

Differing Expansion Contributions of Three Investment Materials Used for Casting Titanium

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Keywords

Setting expansion; phosphate-bonded investment; titanium casting; crown accuracy; setting temperature.

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Abstract

Purpose: This laboratory study aimed at investigating the effect of setting expansion (SE), which could enlarge a wax pattern by concurrent exothermic reaction.

Materials and Methods: Two phosphate-bonded investment materials (Rematitan Plus, T-invest C & B) and alumina-magnesia-based investment material (Titavest CB) were subjected to setting temperature and SE measurements ($n = 10$). Full-crown wax patterns were prepared from metal dies having the same design. Crown castings ($n = 6$) were made using a one-chamber high-pressure casting machine. Commercially pure titanium ingot (Japanese Industrial Standard Class II) was used for each casting. Dimensional accuracy of the cast crowns was expressed by marginal discrepancy for a pair of wax pattern and casting prepared from the same die. All results were then subjected to regression analysis.

Results: Rematitan Plus gave the highest setting temperature (about 70°C) and the highest SE ($1.16 \pm 0.01\%$) almost concurrently. The result was that Rematitan Plus produced oversized crowns from the lowest thermal expansion (TE) value ($0.53 \pm 0.05\%$) reducing the linear correlation to 0.80 ($p < 0.05$). No significant linear relationship was found between SE of investment material and crown-casting accuracy.

Conclusions: The almost concurrent occurrence of high SE and high setting temperature generation in Rematitan Plus enlarged the wax pattern and effectively supplemented its low TE to produce oversized crowns. Despite this adverse effect, the correlation between TE and crown accuracy was still high. It is generally expected that the TE values given by manufacturers predict crown accuracy, when uniform TE, small SE, and minimum heat generation during setting are assured.

The concept that the total of setting and thermal expansions of an investment material compensates for metal shrinkage can be traced back to investigations of dental gold casting at the beginning of the last century.¹⁻³ The total expansion of investment materials is a function of various factors, such as the chemical setting contraction (change in crystal structure of matrix phase), physical setting expansion (SE) (crystal impingement), thermal SE (heat rise during reaction), thermal expansion (during heating), and refractories and binder transformation (during heating/cooling). One practical way of assessing the possible uneven contribution of factors in the making of accurate castings is to establish a couple of individual linear correlations of each individual factor. Then, by following multiple linear regression analysis of all factors, the best model to predict the dimensional accuracy of castings can be found.⁴

Contemporary dentistry has an opportunity to revisit this traditional compensation theory with the study of titanium (Ti)

casting restorations. Previous experiments that adopted the full-crown design and a statistical approach found a high correlation ($R = 0.87$) between investment thermal expansion (TE) and Ti crown accuracy when the TE values of four investments were determined by a standardized laboratory method.^{4,5} A linear regression analysis of casting accuracy was made on full-crown castings made from these investments.^{4,5} The dimensional accuracy of crown casting (Y) was expressed as the marginal discrepancy (μm) between wax pattern and Ti crown casting both seated on the same metal die, and a formula ($Y = 1041X - 1004$) was obtained to predict Ti casting accuracy from TE (X, %). Thus, a TE value of 0.97% was required to make a zero discrepancy casting; however, no correlation was found between SE and Ti crown accuracy, although this part of the analysis used SE values given by the manufacturers.⁴ This result agrees with the view that the role of SE from investment material is minimal in the casting accuracy of full-crown

Table 1 Investment materials used and data given by the manufacturers

	Titavest CB (A)	T-invest C & B (B)	Rematitan Plus (E)
Manufacturer	J. Morita, Kyoto, Japan	GC, Tokyo, Japan	Dentaurum, Pforzheim, Germany
Batch number	Powder: 1296 Liquid: 1039	Powder: 200451 Liquid: 140751	Powder: 059530 Liquid: 079411
Recommended			
L/P ratio	0.15	0.13	0.16
SE (%)	–	0.66	0.34
TE (%)		0.64	0.54
Total expansion (%)	1.5 or less	1.33	0.88
Target mold temperature (°C)	Room temperature	600	430

restorations, because the wax pattern that surrounds the central core section of an investment mold resists the expansion exerted by the setting investment material.⁶⁻¹⁴

