Accuracy of Apatite-based Glass Ceramic in Dental Cast Ethyl Silicate-bonded Investment: Theoretical Consideration

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ABSTRACT

Dimensional accuracy of dental apatite-based glass ceramic which was cast into the ethyl silicate-bonded investment mould was examined in relation to their mechanical and thermal properties, and theoretical consideration was given by thermal changes of investment and glass ceramic during ceramming and cooling. Their mixed compositions of ethyl silicate (a bonding agent) and silica particle (a refractory material) were available as the investment mould for dental casting, as reported by calculation model of stress analysis which was proposed for the estimate of strength in dental cast investment. Apatite-based glass ceramic ($20CaO/10P_2O_5/10MgO/10Al_2O_3/50SiO_2$; B_2O_3 and CaF_2 as very small amount) expanded linearly associated with glass softening at test temperature above 800°C. Ceramming treatment of glass ceramic was thus tried at above 800°C within the investment mould. This study showed that the investment mould had enough thermal properties (setting and thermal expansions) to compensate the casting shrinkage of glass ceramic and also enough mechanical strength to cast apatite-based glass ceramic. Theoretical consideration proposed that the accuracy of cast crown and inlay depended on the values of dimensional changes of investment and glass ceramic during ceramming and cooling.

INTRODUCTION

The dental investments employed with gold casting alloys were a hemihydrate of gypsum and a form of silica[1]. The gypsum product served as a binder around the silica refractory material to provide the rigidity and strength of investments. New cast investments have been developed for casting of castable glass cetramics and titanium or titanium alloys[2-8]. The matrix of investments was strengthened by bonding material around the refractory fillers. This was estimated by the analysis of stress and strain relations which were determined by each stress and strain behaviour of the investment composite[9]. Thus, this study was to clarify mechanical and thermal properties of dental ethyl silicate-bonded cristobalite/quartz investment and apatite-based $20CaO/10P_2O_5/10MgO/10Al_2O_3/50SiO_2$ glass ceramic and to consider theoretically the effect of ceramming and cooling behaviours (investment and glass ceramic) within the investment mould on dimensional accuracy of the castings.

MATERIALS AND METHODS

Two kinds of developed dental materials in this study were ethyl silicate-bonded investment and apatite-based glass ceramic. The ethyl silicate-bonded investment had the same compositions as the previous reports, which were composed of ethyl silicate and silica[6-10]. The basic compositions (α cristobalite/α quartz=55/45 (wt%)) of cast investment (Nippon Electric Glass Co, Shiga) were measured by X-ray diffraction analysis (XD-D1, Shimadzu Co, Kyoto). The cristobalite and quartz had respectively 15 and 5 μm as a median particle size (SALD-2000, Shimadzu Co, Kyoto). The sol-gel reaction proceeded between two mixture solutions of $Si(OC_2H_5)_4$ (14.5 mL) and H_20/NH_4OH (aqueous solution; 1.0 mL) when their liquids were mixed together with investment powders. The mixture gel binded the investment powders during heating[10]. The liquid /powder (L/P) ratios used were 0.28, 0.32 and 0.34, whereas one phosphate-bonded

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investment (Ceravest G, GC Co Inc, Tokyo) 0.22, 0.24, 0.26 as a control investment sample (α cristobalite=30 μm; α quartz=10 μm). A control sample had enough higher strength (10 MPa) and greater total expansion value (2.2%) to cast the present apatite-based glass ceramic[12].

The compression strength values of two dental investments (d; diameter of samples=6 mm, h; 12 mm long) were calculated by $4L/\pi d^2$ (L; load at fracture, kgf). The specimen samples were mixed at three L/P ratios for each, and were obtained after 1 day, holding at room temperature (after mixing), after heated to 800°C using the sample held for 1 day and/or at 850°C×30 min after mixing. Setting expansion (sample dimension=10×100×10 mm) was measured with a Travelling microscope (Seiki-shya, Tokyo), as reported already by us[2,3,5]. Thermal analyses (differential thermal analysis (DTA), thermogravimetry (TG) and thermal expansion) were carried out using DT-30 and DT-50 (Shimadzu Co, Kyoto) under the same measuring conditions as our reports[5,9]. Ten samples were used for each measurement.

