**Effect of burnout temperatures on strength of phosphate-bonded investments — Part II: effect of metal temperature**

H. W.-K. Luk*, B. W. Darvell†

*Dental Technology Unit, The University of Hong Kong, Faculty of Dentistry, Prince Philip Dental Hospital, 34 Hospital Road, Hong Kong
†Dental Materials Science Unit, The University of Hong Kong, Faculty of Dentistry, Prince Philip Dental Hospital, 34 Hospital Road, Hong Kong

**ABSTRACT**

Objectives: It had previously been found that the strength of phosphate-bonded investments is temperature sensitive. However, while the effect of heat from the cast metal is expected to have some effect on continued reactions and melting, there is no published report dealing with this. It was the purpose of this study to consider further the effects of burnout temperature on strength and the contribution of metal casting temperature and investment composition.

Methods: The disc-rupture test (Luk, H. W.-K. and Darvell, B. W., Strength of phosphate-bonded investments at high temperature. Dental Materials 1991, 7, 99–102) was employed to determine the effect of burnout temperature (ranging from 400 to 1000°C) on the 'actual' strength of six phosphate-bonded investments; the cast metal temperature was controlled to be the same as that of the mould. Atomic absorption was employed to investigate aspects of the composition of the investments.

Results: The 'actual' strength obtained in this test was always higher than the 'service' strength observed in similar testing with the metal at a fixed temperature (1460°C). The 'actual' strength was also temperature sensitive, but with a different pattern of variation compared with the 'service' values. All investments tested were found to behave plastically at high temperatures. Calcium, sodium, zinc and iron were found in appreciable quantities in the investments.

Conclusion: Heat from the high-temperature casting metal has a material effect in decreasing the strength of phosphate-bonded investments. Such heating increases the plastic behaviour of the investment and this, together with the casting pressure, may result in a distorted mould and thus inaccurate castings, a hitherto unrecognized source of error. 'Actual' strength gives no guide to the 'service' strength, emphasizing the need for tests under service conditions. Composition affects investment high-temperature strength substantially. © 1997 Elsevier Science Ltd.

**KEY WORDS:** Phosphate-bonded investments, Strength, Casting

J. Dent. 1997; 25: 423–430 (Received 20 February 1996; accepted 1 June 1996)

**INTRODUCTION**

The importance of understanding the high-temperature strength of dental investments has been emphasized in an earlier paper. Phosphate-bonded investment (PBI) has been used for casting a wide range of dental alloys, e.g. those of gold, base metals, titanium, etc. A sound and accurate casting can be obtained only when the investment can withstand the heat (emitted from the casting metal) and the casting pressure. The casting temperature of dental alloys varies greatly, from 950 to 1500°C. Since the strength of PBIs is temperature sensitive, it is to be expected that not only will the initial (burnout) temperature of the investment be important,
Table 1. Batch number and the compressive strength of the six phosphate-bonded investments

<table>
<thead>
<tr>
<th>Investment</th>
<th>Batch</th>
<th>Strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Auroplus</td>
<td>540010078</td>
<td>9 'Resistance to pressure'</td>
</tr>
<tr>
<td>Biosint Supra</td>
<td>0956265</td>
<td>15-20</td>
</tr>
<tr>
<td>Ceramigold</td>
<td>067530984</td>
<td>6.9 (2 h); 20.7 (dry)</td>
</tr>
<tr>
<td>DVP</td>
<td>098340184</td>
<td>27.6 (2 h); 55.1 (dry)</td>
</tr>
<tr>
<td>Hi-Temp</td>
<td>027610684</td>
<td>6.8 (2 h)</td>
</tr>
<tr>
<td>X-20</td>
<td>093730184</td>
<td>Not given</td>
</tr>
</tbody>
</table>

but the casting temperature of the metal will also have a substantial effect. However, there is no known prior report on this. It was therefore the purpose of this study to consider the effect of casting metal temperature on the high-temperature strength of phosphate-bonded investments. In addition, a preliminary examination of the composition of the investments was made in an attempt to account for the variation in observed strength.

