BASIC SCIENCE RESEARCH Dimensional Accuracy of 7 Die Materials

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<u>Purpose</u>: The purpose of this study was to compare the linear dimensional accuracy and the handling characteristics of 7 die materials.

<u>Materials and Methods</u>: A master die analogous to a complete veneer crown preparation was machined from medical grade stainless steel, and 3 measurements (1: vertical; 2 and 3: horizontal) were made from 3 scribed reference lines. Individual polyvinylsiloxane impressions were made (n = 10) for each of the specimens. The fabricated dies were measured ($50 \times$) to the nearest 0.0001 mm. Data were subject to ANOVA/Duncan tests at significance level 0.05 and pairwise comparisons.

<u>Results</u>: Type IV resin-impregnated dental stone and copper-plated dies most closely approximated the dimensions of the master die, and were not significantly different from each other in any of the pairwise comparisons. Conventional Types IV and V dental stone dies exhibited setting expansion within the range appropriate for gypsum. Epoxy resin die materials demonstrated shrinkage comparable to the expansion of the Types IV and V dies. Polyurethane dies displayed a combination of linear expansion and shrinkage. Bis-acryl composite resin dies had excessive shrinkage.

<u>Conclusions</u>: Type IV resin-impregnated dental stone and copper-plated dies were more dimensionally accurate than the other die materials tested.

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INDEX WORDS: Type IV die stone, dental gypsum, epoxy resin, copper plating, dental materials

 $O^{\rm NE}_{\rm the}$ of the potential sources of error in the fabrication of a fixed prosthesis is the

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Accepted June 30, 2004. Copyright © 2005 by The American College of Prosthodontists 1059-941X/05 doi: 10.1111/j.1532-849X.2005.00007.x die material used during the lost wax process. A number of materials are currently available, yet none of these satisfy all the desirable qualities of a die system.¹ ISO (International Standards Organization) Types IV (high-strength, low-expansion) and V (high-strength, high-expansion) improved stones are the most commonly used die materials, due to their perceived dimensional accuracy, low cost, and ease of use.² Improved dental stones have less than ideal abrasion resistance,²⁻⁶ strength, and detail reproduction,² however. Those characteristics become more important as the complexity and the span of a fixed prosthesis increases, or when porcelain margins are fabricated.

Alternative die materials such as copperplated, resin-impregnated gypsum, epoxy, and polyurethane resins have been shown to possess superior abrasion resistance,^{2,7} strength, and detail reproduction compared with improved dental stones;^{6,8-11} however, results regarding their dimensional accuracy are equivocal. Bailey et al¹² and Cassimaty¹³ found electroplated dies to be more dimensionally accurate than stone dies while others have demonstrated the reverse.¹⁴ Type IV resin-impregnated dies have been shown to be more dimensionally accurate than conventional Type V stone,¹⁵ while another study found no significant differences between conventional gypsum and Type IV resin-impregnated stone.⁹ Epoxy resin dies exhibit shrinkage in the range of 0.1-0.4%,^{12,16} yet in one study this material more closely approximated a metal master when compared with Types V and IV resin-impregnated materials.¹⁵ Other results indicate there are no significant differences in dimensional accuracy between stone and epoxy resin dies.¹² The limited number of dimensional studies of polyurethane resin have shown it to undergo polymerization shrinkage comparable to or slightly less than epoxy resin.^{10,17} Manufacturers claim that the tested epoxy and polyurethane have improved handling properties and polymerization shrinkage 0.001% and 0.025%, respectively.

