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Dimensional Accuracy of an Epoxy Die Material Using Different Polymerization Methods

Rosario Prisco, MD, DDS,[1] Giancarlo Cozzolino, CDT,[1] & Paolo Vigolo, DrOdont, MScD[2]

[1] Private Practice, Naples, Italy
[2] Assistant Professor, Department of Clinical Odontostomatology, University of Padova, Institute of Clinical Dentistry, Padova, Italy

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Correspondence
Paolo Vigolo, Via Vecchia Ferriera, 13 Vicenza 36100, Italy. E-mail: paolovigolo@virgilio.it

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Abstract

Purpose: Shrinkage of resinous die materials during setting reaction limits their acceptance, even though these materials show several advantages compared to stone die materials with respect to strength, abrasion resistance, and detail reproduction. The purpose of this study was to determine if retarding the setting reaction during polymerization and altering the base-to-catalyst ratio, as suggested by previous studies, can be recommended for resinous die materials to reduce the inaccuracy in transferring the spatial position of teeth or implants from the oral cavity to the master cast.
Materials and Methods: A Blue Star Type E epoxy resin die material was tested. A reference triangular metal master die was fabricated. Forty medium-consistency polyether impressions of this model were made. Four groups (S, M, N, P) were compared, and ten dies were fabricated for each group. In the S group, the epoxy resin die material was manipulated according to the manufacturer's instructions; in the other three groups, the epoxy resin die material was manipulated by retarding the setting reaction and by modifying the epoxy resin base/activator ratio.
Results: One-way ANOVA revealed significant differences between the four groups of the epoxy resin die material ($p < 0.0001$). Tukey's multiple comparisons test ($p < 0.05$) revealed that none of the resin groups was similar to the metal master die for each of the tested dimensions (A, B, and C). For the specific dimension C, however, the P group was statistically closer to the metal master die than the S group.
Conclusion: The epoxy resin die material tested in this research did not improve its dimensional accuracy following retarding polymerization or modifying the epoxy resin base/activator ratio. The epoxy resin material exhibited higher contraction variability across all tested groups. This shrinkage can significantly affect the dimension of the master cast.

The master casts on which fixed prostheses are fabricated must properly reproduce an abutment whether the case involves a single unit or multiple units in an entire arch rehabilitation.[1–3] They must achieve certain specifications with respect to dimensional accuracy, detail reproduction, surface hardness, abrasive resistance, compatibility with impression materials, transverse strength, ease of manipulation, and lack of toxicity.[4]

Improved dental stone and epoxy resin are the most popular materials in dentistry for constructing master casts for fixed prostheses, even though a resin-reinforced gypsum product, a polyurethane resin, and silver and copper electroplated die systems have all been used. Type IV (high strength, low expansion) gypsum products that meet American Dental Association (ADA) specification No. 25 are the most common improved dental stones used as die materials.[5] Gypsum-based materials, even though easy to manipulate and consistent in results, have shown poor abrasion resistance and inadequate tensile strength, as well as potential variability with respect to fine detail reproduction, and setting expansion between 0.01 and 0.1%.[4–5] Die stone with improved properties and improved strength has been marketed as a type V (high strength, high expansion) material.[4] The importance of sawing the master cast as quickly as possible after the setting of dental gypsum product to help control the setting expansion of dental stone die materials has been demonstrated.[6] Setting expansions of types IV and V are essentially completed within 96 hours.[7]

Resin-reinforced gypsum was developed to produce a die material with improved properties. In a study by Duke et al, the materials tested, however, were not significantly different from conventional type IV gypsum products.[8] In a previous study by Kenyon et al, the linear dimensional accuracy and handling characteristics of seven die materials were compared.

Type IV resin-impregnated dental stone and copper-plated dies were evaluated more dimensionally accurate than any other die materials tested.[9]