MATERIALS AND METHODS

The disc-rupture test described by Luk and Darvell was again employed. The materials and methods follow those of Luk and Darvell with the following exceptions:

(1) Different batches of the six phosphate-bonded investments (Table I);
(2) the casting metal was lead, melted in a carbon lined crucible;
(3) the metal casting temperature was matched to the mould temperature, which ranged from 400 to 1000°C in steps of 100°C. The casting temperature was controlled by a K-type thermocouple because the casting machine could not read temperatures lower than 800°C.

The purpose of this approach was to avoid raising the temperature of the investment above that of burnout by heat from the metal. Lead provides a convenient means of doing this. A total of 332 castings were made (Table II).

Aspects of the composition of the six investments, in the batches used by Luk and Darvell, were investigated using atomic absorption spectrophotometry in a preliminary analysis. This method was employed to test for cations present in an acid extracted solution of each of the six PBIs. Ten grams of investment was added to 50 ml deionized water, 10 ml of HCl (12 M, AR grade, Hopkin and Williams, Essex, UK) was added and the mixture stirred, using a PTFE-coated magnetic stirrer, for about 15 min. The mixture was then filtered through Whatman No. 42 paper. The filtrate was made up to 100 ml with deionized water. This solution was then examined using an atomic absorption spectrophotometer (Smith-Hieftje 12, Thermo Jarrell Ash, Franklin, MA, USA). The concentrations of eight elements were checked: lead, copper, iron, zinc, manganese, sodium, calcium and magnesium. Potassium was also examined but in a different centre and machine (SpectrAA, Varian, Springvale, Australia) because the first did not have a potassium lamp. A blank, made by mixing 10 ml HCl and 90 ml deionized water, was also examined as a control. Five readings were taken for each ion (except potassium, for which only one reading was taken); the mean and standard deviation were recorded.

RESULTS

The strength results are shown in Fig. 1. The relative strength of X-20 at 400 and 500°C, and that of DVP at all temperatures, were all too high to be measured with the chosen disc thickness. The strength was generally highly temperature sensitive. Strong (local) minima were found between 500 and 600°C, and around 900°C, for some products.

The results of the acid extract of the tested investments are shown in Table III. Noteworthy is that the amount of zinc in Auroplus was very much greater than what appears to be background contamination in the other investments, while the sodium content of Auroplus and Biosint Supra was rather low. DVP had a very high magnesium content. An appreciable amount of calcium was present in all products.

DISCUSSION

Temperature and strength

In the former 'fixed' test the metal (Co-Cr) casting temperature was fixed at 1460°C, while in the present 'matched' test the metal (Pb) casting temperature was matched to that of the mould. Each of these tests has its own special meaning. The 'fixed' test was designed to simulate the actual procedure and conditions in dental casting so that the strength obtained could reflect the actual service performance of the materials; in the 'matched' test the effect of heat from the high-temperature casting alloy was eliminated, allowing an independent observation of the effect of furnace temperature on the strength of dental investments. Thus, when the strengths obtained in these two cases are compared, the effect of heat from the casting alloy can be deduced.

The present results are compared with those of the former study in Fig. 2. It is striking that the observed strength in the 'fixed' case was always less than that obtained in the present 'matched' case. The only differences in the conditions for the two cases were the casting metal and the casting temperature. In the 'matched' test, the casting metal (lead) and the investment mould were meant to be isothermic, no effect of extra heat on the strength of the investment was expected. Whilst it is possible that differences in the
Table II. Number of test castings made for each product and temperature. No failures were observed with DVP at any load up to the maximum possible

<table>
<thead>
<tr>
<th>Temp. (°C)</th>
<th>Auroplus</th>
<th>Biosint Supra</th>
<th>Ceramigold</th>
<th>DVP</th>
<th>Hi-Temp</th>
<th>X-20</th>
</tr>
</thead>
<tbody>
<tr>
<td>400</td>
<td>5</td>
<td>10</td>
<td>5</td>
<td>2</td>
<td>10</td>
<td>7</td>
</tr>
<tr>
<td>500</td>
<td>6</td>
<td>5</td>
<td>8</td>
<td>3</td>
<td>6</td>
<td>4</td>
</tr>
<tr>
<td>600</td>
<td>11</td>
<td>9</td>
<td>5</td>
<td>4</td>
<td>14</td>
<td>9</td>
</tr>
<tr>
<td>700</td>
<td>8</td>
<td>8</td>
<td>5</td>
<td>3</td>
<td>16</td>
<td>10</td>
</tr>
<tr>
<td>800</td>
<td>5</td>
<td>11</td>
<td>6</td>
<td>3</td>
<td>12</td>
<td>11</td>
</tr>
<tr>
<td>900</td>
<td>18</td>
<td>18</td>
<td>9</td>
<td>2</td>
<td>9</td>
<td>7</td>
</tr>
<tr>
<td>1000</td>
<td>7</td>
<td>10</td>
<td>9</td>
<td>3</td>
<td>9</td>
<td>10</td>
</tr>
</tbody>
</table>