Ease of use and time required for fabrication are factors to consider when selecting a die material. Fabrication of a copper-plated die, for example, involves more processes than other types of die systems. It also requires the use of sulfuric acid, and dies are usually not ready until the next day. Dental stone can be easily vibrated into the impression and is claimed to be ready for use within an hour; however, a recent investigation¹⁸ found that dental stones continue to expand for 96 hours after mixing. No current die materials offer a genuinely fast set, which would facilitate more rapid initiation of transfer copings, diagnostic waxups, or other in-house or chair-side laboratory procedures. While not specifically developed for use as a die material, the manufacturer claims that bis-acryl composite resin (Integrity, LD Caulk, Dentsply Int., Technical Support) shrinks only 0.06-0.08%. Due to its potential advantage of rapid set and flow characteristics that permit injection into an impression, bis-acryl composite resin was included as one of the tested die materials.

The objective of this study was to compare the linear dimensional accuracy and, to a lesser extent, the convenience of dies fabricated from 7 materials: conventional Type IV dental stone, Type V dental stone, resin-impregnated Type IV dental stone, epoxy resin, polyurethane resin, copperplated, and bis-acryl composite resin.

Materials and Methods

A master die (Fig 1) analogous to a complete veneer crown preparation was machined from medical grade

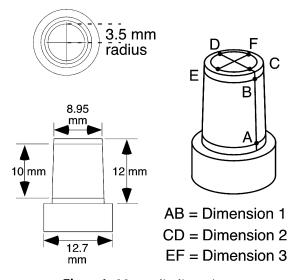


Figure 1. Master die dimensions.

stainless steel to the specifications originally described by Bailey et al.¹² Three measurements were made from scribed reference lines, one vertical and two horizontal. Dimension 1 was the vertical measurement from Point A to Point B. Dimension 2 was the horizontal measurement from Point C to Point D. Dimension 3 was the second horizontal measurement from Point E to Point F.

The master measurements for the die were taken from the mean of 4 readings using a Unitron Microscope Model DMM 200. The approximate power of magnification used was $50 \times$. A single operator performed all procedures throughout the investigation to eliminate multioperator optical error in cross hair alignment of the microscope.

Three millimeters of relief was placed over the master die and 10 individual impression trays were fabricated from custom tray material (Triad® Tru-TrayTM, Dentsply Int., York, PA). An alignment procedure was used to maintain the desired thickness of material when making impressions (Fig 2). Mechanical and adhesive retention was used within the trays and an addition silicone material was used for all impressions (Extrude Xtra Putty and Wash, Kerr USA, Romulus, MI). Individual impressions were made for each fabricated die, and 10 dies were made for each material (Fig 3), a total of 70 dies. All die materials were used per manufacturers' instructions.

The 3 gypsum-based die materials were proportioned and mixed with distilled water at the ratios shown (Table 1). Materials were hand mixed in a 200-ml Vac-u-Mixer bowl (Whip Mix Corp, Louisville, KY) until the powder was wetted and then mechanically mixed under approximately 30 lb. vacuum at 400 rpm in a Vac-u-Vestor (Whip Mix Corp, Louisville, KY). Impressions were poured



Figure 2. Impression taking device.

with each mix, and the gypsum specimens were separated from their impressions 1 hour after mixing.

Die-Epoxy base and hardener were mixed by hand in a 200-ml Vac-u-Vestor at a ratio of 100 g/10 ml for 1 minute, then for 30 seconds under vacuum. Impressions were poured. The epoxy dies were separated from their impressions after 5 hours.

 $Model-Tech^{TM}$ polyurethane resin base, hardener, and filler were mixed by hand for 30 seconds in a Vac-u-



Figure 3. Seventy die specimens.