The casting was first carried out by a standard procedure (mould-fill of molten glass was achieved by pressurization with air and suction through the investment), employing glass pellet (Nippon Electric Glass Co, Shiga), the casting machine (PROTOTYPE II, J. Morita Co, Kyoto) and ceramming furnace (PROTOTYPE I, J. Morita Co, Kyoto), according to each casting and ceramming schedule, as reported by Wakasa et al[4], Nomura[12], Matsui[13] and Nomura et al[14]. The ceramming of as-cast samples was treated by setting it to ceramming furnace (heated until 500°C) after casting, and heated to each temperature (800, 890 and 980°C) for 2 hours.

The dimensional accuracy of glass ceramic was considered theoretically, as described by Phillips[15], Ishida et al[16] and Wakasa and Yamaki[17], which was done by the control of the shrinkage compensation of metal alloys or castable glass ceramics.

RESULTS

Table 1 indicates compression strength of phosphate-bonded investment (a control sample) which was mixed at L/P ratios of 0.22, 0.24 and 0.26 for two holding conditions of specimen samples. After 1 day and after heated to 800°C after 1 day' holding, the values were measured. The heated samples had lower values than those obtained after 1 day. Table 2 and 3 indicate compressive strength and setting expansion of ethyl silicate-bonded investment at L/P ratio=0.28, 0.31 and 0.34: After 1 day, after heated to 800°C after 1 day' holding and at 850°C×30 min for compression test (three conditions of samples) and at 15 and 60 min after mixing for setting expansion (two measuring conditions). The strength values ranged from 1.1 to 2.2 MPa, and setting at 60 min after mixing had shrinkage (minus; −0.40 to −0.62%).

Ceramming of apatite-based glass ceramic was done

Table 1 Compressive strength of phosphate-bonded investment at L/P ratio=0.22, 0.24, 0.26 for two conditions, after 1 day and after 1 day and heated to 800°C. Mean value (standard deviation).

L/P	Compressive strength (MPa)	
	1 day	1 day→Heated to 800°C
0.22	9.7 (1.2)	6.4 (1.3)
0.24	9.5 (1.7)	5.5 (1.6)
0.26	8.0 (1.0)	5.4 (1.0)

Table 2 Compressive strength of ethyl silicate-bonded investment at L/P ratio=0.28, 0.31 and 0.34. After 1 day, after 1 day and heated to 800°C and 850°C×30 min. Mean value (standard deviation).

L/P	Compressive strength (MPa)		
	1 day	1 day→Heated to 800°C	850°C×30 min
0.28	1.1 (0.1)	2.2 (0.6)	1.3 (0.2)
0.31	2.1 (1.0)	1.6 (0.3)	1.7 (0.4)
0.34	2.2 (0.4)	1.4 (0.2)	1.8 (0.4)

Table 3 Setting expansion after mixing at 15 and 60 min. L/P ratio=0.28, 0.31, 0.34.

L/P	Setting expansion (%)	
	After mixing	
	15 min	60 min
0.28	0.15	−0.41
0.31	−0.01	−0.62
0.34	0.04	−0.40

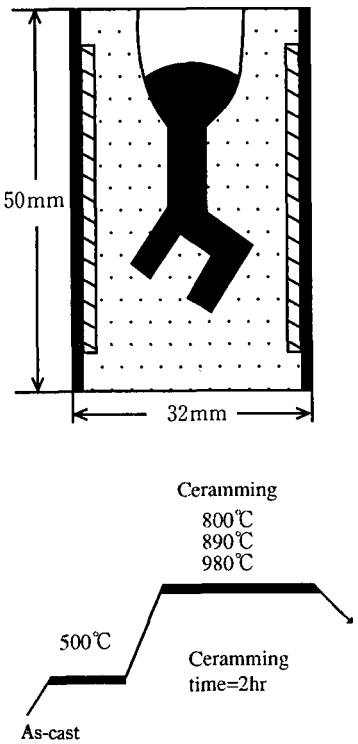

Figure 1 Ceramming procedure of as-cast glass sample within the investment mould.

