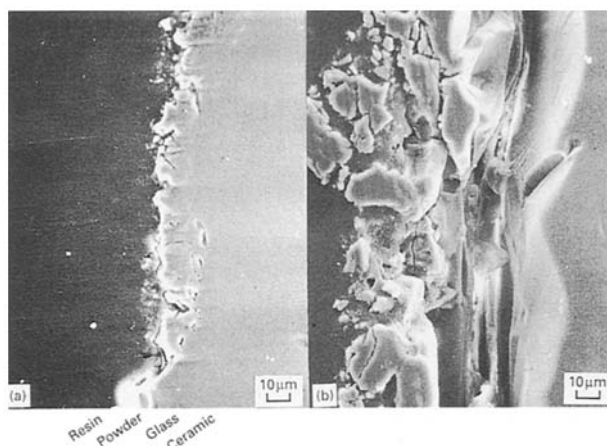


Figure 2 EPMA observation of the samples (a) as-cast and (b) ceramming at 800 °C for 2 h.

TABLE I Colour change vector and lightness of as-cast sample and samples ceramming at 800 to 980 °C for 2 h (10 × 10 × 2 mm). Bovine enamel and human tooth enamel were used as the control samples

Ceramming temperature	Colour change vector	Lightness
As-cast	22.41 (0.14)	65.62 (0.42)
800 °C	22.45 (0.22)	65.04 (0.22)
820 °C	25.05 (0.24)	64.05 (0.21)
845 °C	30.24 (0.31)	64.07 (0.22)
870 °C	30.56 (0.25)	64.07 (0.23)
890 °C	37.05 (0.18)	55.77 (0.20)
980 °C	17.60 (0.22)	71.42 (0.54)
Bovine enamel	31.56 (0.54)	67.82 (0.87)
Human enamel	34.52 (1.25)	64.22 (0.95)

TABLE II Colour change vector and lightness of the ceramming samples (10 × 10 × 2 mm; ceramming condition 890 °C for 1–3 h)

Ceramming period (h)	Colour change vector	Lightness
1	37.02 (0.15)	55.82 (0.20)
1.5	37.00 (0.12)	55.20 (0.14)
2	37.05 (0.18)	55.77 (0.12)
2.5	35.55 (0.25)	55.50 (0.20)
3	33.30 (0.21)	55.55 (0.25)

thickness 1.0 to 4.2 mm when ceramming at 890 °C for 2 h. Colour change vector decreased with reduction in the sample thickness, and lightness increased. Fig. 3 shows EPMA micrographs of the samples ceramming at 890(a) and 980 °C(b) for 2 h using as-cast samples

TABLE III Colour change vector and lightness of samples 10 × 10 mm; with 1.0 to 4.2 mm thickness when ceramming at 890 °C for 2 h

Sample thickness (mm)	Colour change vector	Lightness
1.0	25.01 (0.02)	66.00 (0.04)
1.1	26.10 (0.02)	63.22 (0.12)
1.2	27.50 (0.12)	62.52 (0.09)
1.3	27.86 (0.24)	62.06 (0.40)
1.4	29.60 (0.18)	59.77 (0.12)
2.8	41.66 (0.20)	48.76 (0.04)
4.2	41.15 (0.25)	44.54 (0.02)

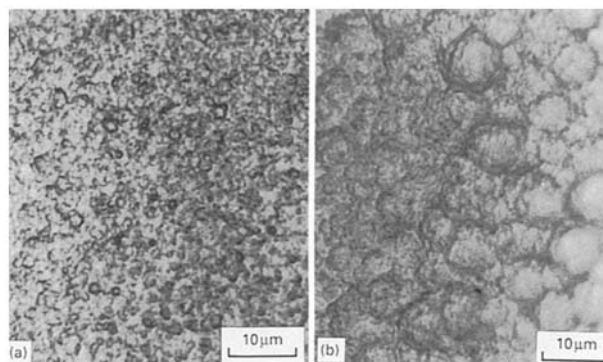


Figure 3 EPMA observation of the ceramming samples etched by HF solution: (a) 890 °C; (b) 980 °C for 2 h.

within the mould. The ceramming of glass ceramics affected the magnitudes of colour change vector and lightness.

Thermal treatment (ceramming) of glass ceramics resulted in different values of colour change vector and lightness. Comparison with bovine enamel and human tooth enamel showed almost the same results as the cerammed glass ceramic sample. The lightness value of bovine enamel and human tooth enamel was 67.82 and 64.22, a value nearly equal to the as-cast amorphous state of the samples. The magnitudes of colour change vector were between the values of cerammed samples which were 30.56 (870 °C) and 37.05 (890 °C). Hydroxyapatite and diopside crystals occurred at 890 °C, although an amorphous state was found for as-cast samples and thermally treated samples (800 and 845 °C) [10]. Also, beta-tricalcium phosphate was formed at 980 °C [10]. The effect of crystal types on colour properties, especially colour change vector, was thus clarified. The results suggest that appropriate ceramming temperatures should be selected for colouring of cast glass ceramic dental crowns.

Colour properties of apatite-based glass ceramics were controlled at each cerammed temperature. The thermal treatment for ceramming was carried out at 800 to 980 °C for each ceramming period within the CQ investment mould. Colour properties were influenced by the formation of the crystals within the glass ceramics. Values approximating to bovine enamel and human tooth enamel were designed for the glass ceramic crown by selection of the appropriate ceramming condition for colouring.

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