Distortion of wax patterns has been reported for cast-crown restorations cast into investment molds prepared from phosphate-bonded investment materials.^{6,15-19} The problem is generally related to the so-called high hygroscopic setting expansion, which can be up to 5%, and commonly occurs with the use of wet liner or water bath.^{6,15} This distortion problem becomes more serious when exothermic setting reaction is considerable, in particular with phosphate-bonded investment materials. The heat released will soften the wax pattern and contribute an inseparable TE component to the measured SE of investment material; however, little is known of such combined effects of exothermic reaction and SE of investment material on crown-casting accuracy. Currently, only two international standards are available for gypsum-bonded and phosphate-bonded dental investment materials.^{20,21} Among these standards, methods for measurement of setting and thermal expansions of dental investment materials are reported; however, evaluation of other setting temperature behavior is not available.

This investigation used a phosphate-bonded investment material, which appeared to generate considerable heat during setting reaction, to study the unknown effect of exothermic reaction and SE investment material on crown-casting accuracy. The research hypothesis was that the amount of SE of investment material would have an insignificant effect on dimensional accuracy of full-crown casting restorations. This laboratory study aimed at investigating the effect of SE, which could enlarge a wax pattern by concurrent exothermic reaction.

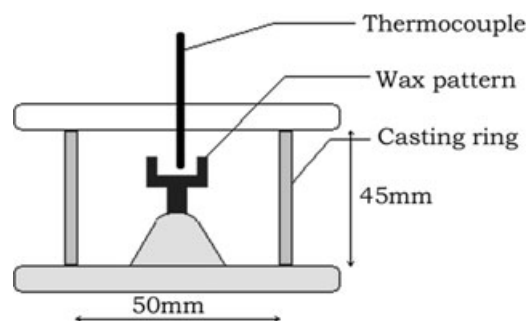
Materials and methods

The three investment materials in this study, together with data provided by the manufacturers, are summarized in Table 1. For the purpose of this study, SE and setting temperature were measured for all three investment materials. The TE of investment material was also measured to supplement the study of Ti crown-casting accuracy. TE of investment materials was measured using a differential dilatometer (TMA 8140 & TAS 100, Rigaku Denki Corp., Tokyo, Japan). The standard error of the differential dilatometer is less than 0.1% for each temperature unit of measurement. Measurements were made according to the manufacturer's instructions.⁴ Ambient laboratory condi-

tions during the complete series of experiments were $21 \pm 1^\circ\text{C}$ and $50 \pm 10\%$ relative humidity.

This experiment was conducted according to the method given in ISO 7490.²⁰ The apparatus was connected to a linear transducer (050 DC-D, Schaevitz Engineering, Pennsauken, NJ), which had been calibrated with a digital micrometer. Outputs from the transducer are fed to a data-logger (Data Taker 500, Datataker, Scoresby, Australia) each second to obtain a continuous SE curve. SE was determined to the nearest 0.01 mm and expressed as a percentage of the original specimen length. The commencement of mixing was taken as zero time, and the final SE was determined at 2 hours. Ten measurements were made for each investment material.

A schematic diagram of the temperature measurement conducted in this study is shown in Figure 1. The wax pattern was prepared from a set of standardized metal dies. A sprued wax pattern was mounted on a crucible former in such a way that the length of exposed sprue was about 5 mm, and placed in a plastic ring, 50 mm in diameter and 45 mm in height, having a vertical slit without ring liner. The investment mix was poured into the casting ring, and then a Type-K thermocouple was inserted in the center of the wax pattern through a hole in a tightly fitting Perspex lid to locate the hot junction at 5 mm below the shoulder margin. Two glass beakers were placed to cover the mold to minimize heat loss. The thermocouples were connected to a data-logger (DT500, Datataker), which had been calibrated with a digital thermometer. The measurement continued for 2 hours, taking the commencement of mixing as zero time. Ten measurements were made for each investment material.

