

Mould Interface Observation of Castable Ceramic Crowns in Casting and Ceramming

Kunio Wakasa and Masao Yamaki

(Received for publication, September 30, 1993)

ABSTRACT

Apatite-based glass ceramics were cast and cerammed within both phosphate-bonded (P) and cristobalite-quartz (CQ) investments. The former was used after setting (60 min) and heated from room temperature to 700°C (kept for 30 min), and the latter was set to furnace (910°C for 30 min) immediately after setting of the investment mould (setting time; 22 min). The mould interface was disclosed by electron probe microanalyzer (EPMA) observation, because apatite-based glass ceramic was cast and subjected to heat treatment for crystallization at each selected temperature (890 and 980°C for 2 hr) within a steel ring including the mould. Both moulds affected mould interface morphology after casting and crystallization, showing that there occurred a smooth surface without a porosity under EPMA observation of the glass ceramic and CQ mould interface.

INTRODUCTION

Castable glass ceramic was cast into the investment mould using a lost wax process^{1,2)}. Setting and thermal properties of dental investments were controlled, improving the mixing liquid to phosphate-bonded investment powder (silica, magnesia and phosphate)^{3,4)}. 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 μm ⁵⁾. The magnesium ammonium phosphate (poly-

meric) formed when mixed by H₂O in the presence of mono-ammonium phosphate (NH₄H₂PO₄) and magnesia (MgO)⁶⁻⁸⁾. After casting or crystallizing, the sand-blasted sample with aluminum oxide particles was conventionally used for cast crown^{9,10)}. Setting with temperature increase occurred in CQ mould¹¹⁻¹³⁾ and mechanical strength of the mould was enough to cast glass ceramic sample¹⁴⁾. This study was to examine the mould interface after casting or crystallizing (ceramming) of apatite-based glass ceramics.

MATERIALS AND METHODS

Wax patterns for ceramic crowns were invested in P and CQ investment moulds within a steel ring (32 mm diameter and 50 mm height). Two types of investments tested were conventional phosphate-bonded investment (P) and newly developed cristobalite-quartz investment (CQ) as the mould of cast and cerammed glass ceramics. 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 μm) and alpha quartz (10 μm). The binder consisted of magnesia and a phosphate in P mould. CQ mould had alpha cristobalite (15 μm) and alpha quartz (5 μm), 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°C with a hold time of 30 min immediately after hardening. The setting expansion was zero and thermal expansion was 2.1% (CQ mould)¹¹⁾. Apatite-based glass ceramic was experimental 20CaO/10P₂O₅/10MgO/

Department of Dental Materials, Hiroshima University School of Dentistry (Chairman: Professor Masao Yamaki).

Correspondence to Dr K. Wakasa, Hiroshima University, School of Dentistry Department of Dental Materials, Kasumi 1 chome, Minamiku, Hiroshima City, 734 Japan

$10\text{Al}_2\text{O}_3/50\text{SiO}_2$ one, which included B_2O_3 and CaF_2 by a very small amount for crystallization^{14,15}. The ceramic was cast into P and CQ moulds using vacuum — pressure casting machine (PROTOTYPE II, J. Morita Co, Kyoto).

A stainless steel die for wax pattern construction (9.89 mm, the lower side; 8.90 mm, the upper side and 9.92 mm, height) approximating crown preparation (maximum thickness=2.0 mm at the upper surface) was used¹⁶.

The sprue dimension was 4.0 mm diameter and 10.0 mm length (ready casting wax, GC Co, Tokyo). After remaining the investment powders on the surface, the sample including an interface between mould and ceramic was embedded in unfilled resin to observe using electron probe microanalyzer (EPMA-8705, Shimadzu Co, Kyoto).