An epoxy resin die material, which is compatible with most impression materials, has exhibited better detail reproduction, abrasion resistance, and transverse strength than gypsum-based materials;[10–13] however, the epoxy resin material is more time-consuming and undergoes polymerization shrinkage on setting (in the range of 0.1% to 0.4%).[10–13] In a previous study by Moser et al, the epoxy resin material demonstrated properties comparable to those of gypsum.[14] Centrifuging the epoxy resin die material produced undistorted bubble-free dies. Although compressive strength, abrasion resistance, and detail reproduction of the epoxy resin were superior, the surface hardness was lower. Epoxy resin produced undersized dies. Working times, setting times, handling properties, and compatibility with impression materials were nevertheless judged to be appropriate for clinical use.[14] Vermilyea et al studied three epoxy resin die materials that showed a hardness and tensile strength superior to gypsum. In the early stages, the compressive strength of the resin was greater than that of the gypsum; at 24 hours, they seemed to be similar.[15] Nomura et al evaluated three epoxy resin materials for detail reproduction, hardness, and accuracy of fit with respect to a cast restoration. The resins demonstrated that, though their detail reproduction was comparable to die stone, the hardness of the materials tested was less than that of die stone, and with respect to a completed crown, the resin dies were undersized.[16] Aiach et al investigated the relationship between epoxy resins and different impression materials; they reported that some die shrinkage occurred. An excellent detail reproduction was seen combining epoxy resins to addition silicone and polyether.[17] Bailey et al studied the dimensional accuracy of dental stone, silver-plated, and epoxy resin die materials, finding no statistical differences.[18] The complete arch form developed by Chaffee et al showed that a newer epoxy resin material produced models that were slightly undersized in dimension from the right first molar to the central incisors, and from the left first molar to the central incisors.[19,20]

Because of the contraction of resin materials, their dimensional accuracy has been questioned, and attempts have been made to reduce shrinkage by modifying the manufacturer's instructions. An important strategy in reducing epoxy resin shrinkage might be to modify the setting reaction, thus allowing more resin to flow in the critical area of the impression, and a greater relaxation of the resin during gelation. Paquette et al[21] determined whether a retarded setting reaction and the alteration of the manufacturer's proportion instructions regarding base and catalyst could improve the accuracy of an epoxy resin die system. Four groups were compared: an epoxy resin (Ivoclar, Schaan, Liechtenstein) manipulated according to manufacturer's instructions; the same epoxy resin manipulated to undergo a retarded set; high strength, high expansion gypsum (Die Keen, Heraeus Kulzer GmbH, Hanau, Germany); and a resin-filled gypsum (Resin Rock, Whip Mix Corp., Louisville, KY). Ten dies were fabricated for each group from a metal master die. The study showed that retarding the setting reaction of an epoxy resin die material improved its accuracy, and among the materials tested, retarded set epoxy dies had the lowest mean dimensional change from the metal master; however, this study did not test different brands of epoxy resins, which may be different from each other, due to their chemical structure or percentage of chemical ingredients.

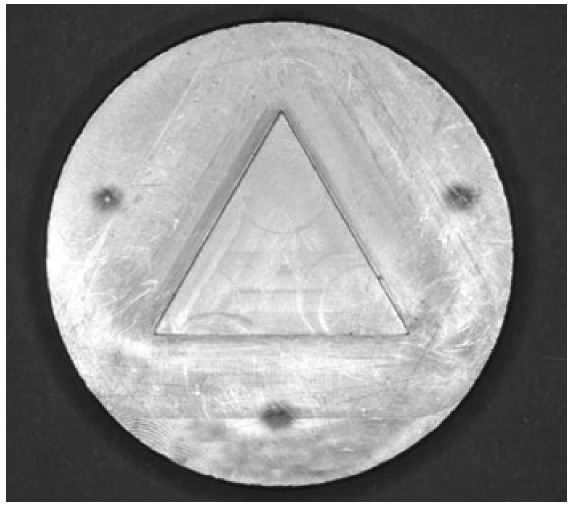

Figure 1 Carbon steel triangular master die.

Martignoni and Schonenberger stated that the master cast should be considered the most important starting point for the execution of dental technology. On the master cast all technical steps leading to completed restoration must be performed.[6] The ability of the resin material to replicate details has already been proved in many articles.[10–13,16,17] The capacity of the resin material to correctly reproduce distances is very important when, for example, a passively fitting implant structure has to be realized.[22,23]

The purpose of this study was to determine if retarding the setting reaction during polymerization and altering the base-to-catalyst ratio, as suggested by a previous study,[21] can be recommended for all resinous die materials to reduce the inaccuracy in transferring the spatial position of teeth or implants from the oral cavity to the master cast, allowing the laboratory technician to fabricate a restoration that requires fewer corrective clinical procedures.

