Rapid burnout type gypsum–bonded investment materials were developed to shorten the time required for dental casting procedures. With these materials, molds can be prepared by rapid heating at 700 °C for 30 min from 30 min after the start of mixing. When the investment block was rapidly heated at 700 °C, no fractures were observed in the rapid burnout type investments with one exception, while a conventional cristobalite investment broke into pieces shortly after being placed in the furnace. Casting fins were sometimes induced only for the material which showed fracturing on rapid heating. No practical problems were found in the surface roughness of the castings. The 30 min–setting expansion was significantly different among the materials although there were no differences in thermal expansion, and the material showing greater 30 min–setting expansion was efficient to obtain better fit of the crown as in the conventional casting procedures.

Key words: Rapid burnout gypsum–bonded investment, Crown, Fit

INTRODUCTION

Recently, methods to reduce the time required for the dental casting procedure have been developed1–6), and several commercial products of the rapid burnout type gypsum–bonded investment are now available. Molds prepared with these materials is quickly placed in a furnace kept at 700 °C 30 min after the start of mixing and heated for 30 min before casting. Thus, the investment is exposed to extremely severe working conditions, and the potential of mold fracture, surface roughness and poor fit of the casting may be anticipated. Some inherent characteristics of the products have been reported7). The authors previously examined the experimental rapid burnout investments with different expansion behaviors, one showing greater setting expansion and the other greater thermal expansion with total expansion being almost the same, and reported that while both could well tolerate rapid burnout, the mold with greater setting expansion was more effective in obtaining a better fit of the cast crown8) as in conventional investment materials9–11). In the present report, the applicability of the commercial products was evaluated in terms of fracture resistance of the mold and surface aspects and fit of the cast crown associated with the expansion behavior and the rapid burnout procedure.
MATERIALS AND METHODS

Materials
Three commercially available rapid burnout type gypsum-bonded investments were employed in this study (Table 1). A conventional type cristobalite investment was also used as a control for comparison.

Expansion of the investment
The investment materials were mixed with a mechanical mixer* according to the manufacturers’ instructions, and the linear setting expansion was measured through a differential transducer for specimens 100 mm in length with a cross-sectional area of 20×20 mm. The measurements were carried out in a room at 23±2 °C and 60±10 % relative humidity. For the measurement of thermal expansion, specimens 6 mm in diameter and 50 mm long were prepared by pouring the investment slurry into a mold. At 30 min after the start of mixing, the specimen was placed in a fused quartz tube in the furnace of the thermal expansion meter** maintained at 700 °C. The expansion during heating was measured through a differential transducer for 30 min.

Resistance to fracture by rapid heating
The investment slurry was poured into a mold to form a block 30 mm in diameter and 15 mm in height with a quadratic prism hollow prepared with wax. At 30 min after the start of mixing, the block was placed in the furnace at 700 °C and held there for 30 min. The apparent change in configuration of the block was examined during heating.

Casting of crowns
A shouldered type metal die 8 mm in cervical diameter, 6 mm in height and 1/10 in taper was used as the abutment for testing. A duplicate of the die was made using a vinyl silicone impression material# and a modified dental stone##. The wax pattern was prepared on the stone die. The tight fit of the wax pattern at the margin was checked with a stereoscope and a sprue 2.5 mm in diameter and 4 mm long was attached to the line angle of the pattern. The investments were mechanically mixed at the water/powder ratios recommended by each

Table 1 Rapid burnout gypsum-bonded investment materials used

<table>
<thead>
<tr>
<th>Code</th>
<th>Material</th>
<th>w/p</th>
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<tbody>
<tr>
<td>NC</td>
<td>Noritake Cristobalite F* F*¹</td>
<td>0.35</td>
</tr>
<tr>
<td>SC</td>
<td>Cristobalite PF*²</td>
<td>0.35</td>
</tr>
<tr>
<td>CQ</td>
<td>Cristoquick II*³</td>
<td>0.33</td>
</tr>
<tr>
<td>CR</td>
<td>Cristobalite Micro*⁴</td>
<td>0.33</td>
</tr>
</tbody>
</table>

*¹ Noritake Co. Ltd., Nagoya, Japan
*² Shofu Inc., Kyoto, Japan
*³ GC Corp., Tokyo, Japan
*⁴ GC Corp., Tokyo, Japan, as a control

