# Surface Roughness Value in Cast and Cerammed Apatite-based Glass Ceramic

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#### ABSTRACT

Surface roughness was examined on as-cast and cerammed apatite-based glass ceramics ( $20\text{CaO}/10P_2O_5/10\text{MgO}/10\text{Al}_2O_3/50\text{SiO}_2$ ) when cast into phosphate-bonded (P) and ethyl silicate-bonded cristobalite-quartz investment (CQ) moulds. After removing the investment powders on the interface between mould and glass ceramic the glass ceramic surface was evaluated by surface roughness value. For as-cast and cerammed samples, surface roughness value in P mould group was significantly larger than that in CQ mould group. There was no significance of surface roughness value for P (15.2 to 17.3 micron) or CQ mould group (5.2 to 6.8 micron). CQ investment developed using ethyl silicate solution was available for casting glass ceramic.

## INTRODUCTION

Castable glass ceramic was cast into the investment mould using a lost wax process<sup>1,2)</sup>. Setting and thermal properties of dental investments were controlled, improving the mixing liquid to phosphate-bonded investment powder (silica, magnesia and phosphate)<sup>3–5)</sup>. Addition of BN (boron nitride) to the powders gave no adhering oxide layer on the cast surface of nickel-based alloys, showing lower maximum surface roughness values of 3.1 to 4.1 micron<sup>6)</sup>. The magnesium ammonium phosphate

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(polymeric) formed when mixed by  $\mathrm{H_2O}$  in the presence of mono-ammonium phosphate ( $\mathrm{NH_4H_2PO_4}$ ) and magnesia ( $\mathrm{MgO}$ )<sup>7-9)</sup>. After casting or crystallizing, the sand-blasted sample with aluminum oxide particles was conventionally used in measuring linear dimensional change<sup>10,11)</sup>. Setting with temperature increase occurred in cristobalite-quartz mould and mechanical strength of the mould was enough to cast glass ceramic sample<sup>12)</sup>.

This study was to examine surface roughness of cast and crystallized (cerammed) apatite-based glass ceramics when cast into two types of investment mould. And also the cast interface was observed by electron probe microanalyzer (EPMA) after removing investment powders by ultrasonic cleanser in distilled water.

#### MATERIALS AND METHODS

Two types of investments tested were conventional phosphate-bonded investment (P) and ethyl silicatebonded cristobalite-quartz investment (CQ) as the mould of cast and cerammed glass ceramics. Wax pattern for cast sample  $(10\times10\times2 \text{ mm})$  was invested in P and CQ investment moulds within a steel ring of 32 mm diameter and 50 mm height (sample size=10). The former was mixed with an aqueous colloidal silica suspension (Shofu Inc. Kyoto) and the latter with special liquid (A and B) (A liquid (16 mL), pH-adjusted silica sol; B liquid (1 mL), ammonium aqueous solution). P mould consisted of refractory fillers and a binder, and the filler is silica powders in the form of alpha cristobalite (particle median size; 30 micron) and alpha quartz (10 micron). The binder consisted of magnesia and a phosphate in P mould. CQ mould had alpha cristobalite (15 micron) and alpha quartz (5 micron), and the binder was silica gel which reverted to silica on heating. The liquid-to-powder ratio was 0.32 for the former and 0.34 for the latter. P mould was heated from room temperature to 700°C after hardening of 60 min. CQ mould was set to burn-out furnace heated to  $910^{\circ}\text{C}$  with a hold time of  $30\,\text{min}$  immediately after hardening. The setting expansion was zero and thermal expansion was 2.1% (CQ mould)<sup>13)</sup>.

Apatite-based glass ceramic was experimental  $20\text{CaO}/10\text{P}_2\text{O}_5/10\text{MgO}/10\text{Al}_2\text{O}_3/50\text{SiO}_2$  one, which included  $\text{B}_2\text{O}_3$  and  $\text{CaF}_2$  by 0.01 wt% $^{12,14)}$ . The ceramic was cast into P and CQ moulds using vacuum-pressure casting machine (PROTOTYPE II, J. Morita Co, Kyoto). The sprue dimension was 4.0 mm diameter and 10.0 mm

length (ready casting wax, GC Co, Tokyo).

As-cast and cerammed samples were observed using electron prohe microanalyzer (EPMA-8705, Shimadzu Co, Kyoto). The maximum surface roughness (Surface roughness analyzer, Kosaka Lab, Osaka) was measured on the surface of the cast and cerammed samples. The investment on the surface was removed by ultrasonic cleaning in distilled water in measuring the surface roughness value.