Die Materials	Manufacturer	Manufacturer Claimed Expansion (%)	$H_2 O/Powder$	Mixing Times	Ready to Use
Vel-Mix /T.m. IV.)*	Kerr Bomulue MI	0.06	20 ml/100 g	30 seconds hand	30 minutes
Hard Rock	Whip Mix Corp.	0.28	21 ml/100 g	30 seconds hand	30 minutes
(Lype V) Resin rock	Whip Mix Corp.	0.08	20 ml/100 g	30 seconds hand	30 minutes
Die Epoxy fast set	Amer. Dent. Sup.	-0.001	100 g base /10 ml hardener	1 minute hand	4–5 hours
(Epoxy resin) Model-Tech (D-1	Easton, FA Ivoclar No. Amer.	-0.025	10 ml base/ 5 ml hardener/ $\frac{1}{2}$	30 seconds vacuum 30 second hand	60 minutes
(rolyuretnane resin) Integrity	Dentsply Caulk	-0.06-0.08	level measures of miler –	ou seconds vacuum Automix cartridge	7 minutes
(bis-Acryl Composite) Copper-plated	MIIIOTG, DE Amer. Dent. Sup. Easton, PA	-0.07-0.04	I	Ι	12–15 hours

Vestor, then for 30 seconds under vacuum. Impressions were poured and dies were removed after 60 minutes.

To decrease viscosity, a cartridge of IntegrityTM bisacryl composite resin was warmed in a Hygrobath (Whip Mix Corp, Louisville, KY) at 100°F (38°C) for 1 hour prior to being dispensed into the impressions. Dies were removed after 7 minutes.

A copper-plating apparatus was used to fabricate the metal dies. The impression surfaces were coated with a conductor of electricity, Silver Plate[®] (American Dental Supply, Easton, PA), allowed to dry, attached to copper lead wires, and placed in the plating bath. Current was set at 15 mA for 1 hour and then increased to 45 mA for the next 12 hours. The copper-plated impressions were removed from the bath and poured up with Resin Rock[®].

The dies were measured in the same manner as the master die (the recorded dimension was the mean of 4 measurements taken) 96 hours after separation from the impressions. To further minimize optical error, a reference platform was constructed to standardize the orientation of dies beneath the microscope. Data were recorded to the nearest 0.0001 mm.

Results

The master die measurements, mean values, and standard deviations for Dimension 1, Dimension 2, and Dimension 3 for each die material are reported in Table 2. The dimensional differences (in percent) from the master die for each material are shown in Table 3. Type IV resin-impregnated and copper-plated dies most closely approximated the dimensions of the master die and were not significantly different from each other in any of the pairwise comparisons. Table 4 shows the results of the statistical analysis (Duncan *post hoc* test with significance level 0.05). Statistical differences were observed for Type IV resin-impregnated and copper-plated dies in Dimension 1 compared to the other 5 die materials. For Dimensions 2 and 3, significant differences were observed for bisacryl composite resin compared to the other 6 die materials.

Discussion

Type IV resin-impregnated and copper-plated dies most closely approximated the master die with respect to the 3 dimensions measured, and were not significantly different from each other in any of the pairwise comparisons. Statistical differences were observed for Type IV resin-impregnated and copper-plated dies in Dimension 1 only, compared to the other 5 die materials. Bis-acryl composite resin dies were significantly different from the other 6 materials in Dimensions 2 and 3.

The lack of statistically significant difference between Type IV resin-impregnated and copperplated dies compared with the other 5 materials in Dimensions 2 and 3 may be partly accounted for by the smaller length of Dimensions 2 and 3 compared to Dimension 1. Because of the difference in length, the magnitude of dimensional change would be expected to be less. Additionally, setting expansion of gypsum restricted in an impression is not isotropic.¹⁹ Dimensions 2 and 3 were restricted by the impression material, but Dimension 1 was not.

The results of this study were reasonably consistent with prior investigations of the same or similar materials. Other studies have found copperplated dies to be more accurate than gypsum dies.² Type IV resin-impregnated dies have been shown to exhibit less setting expansion than conventional Type IV dies.⁹ Paquette¹⁵ demonstrated that Type IV resin-impregnated dies were more dimensionally accurate than Type V gypsum dies.