Fig. 1. A summary of the strength curves of the six investments for the present 'matched' temperature tests. The temperatures of some major possible reactions or changes during the course of burnout are labelled: a, the decomposition of $\text{NH}_4\text{MgPO}_4\cdot\text{H}_2\text{O}$; b, the $\alpha$-$\beta$ transformation of quartz; c, formation of $\text{SiO}_2\cdot\text{P}_2\text{O}_5$; d, formation of $\text{Mg}_2(\text{PO}_4)_2$.

reactions between the metal oxides formed and the investments could contribute to the observed variation in strength, e.g. PbO contributes to the lowering of the softening temperature of silica glass[4], this is considered to be rather unlikely on the basis that the time available for diffusion and reaction was very short and failure commenced on the other, tension side of the disc to that of exposure. The lower strength in the 'fixed' case is therefore probably due to the heat emitted by the high-temperature metal, which might be expected to cause cracking through thermal shock, but also by softening and melting the investment. The metal casting temperature (1460°C) was higher than even the melting points of the supposed burnout reaction products $\text{Mg}_3\text{P}_2\text{O}_7$, $\text{Mg}_2(\text{PO}_4)_2$, and at least several of the possible binary eutectic mixtures, were they present (Table IV). On the other hand, the high-temperature reaction between oxides formed on the hot metal and the investment[5] might effectively reduce the thickness of the investment disc, the strength thus obtained would be that of a thinner disc. Again, it is a matter of the time available for diffusion into the disc material. This was considered to be a relatively insignificant effect.

According to Luk and Darvell[1], the maximum stress experienced by the disc in the disc-rupture test occurs when the casting arm is at its highest speed of rotation and all molten metal has entered the mould. But measurements showed that in the 'fixed' test the disc could be broken as late as at 5.7 s, which is much longer than the expected timing, i.e. less than 1 s[6]. Therefore, something other than casting force is involved in the breakage. The likelihood is that the heat of the metal, which induced some degree of melting or softening, and thus weakening of the investment, explains the delayed failure of the disc. This process is in fact indicated by the plastic deformation of the disc before breakage (Fig. 3). Investments are normally described as brittle materials, but the present results indicate that all the investments tested, in both the 'fixed' and the 'matched' tests, deformed in a plastic way at high temperatures; this behaviour seems not to have been considered or reported previously in the dental context.

The fracture of the investment disc in the 'matched' test should be dynamic (i.e. fracture before the maximum rotational speed) because there was apparently no other factor than the casting force to cause failure, although this has not yet been explicitly tested. But that in the 'fixed' test was complicated because the effect of heat from the casting metal was included. There were then two situations for failure to occur in this latter test: either the casting force was great enough to cause a close-to-dynamic fracture or it was less than the critical requirement for dynamic fracture, but there was a delayed fracture. The latter kind of fracture may be similar to the so-called 'kinetic fracture' described by Lawn[7]: 'Cracks can often extend at subcritical stress levels, especially in the presence of reactive chemical environments. A major characteristic of this type of fracture is its time dependence'. The three factors that govern the rate of the kinetic fracture are the magnitude of the applied stress, the concentration of chemically active species, and the temperature[7]. High temperature can cause, besides softening of glass and melting, further chemical reactions of the components of the investment and, additionally, reactions between the
investment and the metal oxides from the casting metal although these are discounted now. It is presumed that the time dependency of such reactions resulted in the delayed failure of the investment disc in the 'fixed' test. Because of the time-dependence of the fracture in the 'fixed' test, the solidification time of the casting metal is another factor of concern. Even if the investment became weak enough that the disc could be broken through by the amount of metal in the mould, breakage could not occur if the metal had already solidified. The solidification time of casting metal is governed by the casting temperature, the environmental and mould temperatures, and mould design, as well as the thermal conductivity, specific heat capacity and thermal mass of the mould material. The higher the mould and the metal casting temperatures the longer will be the solidification time. For example, the solidification time of a gold crown casting temperature of 940°C ranged from 4 to 6.5 s for a mould temperature of 650°C but only from 2 to 2.8 s for a mould temperature of 480°C.