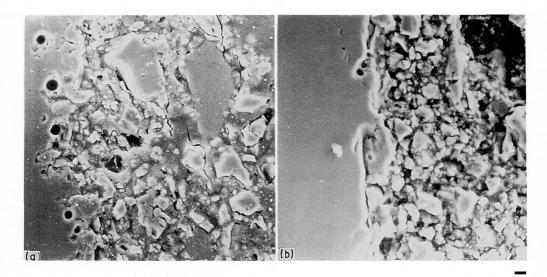


Fig. 1 The samples after casting into P mould. (a) As-cast and (b) cerammed samples at  $800^{\circ}$ C for 2 hr. Scale bar=20 micron.

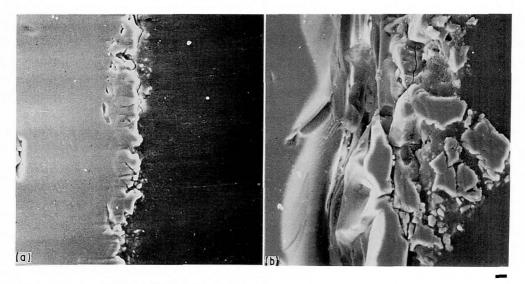


Fig. 2 The samples when cast into CQ mould. (a) As-cast and (b) thermally-heated sample at  $800^{\circ}$ C (2 hr). Scale bar=20 micron.

#### RESULTS

After casting, the mould was cooled to room temperature and set to the temperature-controlled furnace (kept at 500°C for 2 hr and heated to selected temperatures 800, 890 and 980°C for 2 hr). Fig. 1(a) and (b) show typical micrographs of as-cast and thermally-heated (cerammed at 800°C) samples when cast into P mould. After casting or ceramming the investment powders attached on the cast surface. Fig. 2(a) and (b) (CQ mould) show as-cast and cerammed samples, exhibiting that the investment layer was found clearly. In this study surface roughness was examined to clarify the surface characteristics between P and CQ moulds, as indicated in Table 1. The maximum roughness values in CQ mould (5.2 to 6.8 micron) was smaller than that in P mould (15.2 to 17.3 micron). There appeared a significance of surface roughness value between P and CQ moulds for as-cast or thermally-treated samples (p<0.05). No significant difference was found for the roughness values in P or CQ mould (p>0.05).

Table 1 Maximum roughness values on apatitebased glass ceramic surface after casting and ceramming. The samples were cleaned by ultrasonic cleaning. Sample size = 10.

Thermal treatment	Maximum roughness (micron)	
	P mould	CQ mould
As-cast	17.3 (0.9)	5.2 (1.1)
800°C	16.9 (2.1)	5.5 (0.7)
Cerammed 890°C	15.2 (2.4)	6.8 (1.5)
980°C	16.1 (3.7)	6.4 (2.0)

Ceramming time=2 hr.

# DISCUSSION

Apatite-based glass ceramic was developed<sup>12)</sup>, and it was cast into dental investment moulds. After ammonium carhonate aqueous solution was added to each silica sol, CQ mould hardened associated with the start of sol-gel reaction (pH=8.0 to 8.5)<sup>13)</sup>. As apatite-based glass ceramic was 1160°C as the melting temperature<sup>12)</sup>, it is needed that adhering oxides form little on the cast surface. The cast sample showed a crack formation from the surface to the interior when sandblasted with aluminum particles<sup>15)</sup>. The investment including no magnesia and phosphate was thus needed as an investment mould for casting and ceramming of this apatite-based glass

ceramic, because the adhering layer formed associated with the formation of magnesium ammonium phosphate compound at the interface between mould and glass ceramic in P mould.

In this study heat treatment for ceramming was done using the same as that after casting into CQ mould within the steel ring. The ceramming of Dicor ceramic crown was processed again by embedding in a special gypsumbonded investment mould (the chemical constituents were not described; Dicor, Dentsply Int, York, Pa)10). The present apatite-based glass ceramic included both apatite, diopside and beta-tricalcium phosphate in ceramming selected temperature<sup>13)</sup>. As-cast and cerammed surface of the cast samples into P mould had larger maximum surface roughness value than those in CQ mould (Table 1). The surface roughness value was affected by particle size of investment powders. CQ mould was available to cast apatite-based glass ceramic, because more smooth surface in CQ mould than P mould formed after casting and ceramming. The investment mould will be discussed in near future using various sizes of alpha cristobalite and/or alpha quartz to develop it for castable glass ceramics.

## **SUMMARY**

As-cast and cerammed surface roughness of apatite-based 20CaO/10P<sub>2</sub>O<sub>5</sub>/10MgO/10Al<sub>2</sub>O<sub>3</sub>/50SiO<sub>2</sub> samples was examined using surface roughness tester, because the interface between investment mould and glass ceramic was affected by refractory fillers in the form of alpha cristobalite and alpha quartz within the investment mould. In CQ mould with no magnesia and phosphate maximum surface roughness was 5.2 to 6.8 micron, whereas P mould had 15.2 to 17.3 micron for the samples tested. It is expected that smooth surface might be obtained by smaller sizes of alpha cristobalite and/or alpha quartz within the mould when used for glass ceramic.

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