Table 2. Master Die Measurements, Mean Values, and Standard Deviations for Die Materials

	Dimension 1		Dimension 2		Dimension 3	
	AB	SD	CD	SD	EF	SD
Master die	9.9852		6.7008		6.6854	
Copper-plated	9.9778	0.0171	6.7008	0.0218	6.6853	0.0108
Resin rock	9.9874	0.0088	6.6999	0.0104	6.6847	0.0107
Hard rock	10.0154	0.0264	6.7082	0.0324	6.6769	0.0265
Vel-Mix	10.0155	0.0367	6.7178	0.0216	6.6962	0.0196
Epoxy	9.9551	0.0278	6.6816	0.0241	6.6797	0.0169
Polyurethane	10.0280	0.0210	6.6911	0.0236	6.6798	0.0151
Bis-Acryl	9.9365	0.0250	6.6335	0.0225	6.6125	0.0222

Measurements in mm.

Table 3.	Dimensional Differences from Master Die in
Percent	

Material	Dimension	Dimension	Dimension
	1 Change	2 Change	3 Change
Copper-plated Resin rock stone Hard rock stone Vel-Mix stone Epoxy Polyurethane Bis-Acryl	$\begin{array}{c} -0.0740\% \\ +0.0219\% \\ +0.3024\% \\ +0.3038\% \\ -0.3018\% \\ +0.4282\% \\ -0.4875\% \end{array}$	$\begin{array}{r} +0.0016\% \\ -0.0140\% \\ +0.1097\% \\ +0.2536\% \\ -0.2872\% \\ -0.1454\% \\ -1.0044\% \end{array}$	$\begin{array}{r} -0.0010\%\\ -0.0105\%\\ -0.1280\%\\ +0.1612\%\\ -0.0855\%\\ -0.0847\%\\ -1.0914\%\end{array}$

Epoxy die materials contract during setting⁹ and polyurethane has been shown to both contract and expand during setting.¹⁰ Except for the resinimpregnated and copper-plated dies, the percent dimensional changes observed in our study, positive and negative, were of greater magnitude than those indicated on the manufacturers' packaging (Table 1). Differing shape and dimensions of the master models may account for this apparent discrepancy. ANSI/ADA Specification No. 25 uses a largely 1-dimensional linear master model in contrast to the more clinically relevant master die used in this study. Although not statistically significant, Type IV gypsum (Vel-Mix) expansion exceeded that of the Type V dental stone (Hard Rock) in all dimensions. While this was an unexpected finding, other investigations^{15,20} of gypsum die materials have also demonstrated percent dimensional changes of greater magnitude than those indicated on the manufacturers' packaging. In addition, Heshmati¹⁸ found that the setting expansion of Type IV gypsum (Vel-Mix) exceeded the maximum allowed for an ANSI/ADA Specification No. 25 Type IV product when measured 96 hours after mixing. The dies in this study were also measured 96 hours after mixing.

The fit and ultimate clinical success of a cast dental restoration depends on an accurate, strong, abrasion-resistant die material with good detail reproduction. The use of dimensionally accurate materials such as Type IV resin-impregnated and copper-plated dies should result in the development of a crown margin that lies in more intimate contact with the finish line of the tooth preparation, given that the dimensional changes of the wax pattern, alloy, and investment are carefully matched. Bis-acryl composite resin dies demonstrated a curing contraction > 1.0%. Since a linear dimensional change greater than 0.6% is considered excessive,² this material cannot be used when an accurate die is required. Polyurethane is not recommended as a die material because the combination of expansion in 1 dimension and shrinkage in the other 2 dimensions could be expected to result in castings that exceed the incisogingival dimension of the tooth impressed but are smaller faciolingually and mesiodistally. This would be very difficult to compensate for. The shrinkage in all 3 dimensions demonstrated by epoxy die material could result in the fabrication of an unacceptable casting that does not seat clinically unless compensatory laboratory techniques are employed. The use of this material is recommended with caution. Linear expansion of the conventional Type IV and the Type V dental stone was within the range of 0.06-0.5% expected for $gypsum^2$ in 5 of the 6 measured dimensions. The minimal setting expansion of these materials should result in the fabrication of satisfactory crown margins, while simultaneously helping to provide enough space between the casting and the tooth to allow complete seating without binding, and space for a film of cement. These materials are recommended for continued use in routine fixed prosthodontics. To create enough space between the tooth and casting when using the exceptionally accurate Type IV resin-impregnated and copperplated dies, it may be necessary to use die relief. In practice, the dentist needs to evaluate the stability and fit of a crown in the mouth with a given