* VAC-U-VESTOR, Whip Mix Corp., Louisville, KY, USA
** EKO, Eikouseiki, Osaka, Japan
# Exaflex, GC Corp., Tokyo, Japan
## Fujirock, GC Corp., Tokyo, Japan
manufacturer. Two wax patterns of the crown were invested in one casting ring in two ways: (a) with their margins inward, and (b) with their margins outward. The casting ring employed was 46 mm in diameter and 40 mm in height, and was lined with two folds of exclusive ceramic liners 0.7-, 0.5- and 0.7-mm thick for NC, SC and CQ, respectively. At 30 min after the start of mixing of the investment, the green mold was placed in the furnace at 700 °C and left there for 30 min according to the manufacturers' instructions. Immediately after this, the crown was cast in Au-Ag-Pd alloy® with a centrifugal casting machine. Fig. 1 shows castings obtained by the two investing methods described above. When the conventional type investment was used, the mold was slowly heated in the conventional manner from 1 hour after the start of mixing. Ten castings for each investment were made for each of the following examinations

(1) Casting fins
The castings were examined for the presence of fins at their margins.

(2) Fit of cast crowns
The fit of the crown to the abutment die was evaluated by the amount of the vertical gap observed between them at the margin when the crown was put on the die. The gap was measured with a 1/100 mm comparator at four reference points of the crown and the average was calculated from ten castings for each investment.

Surface roughness of castings
A glass plate and a metal die with grooves 28 μm in average depth were used as the original models substituting for the finished and prepared surfaces of the abutment tooth, respectively, following the report of Matsunobu15). The impressions of the models were taken with a vinyl silicone impression material®, wax patterns were prepared on the duplicated stone dies## and casting was performed by the procedures described above. The surface of the casting was examined with a profilometric roughness tester@@. Ten castings were made for each
RESULTS

Fig. 2 shows the setting expansion curves of the rapid burnout gypsum-bonded investments mixed at the standard water/powder ratios recommended by each manufacturer. The expansion of CQ was more rapid than those of the other two materials and was almost constant 60 min after the start of mixing. In NC and SC, expansion occurred relatively slowly and slight increases were still observed even 120 min after the start of mixing. Fig. 3 shows the setting expansion at 30 min after the start of mixing, the thermal expansion when the specimen was subjected to rapid heating at 700 °C for 30 min from 30 min after the start of mixing, and the total expansion summed up from the setting and thermal expansions. The setting expansion of the rapid burnout investments were much greater than that of the conventional investment CR, 0.40 %. Although no marked differences were found in the 120
min-setting expansion among the three investment materials, the 30 min-setting expansion was the greatest in CQ and smallest in SC. The thermal expansion of CR was 1.45% when the specimen was heated conventionally. The rapid burnout investments showed almost the same thermal expansion on rapid heating as CR and there were no significant differences among the three.

Investment blocks rapidly heated in the furnace at 700 °C for 30 min from 30 min after the start of mixing are shown in Fig. 4. The block of the conventional type cristobalite investment broke into pieces shortly after being placed in the furnace at 700 °C. Among the rapid burnout type materials, NC showed the greatest likelihood to fracture in the furnace. Small cracks were sometimes observed in SC, while the CQ block was quite sound even after 30 min rapid heating at 700 °C.

Fig. 5 shows an example of the fins formed at the margin of the cast crown. The frequencies of occurrence for each investment material are listed in Table 2. Fins occurred only with NC investment molds when two wax patterns were invested in one ring with their margins toward the ring wall. No fins were observed in the castings with SC or CQ molds. When the patterns were invested with their margins toward the ring center, on the other hand, no fins were found at the margin for any investment material.

![Fig. 4 Appearances of the investment blocks after rapid burnout at 700 °C for 30 min.](image)
Fig. 5 An example of fins observed at the margin of crown for NC.

Table 2 Number of castings out of ten on which the fins appeared at the margin

<table>
<thead>
<tr>
<th>Mold</th>
<th>NC</th>
<th>SC</th>
<th>CQ</th>
</tr>
</thead>
<tbody>
<tr>
<td>a</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>b</td>
<td>4</td>
<td>0</td>
<td>0</td>
</tr>
</tbody>
</table>

a: Two wax patterns were invested with their margins toward the ring center.
b: Two wax patterns were invested with their margins toward the ring wall.

Fig. 6 shows the surface profiles of the glass plate and metal die with grooves used as the original models for the surface roughness test and of the resultant castings with each investment. The roughness values of the resultant castings are shown in Fig. 7. When a glass plate was used as the original model (roughness: 0 μmRz), the surface of the casting was somewhat roughened (Fig. 7-a). For the rough original surface (roughness: 28 μmRz), in contrast, the casting tended to exhibit less roughness than the original (Fig. 7-b). In both cases, no significant differences were found in the surface roughness among all the castings made from the rapid burnout type investment materials tested, although they showed greater roughness than those made from the conventional type cristobalite investment.