within the investment mould after casting, according to the temperature schedule (Fig. 1). The as-cast samples were kept within the mould, and heated to 500°C and heated to 800, 890 and 980°C for 2 hours, because glass ceramic had different crystallized structures, hydroxyapatite (HA), diopside (D) and β-tricalcium phosphate (β-TCP)[4,10,12]. The investment powder tested had a mixture of α cristobalite and α quartz, and the gel had also silica because of ethyl silicate bonding sol, whereas hemihydrate showed different position between them (c; α cristobalite, q; α quartz, o; disappeared, weak peak at 1 day after mixing) (Fig. 2). The mixed powders (sol= ethyl silicate) had the same DTA and TG behaviours as investment powder with the increased test temperatures from room temperature to 900°C (Fig. 3). The existance of silica was detected at two test temperatures of T_c (α-β cristobalite transformation) and T_q (α-β quartz transformation). Their transformation phenomenon had no weight loss of investment during a heat to 900°C.

Thermal behaviours of ethyl silicate-bonded investment and apatite-based glass ceramic were respectively indicated in Figs. 4 and 5. Thermal expansion had the increased values (above 2.0%) with two steps of α-β cristobalite (T_c) and α-β quartz transformation (T_q) during

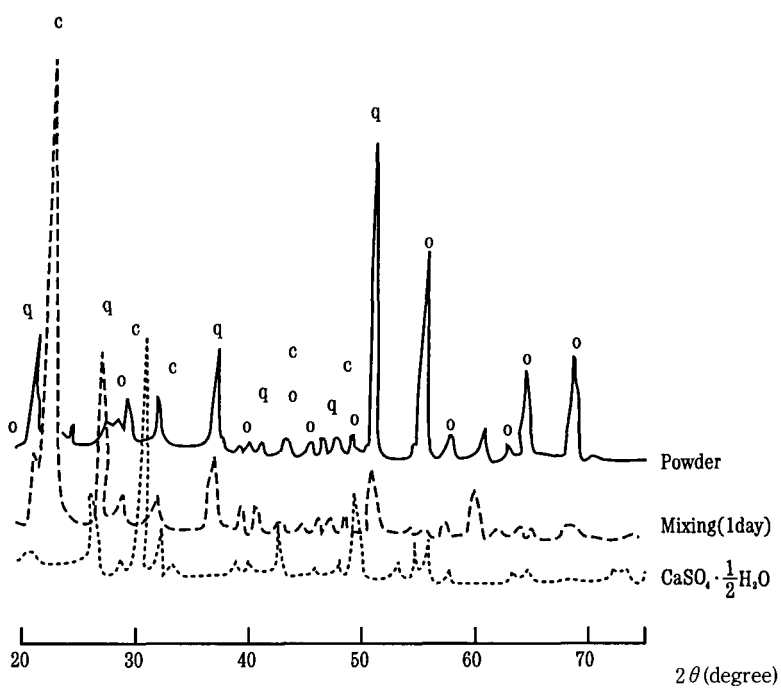

Figure 2 X-ray diffraction patterns of investment powders, mixed investment powders after 1 day, and hemihydrate of gypsum. For key, see text.

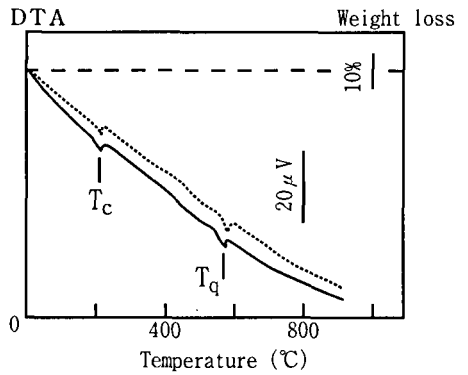

Figure 3 DTA and TG charts of investment powder (—) and mixed investment after 1 day (·····): T_c, temperature of α-β cristobalite transformation, T_q, temperature of α-β quartz transformation. Weight loss is described by (----).

a heat to 850°C (Fig. 4). Thermal expansion of as-cast glass ceramic and cerammed glass ceramics (800, 890 and 980°C×2 hours) increased linearly until 800°C and then softened (Fig. 5). From Fig. 5, thermal expansion coefficient was calculated (a range of 40 to 90×10^{-7} (1/deg)), showing the same value as DIKOR mica glass ceramic (Fig. 6). This result showed that heating rate of glass ceramic affected ceramming preocedures.