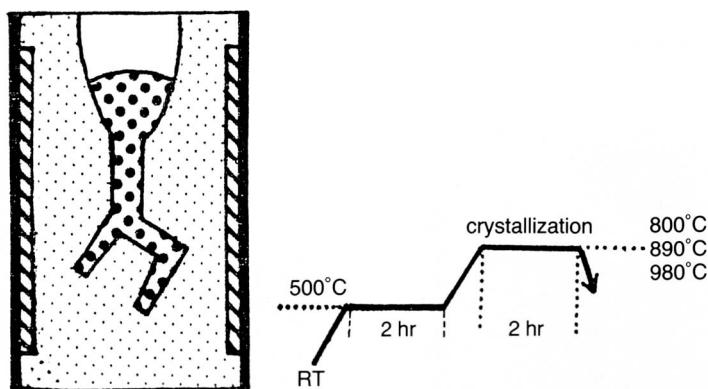


Fig. 1 Schematic figure within the investment mould during heat treatment (ceramming) and ceramming procedure.

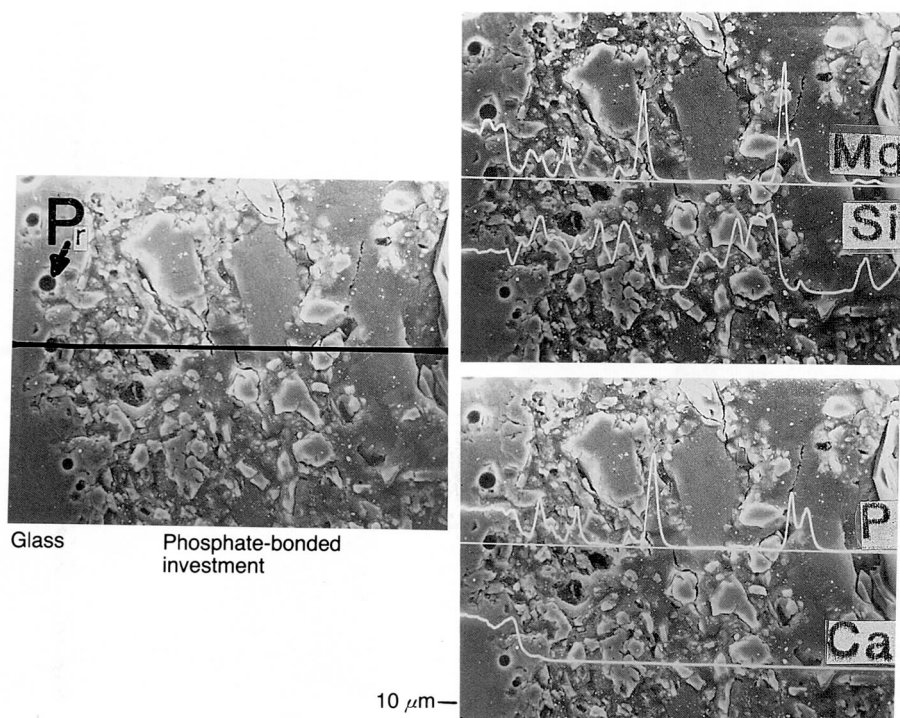


Fig. 2 As-cast sample after casting into P mould. Pr means large-scaled porosity within the castings, indicating Mg, Si, P and Ca line analysis. See text for key.

RESULTS

Fig. 1 shows schematic figure and ceramming procedure for heat treatment of castable glass ceramic within the mould. 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). Heat treatment was done within the mould (Fig. 1), because the softening of glass ceramics was observed around 700 to 770°C¹⁴). Figs. 2 to 7 show as-cast and thermally-heated

samples when cast into P and CQ mould, and represent line analysis (Mg, Si, P, Ca) and each mapping under EPMA observation. The investment powders on the cast glass ceramic surface were remained without eliminating them to observe the investment mould and glass ceramic interface. In Figs. 2 to 4 (P mould) both small and very large sizes of porosity (Pr, porosity) was observed near the surface, showing that P mould had less permeability than CQ mould. In Fig. 5 (980°C for 2 hr, P mould) crack was observed at the depth of 30 µm along the glass ceramic surface, and Mg, Si and P distribution