Fig. 8 shows the apparent fitting state of the cast crowns on their respective dies prepared from the original metal die. The test was performed for the castings obtained by investing the wax patterns with their margins toward the ring center which were apparently sound without fins. When SC or NC was used, the crown showed poor fit to the die and a large gap was observed at the margin between the crown and the die. Almost no marginal gap was visible to the naked eyes in crowns prepared with CQ. The measured gap values are graphically represented in Fig. 9. The average marginal gaps in NC, SC and CQ were 190, 236 and 88 μm, respectively. The crown made in the conventional manner with CR showed a much better fit to the die.
DISCUSSION

With conventional cristobalite type gypsum-bonded investment, the mold is commonly stored for at least 1 hour until the setting of the investment is complete before the start of heating. Furthermore, the mold has to be heated slowly from a low temperature up to 700 °C taking 1.5-2 hours otherwise the rapid heating may induce surface roughening and/or cracking of the mold due to rapid thermal expansion of the cristobalite. According to the demand for shortening the time required for the casting procedure, several commercially available rapid burnout gypsum-bonded investment materials were formulated to allow placement directly into the furnace at 700 °C only 30 min from the start of mixing and heating for 30 min. Even with such early and rapid heating, little or no fractures were observed in the SC and CQ blocks, although the conventional cristobalite type investment (CR) block broke into pieces as shown in Fig. 4. The improvement in the thermal behavior may be attributed to the replacement of part of the cristobalite with quartz to avoid undesirable rapid thermal expansion. In NC, the improvement appeared to be insufficient, and partial fracture was observed in the block on early and rapid heating.

One of the potential problems in adopting these materials is the fear of the occurrence of casting fins due to cracking of the mold during heating. In SC and CQ which exhibited no fractures on examination of the thermal resistance by rapid heating, no fins appeared on the cast crowns. In NC, on the other hand, thin fins were observed at the margin in half of the specimens especially when two wax patterns were invested with their margins toward the ring wall. This may reflect the increased likelihood of fracturing in NC blocks on rapid

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Fig. 6 Surface profiles of the original model and the castings.

a: for glass plate
b: for prepared surface
Fig. 7 Comparison of the roughness values of the casting surface among the investment materials.

a: for glass plate
b: for prepared surface

Fig. 8 Appearances of try-on cast crowns.
heating. The fins could be eliminated by investing the patterns with their margins toward the ring center, although the reason for this was not clarified in this study.

Another potential problem with regard to the surface aspects of the casting is the possibility of surface roughening. Matsunobu\textsuperscript{12)} examined the changes in surface roughness by a series of conventional casting procedures and described that the smooth surface of the original model would become slightly roughened on the resultant casting, and that the roughness of the prepared surface of the abutment tooth would, in contrast, decrease on the casting. The same tendencies were also observed with the rapid burnout procedures. When compared with a conventional investment\textsuperscript{12)}, all the three rapid burnout materials showed greater surface roughness of the casting for both the smooth and rough surfaces of the model. However, the increments seemed to be in a range negligible for clinical practice.

With regard to the setting expansion, when 30 min had elapsed from the start of mixing all the materials were still expanding. Only the expansion of CQ had almost ceased at 60 min as in the convensional investment, but those of NC and SC were still increasing even at 120 min. Although no differences were found in the 120 min-setting expansions among the three materials, the 30 min-expansion was the greatest in CQ and smallest in SC. There were no differences in the thermal expansion among them, and hence the difference in the total expansion was attributed to the difference in setting expansion. Nishioka\textsuperscript{9)}, Kozono\textsuperscript{10)} and Kozono \textit{et al.}\textsuperscript{11)} investigated the contribution of the setting expansion of the investment to better fit of the cast crown for gypsum-bonded and phosphate-bonded investment materials, and demonstrated that it was impossible to make dimensionally precise duplicates of the wax pattern by casting. They showed that good fit of the crown was dependent on a favorable deformation in which only the inside of the crown became larger than that of the wax pattern, and that the larger setting expansion of the investment might be the most efficient for causing such a deformation. Asao \textit{et al.}\textsuperscript{9)} confirmed that this concept could be applied to the casting procedure using rapid burnout type investment from a study of experimental materials. It was found in this study that the commercially available material showing greater 30 min-setting expansion could also produce better fitting cast crowns. However, the marginal gap between the crown and its die observed with the rapid burnout investments was significantly larger than that with the conventional investment, although the
setting expansion of the former was much greater than that of the latter. Different setting expansion values would probably be desired for particular types of investment depending on the setting rate and setting expansion rate.