**Figure 1** Temperature measurement made on the investment mold.

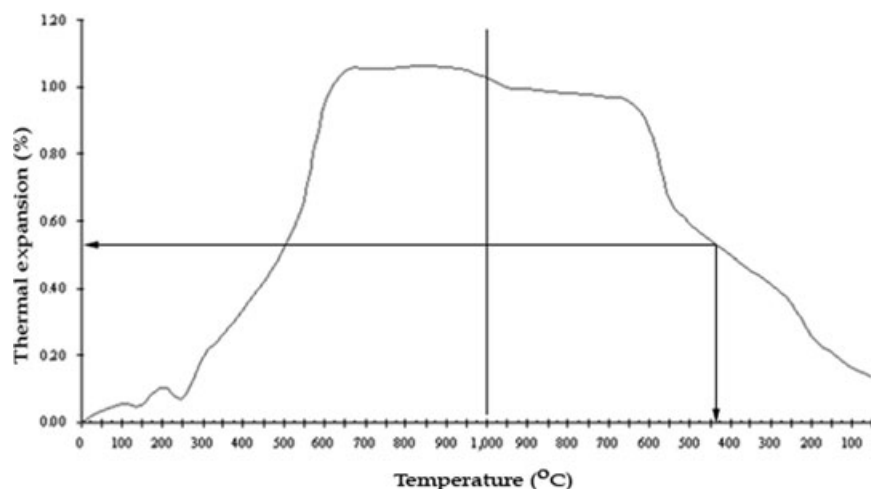


Figure 2 The thermal expansion curve for investment material E (Rematitan Plus). The vertical reference line represents the casting mold temperature (430°C) and the corresponding thermal expansion of 0.53%.

Blue inlay casting wax (Type I, Kerr Corp., Orange, CA) was used to prepare the pattern from these standard dies. The prepared wax pattern was placed in a plastic ring (51 mm in diameter, 46 mm in height), and then invested with investment materials. Set molds were removed from the plastic ring after about 2 hours from mixing and kept in plastic bags overnight. The set molds of each investment material were heated to their target temperatures as recommended by the manufacturer (Table 1). Six crown castings were made for each investment material with a one-chamber pressure casting machine (Autocast H-C III, GC Corp., Tokyo, Japan). A 30-g CP Ti ingot (T-alloy M, GC Corp.) (Japanese Industrial Standard Class II) was used for each casting.

Assessment of Ti crown-casting accuracy was previously reported and was made with a set of standardized metal dies.²² The mean dimensional accuracy expressed as vertical axial discrepancy was obtained from four measurements for each crown casting. Six crown castings ($n = 6$) were made.

Results of the SE measurement were subjected to two-sample Student's *t*-test assuming unequal variances. The least-squares linear regression analysis was made using all accuracy data collected from each crown against a mean setting expansion value of each investment material. The regression model was subjected to analysis of variance (ANOVA) to determine the significance of the regression model between setting value and casting accuracy. A best-fitted line together with equation was obtained from the regression. The correlation coefficient (*R*) and standard error (*S*) of the regression model after ANOVA were also reported. All analyses were made using a statistical program (Minitab Release 11.12, 1996, Minitab Inc., State College, PA).

Results

A typical TE curve for investment material E (Rematitan Plus) is shown in Figure 2. The mean TE value of E obtained was 0.53% (SD 0.05) at the recommended mold temperature of 430°C. The TE of investment materials A and B was reported previously.⁴ All TE data are summarized in Table 2.

The SE values obtained for the three investment materials are shown in Table 2. It also summarizes results of TE measurement and crown accuracy assessment determined for investment material E, together with data previously determined for investment materials A and B.⁴ The SE values of the three investment materials were significantly different ($p < 0.01$), lowest with investment material A and highest with investment material E. The former showed a maximum of only 0.002% at about 25 minutes and then approached zero at around 1 hour. Quantification is not possible for any shrinkage, due to the design of the measuring device. Investment material E showed the highest SE (1.16%) and lowest TE (0.53%) and produced oversized crown castings ($192 \pm 60 \mu\text{m}$).

The setting temperature measurements of the three investment materials, with average temperature increase (ΔT) from 20°C, are plotted for each material in Figure 3. The average SE curves are also shown. The temperature rise of investment material E (Rematitan Plus) was rapid, giving the largest ΔT value, approaching 50°C at 12.5 minutes. The SE curve appeared to have an inseparable TE component, since both SE and temperature increase occurred almost concurrently. The maximum SE value of 1.16% was obtained for the highest mold temperature of 70°C, and this was maintained to the end. The temperature increase of B (T-invest C & B) was much more modest, giving the largest ΔT value of only 18°C at about 27.5 minutes. The temperature and SE curves were separated, the former preceding

Table 2 Thermal expansion (TE), setting expansion (SE) of investment material, and full-crown casting accuracy

Investment material	SE (%) ($n = 10$)	TE (%) ($n = 10$)	Casting accuracy (μm)
A (Titavest CB)	0.00 (0.00) ^d	1.59 (0.09)*	518 (447) ($n = 6$)*
B (T-invest C & B)	0.37 (0.05)*	0.64 (0.01)*	-316 (93) ($n = 8$)*
E (Rematitan Plus)	1.16 (0.01) ^f	0.53 (0.05)	192 (60) ($n = 6$)

Values are means (standard deviation). *Data from a previous study.⁴ Differing letters within a column indicate significant difference ($p < 0.01$). Negative result indicates undersized casting.