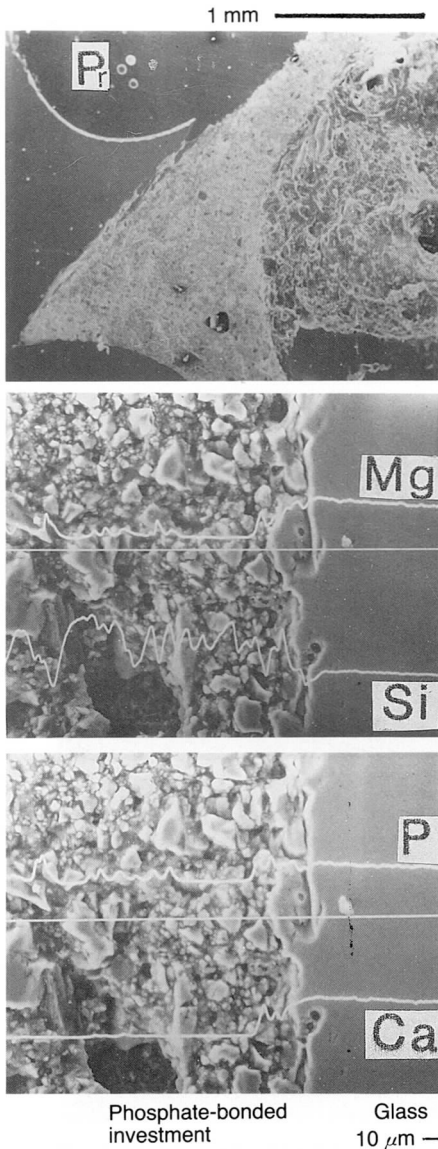


Fig. 3 Cerammed sample at 800°C for 2 hr (P mould).

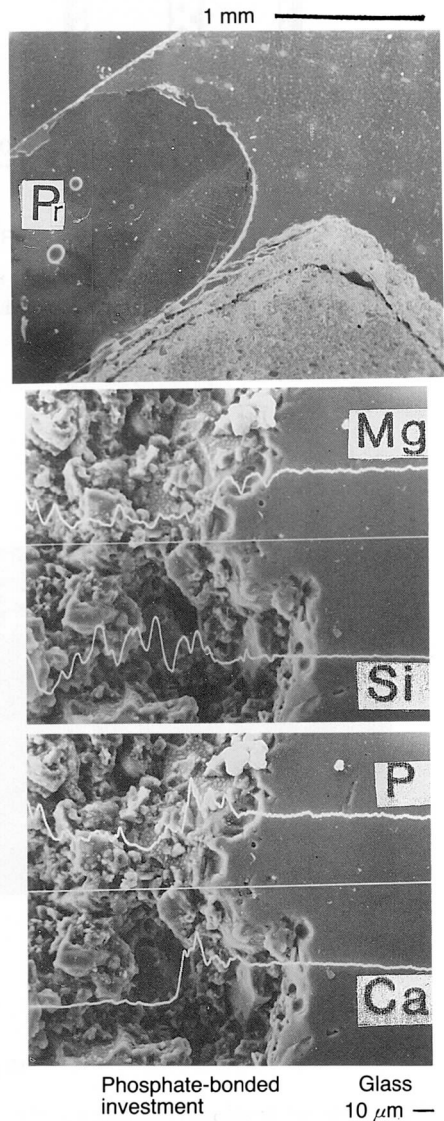


Fig. 4 Cerammed sample within P mould (890°C).