The strength observed in the 'fixed' test must be closer to the 'service strength' of the investments, while that in the 'matched' test might be said to be the 'actual strength' under the chosen burnout conditions as the effect of the heat from the casting metal is eliminated. In the 'matched' test, the strength of DVP was too high to be measured, which is not inconsistent with the data which indicate that DVP has the highest room temperature range from 700 to 1100°C (see Fig. 2). This emphasizes that 'actual strength' is unlikely to give any proper indication of the 'service strength', and this again indicates the importance of accounting for the effect of heat from the casting alloy when the strength of dental investments is being determined. If the 'service strength' of dental investments has to be measured, the method of burnout, the mechanism of loading and the effect of heat from the casting metal must all be taken into account.

The high-temperature plastic phenomenon of dental investments deserves further investigation in actual practice because the casting pressure and the heat of the casting metal may be a hitherto unrecognized cause of distortion in dental castings. 1etereck and Mumford reported that of over three hundred castings constructed using different investments and techniques, none could seat completely. The casting temperature of Co-Cr and alloys for metal–ceramic devices can range from ~1200 to ~1500°C, when both Mg₃P₂O₇ and Mg₃(PO₄)₂ can easily be melted. The chance of distortion due to such softening and melting would be small in thin castings, such as a full denture plate, as the hot casting alloy would solidify very quickly when it reached the mould space because of the high surface area to volume ratio of the denture design. But for the pontic area in the case of a dental bridge, the solidification time may be up to a few seconds or more, depending on the mould and casting temperatures; distortion may then occur. On the other hand, thin walls between gross porosity and the mould space may be easily deformed or broken through, creating defects on the casting. In fact, the porous nature of investmens is such that 'faults' per se are not required for collapse to occur. If PBIs were to be used for titanium casting, the mould may be more likely to be distorted because the casting temperature of titanium is of the order of 1700°C, high enough even to melt the silica. Reduction of silica by titanium might also be expected on thermodynamic grounds. One method to reduce the effect of heat from the casting alloy is to use a low mould temperature, say 600°C, or even 350°C, so that the metal solidifies in a shorter period. But this would lower the casting system effectiveness and increase the risk of an incomplete casting. Investments with high refractoriness are required to resist the effect of heat from the casting alloys. Currently, CaO, MgO and ZrO₂ investments are in the process of development for high-temperature casting, e.g. for titanium and its alloys, because of their high melting temperatures and stability with respect to reaction with the metal.

Figure 2 also shows that the strength curves of the 'fixed' test tend to fluctuate less than those of the 'matched' test. In the cases of Auroplus and Hi-Temp, the curves obtained from the 'fixed' and 'matched' tests are basically of the same pattern, except for the
Fig. 2. The strength curves obtained in the 'fixed' (solid line with F) and 'matched' (dotted line with M) tests are compared for individual investments. A dashed line indicates that the strength was too high to be measured using the 1.4 mm thick disc.
Table IV. Some temperatures of relevance to phosphate-bonded investments

<table>
<thead>
<tr>
<th>Substance</th>
<th>m.p. (°C)</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ca$_2$P$_2$O$_7$</td>
<td>1230</td>
<td>Weast 24</td>
</tr>
<tr>
<td>MgO</td>
<td>2850</td>
<td>Weast 24</td>
</tr>
<tr>
<td>Mg$_3$P$_2$O$_7$</td>
<td>1383</td>
<td>Weast 24</td>
</tr>
<tr>
<td>Mg$_3$(PO$_4$)$_2$</td>
<td>1184</td>
<td>Weast 24</td>
</tr>
<tr>
<td>NaCl</td>
<td>801</td>
<td>Weast 24</td>
</tr>
<tr>
<td>Na$_2$CO$_3$</td>
<td>851</td>
<td>Weast 24</td>
</tr>
<tr>
<td>Na$_2$PO$_4$·12H$_2$O</td>
<td>100</td>
<td>Weast 24</td>
</tr>
<tr>
<td>Na$_2$P$_2$O$_7$</td>
<td>880</td>
<td>Weast 24</td>
</tr>
<tr>
<td>NH$_4$H$_2$PO$_4$</td>
<td>190</td>
<td>Weast 24</td>
</tr>
<tr>
<td>SiO$_2$</td>
<td>1610-1723</td>
<td>Weast 24</td>
</tr>
</tbody>
</table>