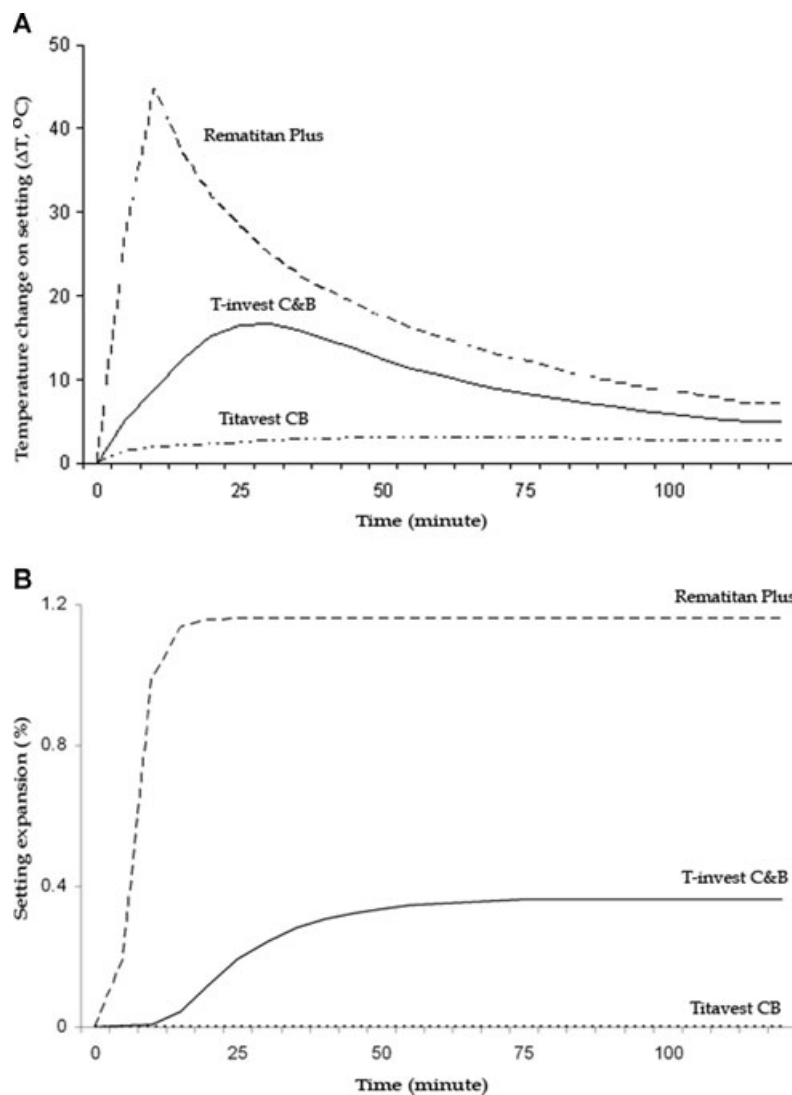


Figure 3 (A) Setting expansion (%) curves during setting of three investment materials. (B) Temperature change curves of investment material during setting.

about 10 minutes. The SE value kept increasing, giving the average maximum of 0.37% in the second half of measurement. In investment materials B (T-invest C & B) and E (Rematitan Plus), the mold temperature was reduced to temperatures close to ambient temperature at 2 hours.

The renewed linear regression analysis made between TE and Ti crown-casting accuracy is shown in Figure 4, where results obtained from investment E are further incorporated into previous analysis.⁴ The new regression model showed a highly significant ($p < 0.001$) correlation ($R = 0.8$, $S = 391$), and an equation of $Y = 1017X - 891$ was obtained. No significant relationship was found between SE and crown-casting accuracy ($R = 0.1$, $S = 441$).