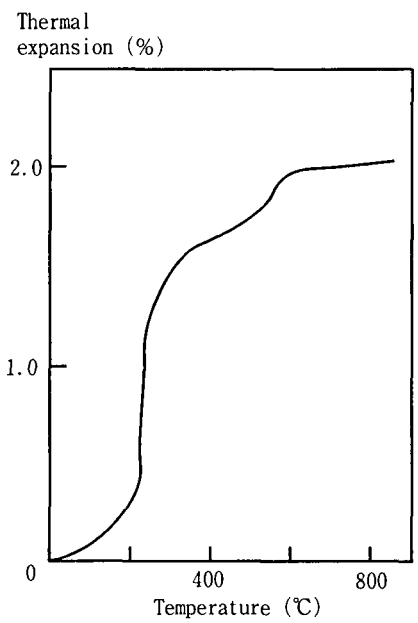

Figure 4 Thermal expansion with α-β transformation of cristobalite and quartz in investment powder.

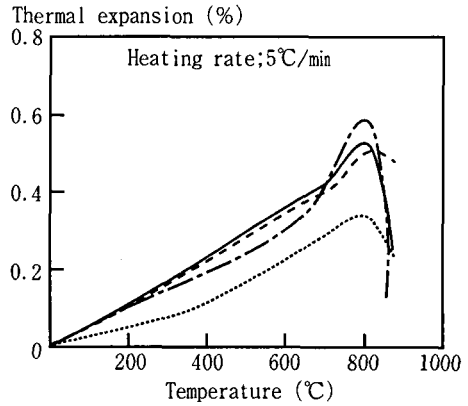

Figure 5 Thermal expansion of cerammed conditions at heating rate of 5°C/min.:
—— As-cast
—·— 800°C×2 hours
------ 890°C×2 hours
····· 980°C×2 hours

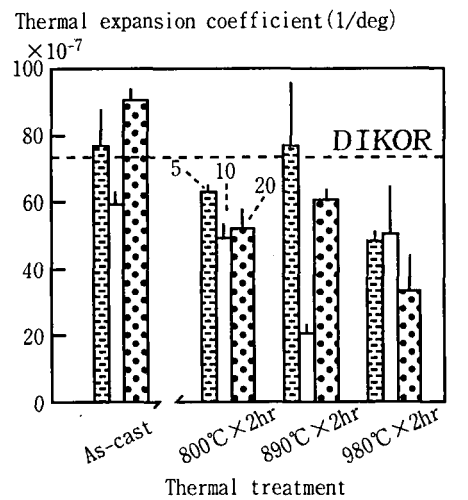

Figure 6 Thermal expansion coefficients of cerammed glass samples: 5, 10 and 20 mean heating rate of specimen samples. DIKOR means Dikor glass sample.

DISCUSSION

The dental cast investments for titanium and castable glass ceramics has obtained popularity because of the simple procedures involved, but it is still developed for the construction of high fusing base metal alloys and the ceramics. These types of investment were ethyl silicate-bonded investment (α cristobalite and α quartz)[18,19], MgO-included investment[20,21] and ethyl silicate-bonded

investment including MgO and phosphate-bonded investment with great amount of porosity in the mould[12,22,23].

In these types of cast investment, two kinds of binders were a silica gel (colloidal silica) and ethyl silicate. The former reverted to silica on heating. A bonding silicic acid gel formed when an acid or an acid salt was added to it. The latter binder was formed by the hydrolysis of ethyl silicate in the presence of hydrochloric acid, ethyl alcohol and distilled water[2].