Materials and methods

Die preparation procedures

A triangular stainless steel master die resistant to abrasion[24,25] was used as suggested by Paquette et al's protocol.[21] Side A measured 20.207 mm, side B measured 20.233 mm, and side C measured 20.201 mm (Figs 1 and 2). The 20 mm length is approximately the dimension of a three-unit fixed partial denture.[10,26] There were distinct angles and points on the master die to ensure dimensional accuracy with respect to measuring all relevant magnitudes. In this study, the ability of the resin material to reproduce details was not considered; the resin's polymerization shrinkage and its dimensional change from the metal master have been exclusively evaluated. More precise measurements were accomplished starting from the sharp angles of the triangular master die.

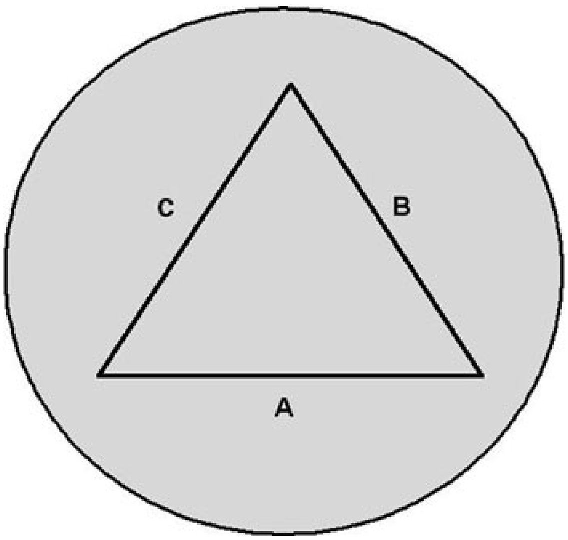

Figure 2 Graphics of carbon steel triangular master die.

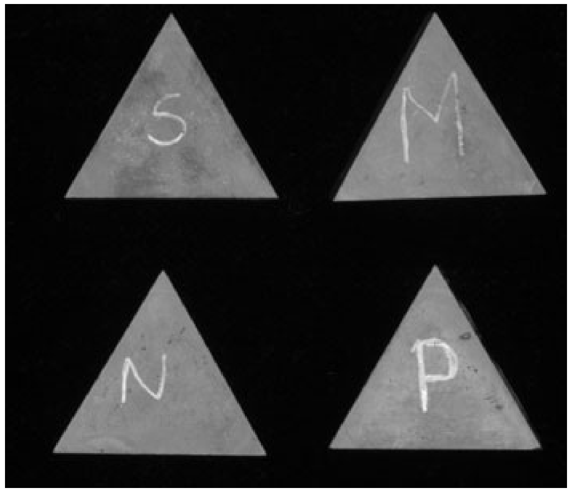

Figure 3 Epoxy resin die samples.

Custom-formed acrylic resin (SR-Ivolen, Ivoclar) impression trays, non-perforated, were produced by using the master die as a sample at least 24 hours before taking the impressions. The trays had a 3-mm gap around the triangular master die to provide sufficient space for the impression material.[27-33] Three location marks (circular depressions 2 mm wide and 1 mm deep) were made on the base of the metal die and included in the impression trays to standardize tray positioning during impression making. Twenty impressions were made in accordance with the mono-phase technique[34-38] at room temperature, using a medium body polyether impression material (Impregum Penta Soft, ESPE Dental AG, Seefeld, Germany); polyether adhesive (ESPE Dental AG) was applied and then allowed to dry on impression trays for 15 minutes, as suggested by the manufacturer. The impression material was machine mixed with Pentamix (ESPE Dental AG). Part of the material was meticulously syringed on the master die; the remaining impression material was used to load the impression trays. The impressions were removed from the master die 6 minutes after initial mixing. Four groups of ten impressions each were randomly created and then left undisturbed for 24 hours before being poured (Fig 3).

Group S: The impressions were poured with an epoxy resin base/activator with a ratio of 49 g of base to 5.36 g of catalyst according to the manufacturer's instructions (Sartorius PT 120, GMBH, Gottingen, Germany). The resin was proportioned in weight to allow better accuracy. Materials were hand mixed for 1 minute. Filled impressions were placed in an electric centrifuge[10,21] (Megafuge, 1.0 R, Heraeus Instruments, Hanau, Germany), counterbalanced, and centrifuged for 5 minutes to eliminate bubbles as suggested in Paquette et al's article.[21] The poured impressions were placed at room temperature (21°C ± 2°) for 24 hours.