Discussion

The thermal expansion behavior of investment B was very similar to that of investment B in a previous study⁴ showing little change at temperatures above 600°C on heating and cooling.

The rapid or isothermal expansions (contractions) due to the inversion of cristobalite and quartz were clear, notably on cooling, at around 250°C and 600°C, respectively. The TE curves were reproducible, and an average TE value of 0.53% was obtained for the recommended mold temperature of 430°C.

The shrinkage of Ti metal from its solidus to room temperature can be estimated as about 1.6% from existing expansion coefficient data ($\alpha = 8.5 \times 10^{-6}/^{\circ}\text{C}$).^{23,24} Of the three investment materials investigated in this study, investment material A will be the material of choice for people who wish to have a total investment expansion close to the theoretical metal shrinkage value (Table 1); however, ranking of the investments by total expansion of investment material ($A > B > E$), based on the manufacturer's values, changed to $E > A > B$ by the present setting and thermal expansion measurements. This was because a large discrepancy with investment material E's SE value (1.16%) was obtained against the manufacturer's value (0.34%). This is not surprising, since SE is generally sensitive to many factors, such as measuring stress and exposure to

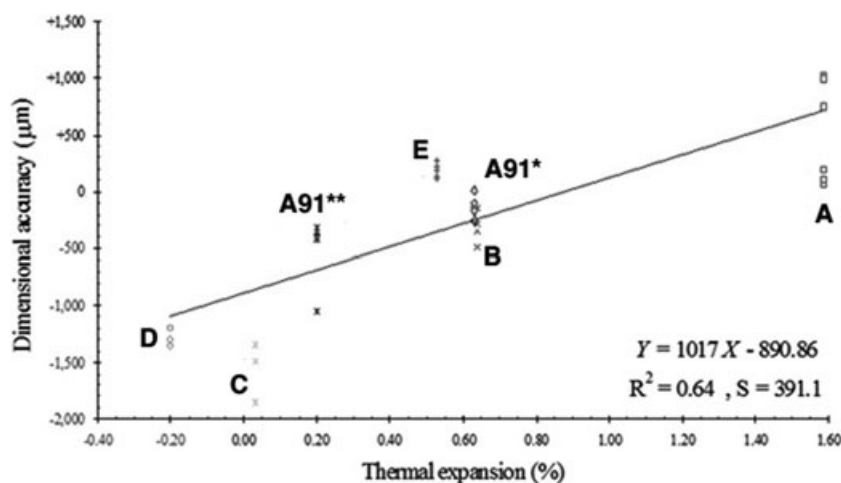


Figure 4 A simple least-square linear regression analysis made between thermal expansion and dimensional accuracy with results from present and previous studies.^{4,5} (A = Titavest CB, n = 6);⁴ (B = T-invest C & B, n = 8);⁴ (C = Taivest, n = 3);⁵ (D = Rematitan, n = 3);⁵ (E = Rematitan Plus, n = 6); (A91* = Titavest CB with highest target temperature of 950°C, n = 7);⁵ (A91** = Titavest CB with highest target temperature of 900°C, n = 6).⁵

water. The discrepancy was particularly large with investment material E.

The high correlation between TE of investment material and crown-casting accuracy found in the previous study⁴ was again confirmed in this study with a small reduction in the coefficient from 0.87 to 0.80. On the other hand, no significant relationship between SE of investment material and crown-casting accuracy, which was previously found by using the manufacturers' setting expansion values,⁴ was again confirmed by the actual measurement ($R = 0.1$, $S = 441$). Based on manufacturer's data, investment material A provided the total expansion value only (Table 1). Assuming its SE as zero, the compensation expected from the manufacturers' TE values is $A > B > E$. The TE values obtained in this study did not change this ranking, since the measured values were almost the same as those claimed by the manufacturers. This is a fortunate situation for general users, who rely on manufacturers' data.

The new equation obtained, $Y = 1017X - 891$, reduced the required TE value (X) from the previous 0.96% to 0.88% for zero discrepancy ($Y = 0$). That is, only investment material A is expected to produce oversized crown castings, and the

ranking for metal shrinkage compensation should be $A > B > E$; however, investment material E also produced oversized crown castings (Table 2), and this is the reason the correlation coefficient in the regression analysis was reduced (Fig 5). The production of undersized crown castings is expected with T-invest C & B for its lower TE value (0.64%), while the production of oversized crowns is unexpected with Rematitan Plus for its lowest TE value (0.53%).