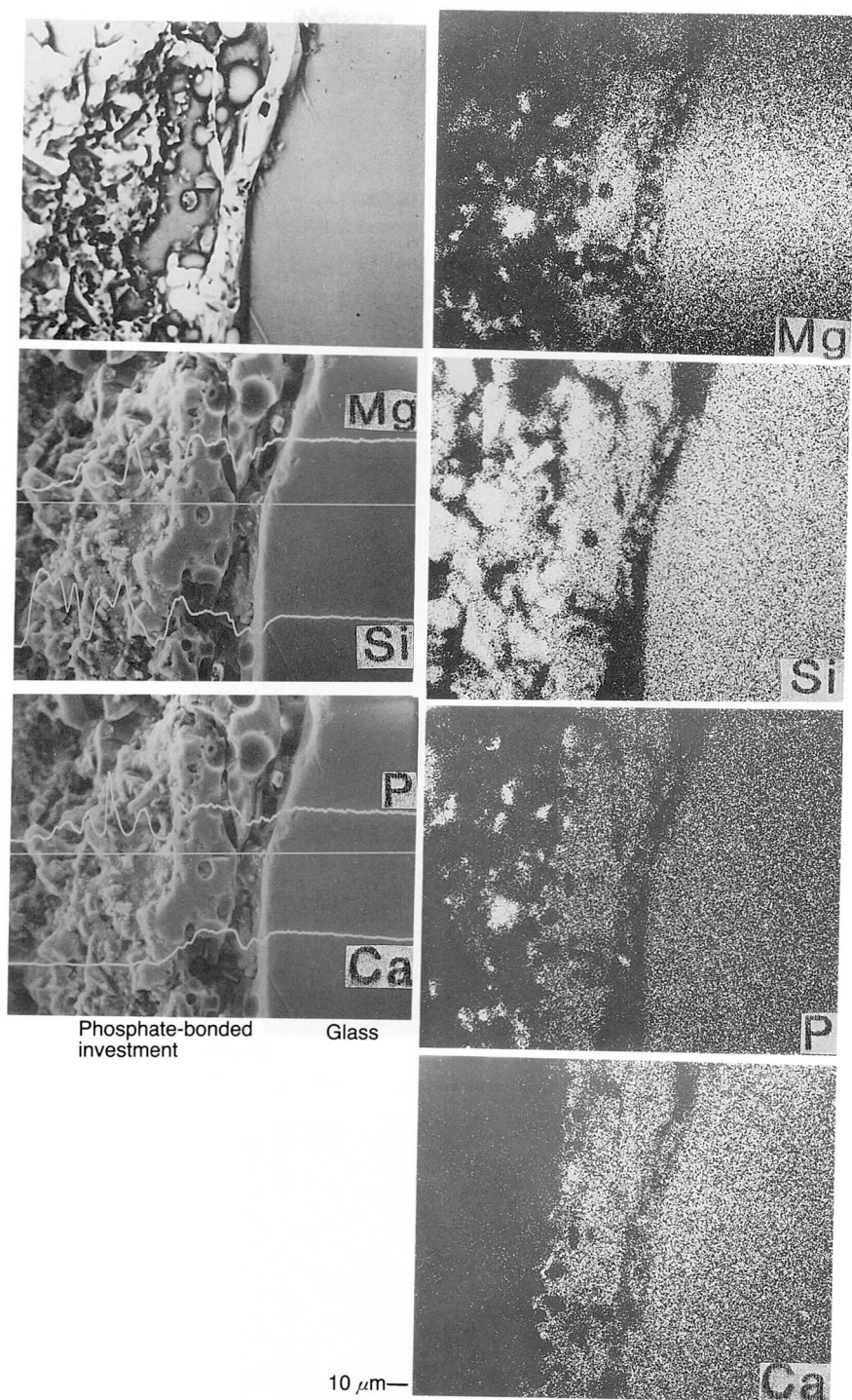


Fig. 5 980°C for 2 hr (P mould).