In this study, ethyl silicate-bonded investment was composed of mixture solution (ethyl silicate) and a catalyst (ammonium carbonate), and thus a colloidal sol of polysilicic acid was produced as a binder. The strength of cast investment had an appropriate strength (about 2 MPa), similar to gypsum-bonded investment[1]. The total expansion values were considered as follows. After mixing of investment, the holding time was 10 min, because setting time was 10 min with setting expansion=0%. This type of investment had minus value of setting expansion (a shrinkage) (Table 3). Thus, total expansion depended on only thermal expansion value (2.1%), as shown in Fig. 4. As a standard casting procedure, the investment mould including as-cast glass sample after casting was set to the furnace, according to ceramming schedule, immediately after setting.

The dimensional change (%), D, of castable glass ceramic is considered as follows:

Total expansion (investment)	$=2.1\%$
Thermal change (wax pattern) for crown	$=0\%$
or for inlay	$=-0.2\%$
Shrinkage of glass ceramic	$=-S\%$
Change of investment during ceramming and cooling	$=I_c\%$
Change of glass ceramic during ceramming and cooling	$=G_c\%$

Thus, dimensional change D was given as

$D=2.1-S+(I_c+G_c)$ for crown

$D=1.9-S+(I_c+G_c)$ for inlay

Three cases were considered theoretically as $D=0$ (no clearance between the casting and investment mould), $D>0$ (positive value for cast crown) and $D<0$ (negative value for cast simple inlay).

$D>0,\ 2.1>S-(I_c+G_c)$ for crown

$D<0,\ 1.9<S-(I_c+G_c)$ for inlay

Thus, the following consideration was given for the cast crown (10 mm diameter) and the cast inlay (3 mm diameter), and the ranges were calculated when the clearance values were 5 to 30 μm:

$+0.1<D<+0.6$ for crown

$-0.3<D<-2.0$ for inlay

Thus, the cast crown had a range of dimensional change as

$S-2.0<I_c+G_c<S-1.5$ for crown

and the cast inlay was such a range of

$S-2.2<I_c+G_c<S-3.9$ for inlay

In this study, apatite-based glass ceramic had a shrinkage value when the thermal expansion coefficient consisted linearly by melting temperature (1500°C)[13] (heating rate of ceramming furnace=5°C/min),

$-S=-0.9\%$

For the cast crown and cast Inlay, respectively, the following equations were given at $D=+0.4\%$ and -1.1% as intermediate values,

$I_c+G_c=-0.8$ for crown

$I_c+G_c=-2.1$ for inlay

From these results, we should consider the thermal changes of investment and glass ceramic within the mould, and thus the cast glass crown has lower thermal change than does the cast glass inlay. Thus, only the nature of cast investment should be considered when the same apatite-based glass ceramic is applied to cast crown or cast inlay. This result suggests that the greater amount of thermal change (+1.3%) is needed for the cast inlay and thus MgO-included ethyl silicate-bonded investment with larger expansion is available effectively to cast the glass ceramic inlay.

This type investment was designed to make silica gel around the silica particles, which were composed of α cristobalite and α quartz with different median sizes to reduce inhomogeneous layer of silica gel. The amount of binder was low because of low L/P ratios=0.28 to 0.34, and was essentially silica which converted to quartz during heating. This investment type is applied to casting of the higher fusing metal alloys, as reported by Wakasa and Yamaki[20,21]. In castable glass ceramic crown, greater total expansion (above 2.0%) compensates the shrinkage value of glass ceramic. In additional consideration, the dimensional change of apatite-based glass ceramic which is cerammed and cooled within the investment mould should be evaluated by their thermal changes (investment and glass ceramic) during thermal treatment.

SUMMARY

It is summarized that ethyl silicate-bonded investment composed of ethyl silicate (bonding agent) and silica filler

(refractory material) had appropriate expansion properties to the shrinkage of apatite-based glass ceramic. Dental glass ceramic was applied into ethyl silicate-bonded investment moulds with appropriate expansion values for cast shrinkage of glass ceramic which was thermally treated (ceramming) at higher test temperatures (800 to 980°C) for 2 hours within the mould. The dimensional accuracy of apatite-based glass ceramic castings (crown and inlay) was theoretically considered by the dimensional changes of investment and glass ceramic during ceramming and cooling.

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