Rematitan Plus showed considerable heat generation (exothermic) during setting (Fig 3). The melting range of the inlay wax used in this present study is 58.1°C to 63.6°C.²⁵ The highest temperature reached with investment material E (Rematitan Plus) was 70°C, where ΔT approached 50°C at 12.5 minutes (Fig 3). Before the wax melts, however, the wax crown pattern is enlarged by the expanding investment core, since the compressive strength and elastic modulus of the wax decreases, 7.77 to 0.24 MPa and 0.696 to 0.003 GPa, for ΔT of 20°C from ambient temperature.²⁶ This could account for why investment material E produced oversized crown castings, even though TE is small (0.53%). On the other hand, the wax pattern does not melt with investment material B, but large

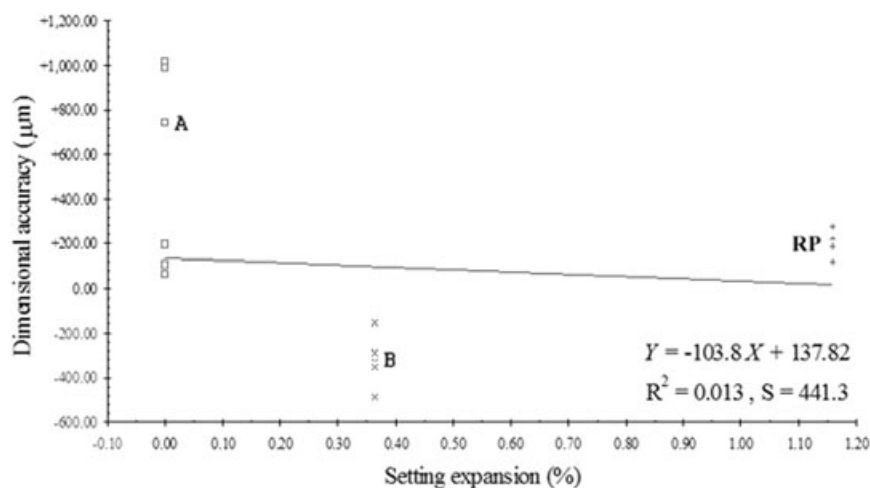


Figure 5 A simple least-square linear regression analysis made between setting expansion and dimensional accuracy. (A = Titavest CB; B = T-invest C & B; RP = Rematitan Plus).

decreases in the mechanical properties will similarly occur in the pattern, as the value of ΔT approaches 20°C at around 25 minutes. Compared with investment material E, however, the degree of wax pattern enlargement remained smaller due to the much smaller SE (0.37%), and thus the cast crowns remained undersized despite similar TE values, 0.64% (B) and 0.54% (E).

The reason the cast crowns made from investment material E showed a small scatter despite the high SE (Table 2, Fig 4) can be explained by the use of a plastic ring with a slit. It is often said that provision of ample cushion by increasing the thickness or number of liners is an essential prerequisite for any assessment of dimensional accuracy; however, it is practically impossible to accommodate high SE of investment materials often encountered with phosphate-bonded investment material using common liners. Liners also complicate the SE of investment material behavior due to the so-called hygroscopic reaction. With the provision of ample cushion to accommodate investment mold expansion, the concept that the total of setting and thermal expansions compensates for metal shrinkage will be valid in the case of Class I inlay casting fabrication. It is also essential to minimize any distortion of wax patterns, and for this use of low SE and low heat investment, elimination of hygroscopic expansion and adoption of the ring-less method are safer approaches. Insufficient TE can be supplemented by the use of a die spacer as well. Without these precautions, any attempt to assess crown-casting accuracy has limited value.

Conclusions

Wax pattern distortion could easily occur with phosphate-bonded investment material when SE accompanied by a concurrent exothermic reaction enlarges the wax pattern by softening and/or melting of the wax pattern. This effectively supplements an insufficient TE of investment material and produces oversized Ti crown castings. Despite the obvious contribution of SE of investment material confirmed with the investment chosen for this study, the correlation between SE of investment material and Ti crown-casting accuracy was negligible, and a high correlation between TE of investment material and Ti crown-casting accuracy was maintained ($p < 0.05$).

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