System Eutectic Pt (°C) Reference

<table>
<thead>
<tr>
<th>System</th>
<th>Eutectic Pt (°C)</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>CaO·P$_2$O$_5$</td>
<td>480</td>
<td>Kreidler and Hillmer 23</td>
</tr>
<tr>
<td>Fe$_2$O$_3$·P$_2$O$_5$</td>
<td>964; 968</td>
<td>Wentrup 27</td>
</tr>
<tr>
<td>Na$_2$O·SiO$_2$</td>
<td>793</td>
<td>Turkdogan and Maddocks 28</td>
</tr>
<tr>
<td>Na$_2$O·P$_2$O$_5$</td>
<td>546</td>
<td>Turkdogan and Maddocks 28</td>
</tr>
<tr>
<td>P$_2$O$_5$·SiO$_2$</td>
<td>900</td>
<td>Mal'shikov and Bondar 29</td>
</tr>
<tr>
<td>ZnO·ZnO·P$_2$O$_5$</td>
<td>630</td>
<td>Kanack and Hummel 30</td>
</tr>
</tbody>
</table>

Fig. 3 The change of the distal disc curvature of the investment disc from flat to curved as the amount of casting metal was increasing in the 'fixed' test.

presence of a peak at 800°C on the curves of the 'matched' test. The cause of the peak must be some reaction, e.g. a formative reaction (although there is no evidence as to what this might be), but these peaks are not found in the corresponding curves of the 'fixed' test. Evidently, the effect of the heat from the Co-Cr was to suppress them, probably by melting or softening any substances formed. In the case of Biosint Supra, the 'fixed' test strength increased steadily after the minimum at 600°C, but the 'matched' test strength dropped steadily after its maximum at 500°C, although the drop was greater from 900 to 1000°C. The other fluctuations are probably not statistically significant.

**Composition and strength**

A number of metals were found in the investments (Table III); there may, of course, be others for which no test was made. Some of these were assumed to be impurities (e.g. lead, copper, manganese, potassium, etc.) because of their low concentration; some seemed to be intentionally added as reactants or modifiers (e.g. zinc in Auroplus, sodium in Ceramigold, DVP, Hi-Temp, and X-20) because of their rather high concentration. Calcium was found in all products to an appreciable amount, but whether as an impurity or an added substance is not known. The latter, however, seems more likely as the proportion (by mass) with respect to the magnesium ranged from about 3 to 30% (atomic proportion: from 2 to 20%). CaO can act as a setting agent similar to MgO to control the setting aspect of the investment. Even so, no extra substances have been mentioned by manufacturers, except for the graphite in Ceramigold.

Nevertheless, no matter whether the above substances are intentionally added or just impurities, their presence is expected to affect the high-temperature strength of investments, the magnitude of the effect depending on how much and what substances are present. For example, sodium compounds tend to fuse at low temperatures (Table IV) so that they are expected to affect the high-temperature strength of investments if they are present in any considerable amount. Moore and Watts 20 suggested not to use sodium compounds, or at least not more than 1–2% by mass, in PBIs.

DVP has the highest content of magnesium, while Ceramigold has the least (Table III). The ranking of the presumed magnesia content for the six investments is therefore:

DVP > X-20 > Biosint Supra > Hi-Temp > Auroplus > Ceramigold

Comparing this order with the strength curves in Fig. 1, it seems that the 'matched' high-temperature strength of the six investments basically follows the above order, with the exception of Auroplus which showed some slight deviation (but this has a high zinc content). Without the heat effect of the casting alloy, DVP was shown to have the highest strength at all temperatures tested, while Ceramigold possessed the least strength at most temperatures (Fig. 1). The relationship between the 'matched' strength and magnesia content is shown in more detail in Fig. 4. Since the strength of DVP was too high to be measured, it could not be entered into the graph but clearly it fits the general trend.