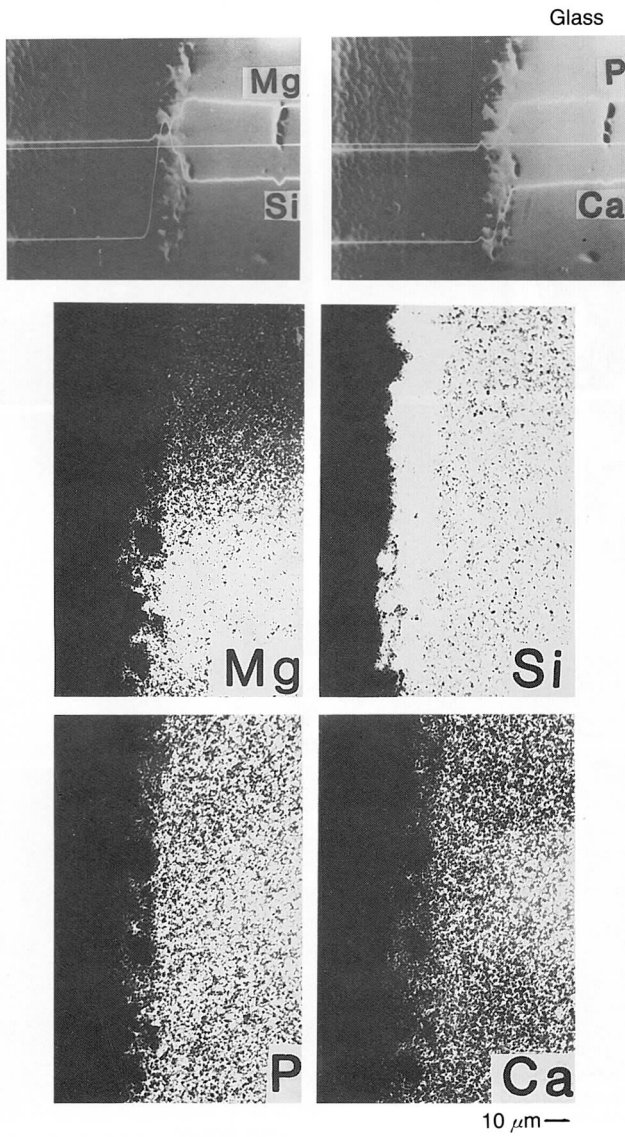


Fig. 6 As-cast samples when cast into CQ mould.

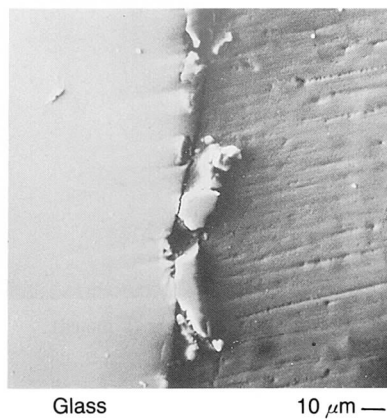


Fig. 7 Thermally-heated sample at 890°C (2 hr) within CQ mould.

was found within the mould and castings, representing that higher heat treatment of as-cast glass ceramic forms the crack as compared with that at 890°C. In Figs. 6 and 7 (CQ mould) as-cast and thermally-heated samples (890°C) had smooth surface and a few investment powders attached to the surface after ultrasonic cleaning (Fig. 7). The interface between glass ceramic and mould was disclosed, showing that cast surface in CQ mould seems to be more smooth than in P mould (Figs. 2 to 7).

DISCUSSION

Since new apatite-based glass ceramic was developed¹⁴, it was cast into dental investment moulds. After ammonium carbonate 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)¹¹. As apatite-based glass ceramic was 1160°C as the melting temperature¹⁵, 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 particles¹⁷. Each mapping of Mg, Si and phosphate suggests that there appeared an adhering oxide along the cast glass ceramic surface (Fig. 5, P mould). The investment including no magnesia and phosphate was thus needed as an investment mould for casting and ceramming of this apatite-based glass ceramic.

In this study heat treatment for ceramming was done using the same as that after casting into CQ mould within the steel ring (Fig. 1). The ceramming of Dicor ceramic crown was processed again by embedding in a special gypsum-bonded investment mould (the chemical constituents were not described; Dicor, Dentsply Int, York, Pa)⁹. The present apatite-based glass ceramic included both apatite, diopside and beta-tricalcium phosphate in ceramming selected temperature¹¹.

P mould had a disadvantage that investment powders adhered to cast surface and less permeability occurred as compared with CQ mould (Figs. 2 to 7). Heat treatment at higher temperature (980°C) exhibited the layer with heavy distribution of Mg, Si and phosphate element which corresponded to the adhering layer. As apatite-based glass ceramic included Mg, P and Si, their elements diffused easily from P mould toward glass ceramic. The result exhibits that a layer along the ceramic surface with different distribution of the elements (Mg, Si, phosphate) forms at inside of the castings. The present CaO/P₂O₅/MgO/Al₂O₃/SiO₂ ceramic was cast into CQ mould and

also cerammed within the same as cast investment mould. This was different from the Dicor castable ceramic system⁹.