Table 4.	Results	of	Statistical Analy	sis
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Variables	Differences
Mean dimensional change in Dimension 1	Type IV resin-impregnated, Copper electroplated < Type IV, Type V, Epoxy, Polyurethane, Bis-acryl
Mean dimensional change in Dimension 2	Bis-acryl < Type IV resin-impregnated, Copper-electroplated, Type IV, Type V, Epoxy, Polyurethane
Mean dimensional change in Dimension 3	Bis-acryl < Type IV resin-impregnated, Copper-electroplated, Type IV, Type V, Epoxy, Polyurethane <denotes (results="" differences="" duncan<br="" of="" significant="" statistically=""><i>post hoc</i> test with significance level 0.05)</denotes>

die material/relief combination and provide this information to the technician. Adjustments can be made in the laboratory until crowns that are stable and fit passively with this combination of materials are consistently produced.

The poor strength and abrasion resistance of conventional Types IV and V dental stone become more significant in complex fixed prosthodontics, such as when transfer copings are necessary, or when fabricating all porcelain margins. Of the tested materials, Type IV resin-impregnated and copper-plated dies are recommended in those cases because of their strength, abrasion resistance,^{8,21,22} and dimensional accuracy. If maximum strength and abrasion resistance are required, copper-plated dies could be used. If less abrasion resistance is needed, or ease of construction is essential, Type IV resin-impregnated dies would be recommended.

The reproduction of detail observed for the epoxy and polyurethane dies was superior, but this feature alone does not compensate for the drawbacks of these materials. Of the remaining 5 tested materials, copper-plated and Type IV resin-impregnated dies had the next best detail reproduction. This is another factor that increases their value as die materials.

Ease of use was confirmed for bis-acryl composite resin, but due to poor dimensional accuracy, it is contraindicated as a die material. A claimed advantage of the new epoxy and polyurethane products tested in this investigation was ease of construction, since a centrifugal casting machine is not necessary. This advantage was outweighed by the difficulty of clean up for these 2 materials.

Convenience is not one of the positive characteristics of the copper electroplating technique. It involves special equipment, materials, and a greater number of processes; requires 10–15 hours until it is ready for use; and is incompatible with many impression materials.¹ In addition, the fabrication of copper plated dies can be more technique sensitive compared with other die materials. The impression surfaces must be delicately coated with a thin layer of powdered silver to achieve an accurate, smooth, even layer of plated metal with good detail. The sectioning and trimming of copper plated dies must be performed carefully to avoid damaging the margin or adjacent proximal contacts.

The gypsum-based products (Type IV, Type V, and Type IV resin-impregnated dental stone) were

the most convenient to use. These materials can be easily vibrated into an impression and be ready for use within an hour. Gypsum-based dies are easily fabricated, sectioned, and trimmed with routinely available equipment.

Conclusions

Within the limitations of this *in vitro* study, which compared duplicate dies with a metal master, the following conclusions were drawn:

- *1.* Type IV resin-impregnated dental stone and copper-plated dies were more dimensionally accurate than the other tested materials.
- Conventional Type IV and Type V dental stones exhibited setting expansion within the range appropriate for gypsum.
- 3. Epoxy resin die materials demonstrated shrinkage comparable to the expansion of the Type IV and Type V dies.
- 4. Polyurethane dies displayed a combination of linear expansion and shrinkage.
- 5. Bis-acryl composite resin dies had excessive shrinkage.
- 6. The gypsum-based products were more convenient to use.

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