The metal die used in this study was of a shape with a sharp edge of line angle between the occlusal and axial planes commonly employed for fit tests. In the clinical preparation of the abutment in contrast, the line angle has a relatively round edge and it may be easier to make well-fitting crowns than for the metal die. Thus, the test using the metal die may be regarded as much more severe than the conditions encountered in clinical practice. Fig. 10 shows the fitting appearances of the molar crowns on their respective dies prepared clinically using NC and CQ. A marginal gap was seen in the crown made from NC (a) although it was markedly smaller than that observed in the test with the metal die. The crown from CQ showed an excellent fit to the die (b) both visually and sensorily. These findings indicate that investments showing marginal gaps up to the level of 80 μm in the test using metal die as in CQ would cause no problems of fit in practice. As crowns have a potential of being ill-fitting even in clinical preparations when NC or SC show gaps as large as 200 μm or more in the test, it will be necessary to consider some means such as applying a coating film to enlarge the die, or increasing the setting expansion of the investment by controlling the mixing conditions or by delaying the start of heating.

Thus, the rapid burnout type gypsum-bonded investment materials were found to be practically very useful; however, it is essential to select the material with a greater setting expansion or devise a plan to increase the setting expansion to obtain better fit of the crown.

CONCLUSION

The applicability of rapid burnout type gypsum-bonded investment was examined using three commercial products. Although the setting process was considered to still being in progress at 30 min after the start of mixing when the mold was placed in the furnace at 700 °C, they could well tolerate this early and rapid heating. Only NC showed a risk of cracking and
occurrence of fins at the margin of the crown. There were no problems with regard to surface roughening of the casting in any of the materials tested. The 30 min-setting expansion was the greatest in CQ and the smallest in SC while there were no significant differences in thermal expansion among the three. In the fit test of the cast crown, materials with greater setting expansion showed better fitting of the resultant crown. Although CQ showed a better fit, the marginal gap between the crown and the die was markedly larger than in the conventional investment. However, it was possible for CQ to produce well-fitting crowns as determined visually and sensorily, for the clinically prepared abutment with a relatively rounded line angle. This indicates the necessity of increasing the inherent 30 min-setting expansion and/or delaying the start of heating in the other two materials to improve the better fit of the crown.

REFERENCES

然浸漬状態（+50〜+100mV）での腐食速度は、窒化チタンでコーティングすることにより1/10以下となることから、最表層はTiO₂皮膜となっており、純チタンの表面皮膜と化学組成が同じであること、自然浸漬状態での耐食性が改善され、ニッケルの溶出量が減少すると予想されることから、生体内で+500mV以上に分極されることがなければNi-50Ti合金に対する窒化チタンコーティングは効果があるものと考えられる。

急速加熱型せっこう系埋没材と鋳造体の性状

第1報 クラウンの表面性状と適合性

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1九州歯科大学歯科理工学講座
2中山医学院牙医学系

鋳造の短時間化を目的として、急速加熱型せっこう系埋没材が開発された。この埋没材で作製した鋳型は、埋没材の練和開始から30分後に700℃の炉内に入れ、30分間加熱するだけで鋳造できるものである。本研究では3種類の市販品について、膨張特性ならびに耐熱性との関連から鋳造クラウンの表面性状および適合性を調べた。練和した埋没材でブロックを作製し、練和開始から30分後に700℃の炉内で急速加熱すると、従来型のクリストバライト埋没材は炉内に入れて間もなく粉々に破壊してしまったが、急速加熱型埋没材ではまったく破壊は生じないが、生じても小さなものであった。この試験で小さなクラックが生じた埋没材においてのみ、クラウンのマージンにわずかなバリが生じることがあったが、全般的なバリの発生はなかった。鋳造体の表面あらゆる部分に大きな影響はみられなかった。急速加熱した際の熱膨張量には用いた3種類の埋没材間でほとんど差は認められなかったが、練和開始から30分後の細化膨張は著しく異なっており、従来の鋳造法の場合と同様に、クラウンの良好な適合性を得るには加熱開始前の硬化程度を大なる埋没材ほど有効であることがわかった。

Bis-GMA および iso-Bis-GMA の NMR スペクトル

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東京医科歯科大学歯学部総合診断部

Bis-GMA はコンポジットレジンやそのポンドング剤のモノマー系に用いられている。Bis-GMA は精製がされていないので、未反応原料や合成過程に生ずる種々の不純物が含まれている。本研究では市販Bis-GMA からHPLC によりBis-GMA およびiso-Bis-GMA をスリフリした後に、رمزレジン及びカーボンの全シグナルの帰属を分析した。