SUMMARY

The as-cast and cerammed surface of the cast samples into P and CQ moulds was examined by EPMA observation, representing that the interface between mould and glass ceramic was affected by particle sizes of refractory fillers in the form of alpha cristobalite and alpha quartz. CQ mould with no magnesia and phosphate was available to cast apatite-based glass ceramic, illustrating that 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.

REFERENCES

- 1) Adair, P.J. and Grossman, D.G.: The castable ceramic crown. *Int. J. Periodontol. Restor. Dent.* 2, 33-45, 1984.
- 2) Kihara, S. and Watanabe, A.: Calcium phosphate glass-ceramic crown prepared by lost-wax technique. *J. Amer. Ceram. Soc.* 67, C-100-101, 1984.
- 3) Finger, W. and Jørgensen, K.D.: An improved dental casting investment. *Scand. J. Dent. Res.* 88, 278-284, 1980.
- 4) Finger, W. and Kota, K.: A modified phosphate-bonded casting investment. *Scand. J. Dent. Res.* 90, 243-248, 1982.
- 5) Wakasa, K. and Yamaki, M.: A modified dental cast investment for nickel base alloy: a preliminary study. *J. Mater. Sci. Letters* 10, 1093-1094, 1991.
- 6) Mori, M., Yanagihara, T., Asai, T., Itoh, S. and Murai, M.: Studied on investment with carbon. Part 2. Mould temperature and adherent substance of castings surface. *Aichi Gakuin Daigaku Shigaku Zasshi* 15, 298-305, 1977.
- 7) Mori, T.: The alpha- and beta-forms of calcium sulphate hemihydrate. *J. Dent. Res.* 65, Abst. No. 38, 1986.
- 8) Mori, T.: Thermal behavior of the gypsum binder in dental casting investments. *J. Dent. Res.* 65, 877-884, 1986.
- 9) Holmes, J.R., Sulik, W.D., Holland, G.A. and Bayne, S.C.: Marginal fit of castable ceramic crowns. *J. Prosthet. Dent.* 67, 594-599, 1992.
- 10) Ishida, H., Nahara, Y. and Hamada, T.: Dimensional accuracy of castable apatite ceramic crowns: The influence of heat treatment on dimensional changes and distortion of crowns. *J. Prosthet.*

- Dent.* 68, 279–283, 1992.
- 11) Wakasa, K. and Yamaki, M.: Thermal behaviour of casting investment during setting. *J. Oral Rehabil.* in press, 1993.
 - 12) Marsaw, F.A., de Rijk, W.G., Hesby, R.A., Hinman, R.W. and Pelleu, G.B.: Internal volumetric expansion of casting investments. *J. Prosthet. Dent.* 52, 361–336, 1984.
 - 13) Stevens, L., Okamoto, A. and Jørgensen, K.D.: Dimensional change in mould space on setting of phosphate bonded investment. *Aust. Dent. J.* 30, 281–284, 1985.
 - 14) Wakasa, K., Yamaki, M. and Matsui, A.: Thermal treatment and crystalline structure in dental cast ceramic materials. *J. Mater. Sci. Mater. Med.* 3, 235–238, 1992.
 - 15) Wakasa, K., Yamaki, M. and Matsui, A.: An experimental study of dental ceramic material: differential thermal analysis. *J. Mater. Sci. Letters* 11, 339–340, 1992.
 - 16) Wakasa, K., Matsui, H. and Yamaki, M.: A thermoanalytical study on non-asbestos ring-lining materials for dental application. *J. Mater. Sci. Mater. Med.* 1, 207–210, 1990.
 - 17) Hobo, S. and Iwara, T.: Castable apatite ceramics as a new biocompatible restorative material. II. Fabrication of the restoration. *Quintessence Int.* 3, 207–216, 1985.