It is known that the mass percentage of the binder ($\text{NH}_4\text{H}_2\text{PO}_4$) in phosphate-bonded investments is about the same as that of the setting agent (MgO). Although there is no evidence that the present products necessarily fit this pattern, it would be reasonable to suppose that it is followed. It has also been reported that the more binder, the higher the strength of the investment at both room and burnout temperatures. Thus, the highest strength of DVP in the 'matched' test could then be explained by it containing more binder.
than the other investments. Similarly, Ceramigold contained less binder than the others and also showed the least strength at most of the test temperatures. The magnesia content (and presumably phosphate also) being highest in DVP means that it would have the highest content of Mg₃P₂O₇ after 650°C or Mg₅(PO₄)₂ after 1040°C, both of which melt below the casting temperature of the Co-Cr (1460°C) in the ‘fixed’ tests. This may explain why the observed strength was the least for DVP in the ‘fixed’ tests from 700°C onward. The present results also suggest that the proportion of the major component indeed has a substantial effect on the strength of the investments.

A forward stepwise multiple regression analysis of the strength data was performed (SigmaStat, v 1.02, Jandel Scientific Software, San Rafael, CA, USA) to test the effect on strength of the amounts of magnesium, calcium and sodium as determined in the atomic absorption analysis (Table III). The results were clear in that magnesium was strongly positively associated with increasing strength in the ‘matched’ case \( b = 1.26 \pm 0.20, P < 0.001 \), but only weakly negatively so in the ‘fixed’ case \( b = -0.10 \pm 0.04, P = 0.009 \). The effect of calcium, which was indistinguishable from that of sodium (but as an alias, not independently), was very strongly associated with decreasing strength \( b = -0.69 \pm 2.08, P = 0.003 \) in the ‘matched’ case. There was no detectable effect in the ‘fixed’ case. Although it might be expected that sodium should have a stronger negative effect than calcium, there are too few data to resolve this question, and more products or formulations will require investigation to clarify such issues.

The above result of the possible effect of calcium on strength has not previously been reported for PBIs. The lowest eutectic point in the system CaO–P₂O₅ is at 481°C, such a system could easily be formed during the burnout process. Both calcium and sodium can form metal ammonium phosphates, similar to that of magnesium, which decompose when heated and would also lead to a decrease in strength; their decomposition is expected to form pyrophosphates (for melting points see Table IV). Na₄P₂O₇ would therefore seem to have a stronger effect in decreasing the strength of the investment as it melts at a comparatively low temperature; this may be why sodium is not recommended to be added in PBIs.

One factor that would be expected to be important in determining strength that has not been considered here is volume fraction of pores. However, because of the functional requirements for porosity and surface finish, it is expected that this will be similar for all products tested and not affect the broad composition effects now detected. Study of this point may refine understanding of behaviour. Even so, this does not affect the main conclusions regarding variation within products according to burnout and metal temperatures.

It can be concluded that variation in strength with temperature and between brands of investment indicates that strength depends very much on composition and the amount of heat supplied. The strength at any temperature is in fact a resultant property of various events. The effect of an individual event may not be easily observed in the strength curves as it may be dominated or cancelled by others. It is difficult to explain the shape of the strength curves unless the effect of each of the components and their reactions at different temperatures are fully recognized. More work is necessary on ‘kinetic’ fracture, especially the effects of composition and the reactions of the components at high temperature, if there is to be any progress in understanding high-temperature strength. It is also worth looking into the timing of the dynamic fracture in the ‘matched’ test as this has not been tested.

Although investments may distort castings by resisting alloy shrinkage, the high-temperature plastic behaviour of dental investment should also be taken into account when distortion is being considered. Further work is therefore necessary on this aspect of the behaviour of dental investments.

Acknowledgements
We thank Dr Lau and Mr Chan of the City Polytechnic University, and Mr Lee of the Oral Biology Unit, University of Hong Kong, for their help in conducting the atomic absorption tests.

References