Group M: Two alterations were made to retard the set. The epoxy resin base/activator ratio was equal to 49 g of base to 5.36 g of catalyst; the impressions were poured, centrifuged as in Group S, and placed in a freezer at −5°C for 5 minutes; they were transferred at 5°C and then left at room temperature (21°C ± 2°C) for 24 hours, as suggested by Paquette et al's protocol.[21]

Groups N and P: Following the same procedures in Group M, these two groups were made by altering the base/activator ratio. Group N was made with a ratio of 49 g base to 4.824 g of activator; Group P was made with a ratio of 49 g of base to 6.968 g of activator.

The 40 dies were measured with a digital micrometer (Model 293, Mitutoyo, Tokyo, Japan) to the nearest micrometer (accuracy of 0.1 μm). A single operator made all the measurements. Epoxy resin dies were only identified by the code number. Individual dies were measured at three separate locations. Each die was described by the mean of these three measurements. The metal master die was measured in the same way with a total of 30 separate measurements. This was to accurately estimate its length and to assess measurement repeatability. Paquette's protocol was kept unchanged so as to have the epoxy resin material as the only variable.

Statistical analysis

Die material means and their associated standard errors were calculated. The Bartlett test was performed to assess the homogeneity of variances between die materials and dimensions ($p < 0.05$). Analysis of variance (ANOVA) was performed to determine whether the die materials differed in size, for each dimension, A, B, and C ($p < 0.05$). Differences were analyzed using Tukey's multiple comparisons to determine which die materials differed from the others ($p < 0.05$). The Kruskal–Wallis rank sum test was performed to assess whether the shrinkage of each die material differed between sides A, B, and C.

Results

The mean length of sides A, B, and C on the master die were 20.207 mm, 20.233 mm, and 20.201 mm, respectively, with standard errors of 0.026 mm, 0.029 mm, and 0.017 mm, respectively (n = 10). Mean die lengths and their associated

Table 1 Mean values (mm) and standard errors for each dimension and resin

	S		M		N		P	
Sides	Mean	Standard deviation	Mean	Standard deviation	Mean	Standard deviation	Mean	Standard deviation
A	20.087	0.03	20.017	0.03	20.048	0.04	20.120	0.03
B	20.087	0.02	20.048	0.04	20.045	0.02	20.123	0.03
C	20.091	0.02	20.062	0.02	20.054	0.03	20.119	0.03

standard errors of sides A, B, and C for resin groups S, M, N, and P are illustrated in Table 1. Figures 4–6 show the plot of the die length for resin groups S, M, N, and P and the master die of sides A, B, and C.

The Bartlett test revealed the homogeneity of variances between the resin groups S, M, N, and P and sides A, B, and C.

One-way ANOVA revealed significant differences among the epoxy resin die materials for each side A ($p < 0.0001$), B ($p < 0.0001$), and C ($p < 0.0001$).

Tukey's multiple comparisons test ($p < 0.05$) revealed that for each side (A, B, and C) none of the resin groups was similar to the metal master die. Moreover, the S group was statistically similar to group N and P for side A, to group M and P for side B, and to group M for side C. For side C in particular, the P group was statistically closer to the master die than the S group. Other details from Tukey's multiple comparisons test can be found in the captions of Figures 4–6. The percentage dimensional changes (shrinkage) of the epoxy resin groups are listed in Table 2.

The Kruskal–Wallis rank sum test revealed that, for resin groups S, M, and N, the shrinkage is statistically different between sides A, B, and C ($p < 0.01$ for group S, $p < 0.01$ for group M, $p < 0.05$ for group N). For group P, the shrinkage is similar, but not significant ($p = 0.056$).

Discussion

Construction of a correctly fitting dental prosthesis, whether on a master cast or in a clinical situation, can be significantly affected by the impression technique, the accurate production of a master cast, the wax pattern, the dimensional change of the investment, and the metal casting alloy shrinkage. Study of the relevant literature highlights the importance of a precise master cast.[1-3] To eliminate any discrepancies in fit, it is essential to work on a master cast that reproduces as accurately as possible the exact positions of dental or implant abutments in the patient's mouth. The type and property of die materials, such as dimensional accuracy, detail reproduction, abrasion resistance, and hardness, have been investigated in the dental literature.[6,8,10-13,17] The purpose of this investigation was to study the consequence of polymerization shrinkage of epoxy die materials.

The shrinkage can affect the dimension of the master cast, thus endangering the fit of the framework. Previous studies evaluated whether retarding the set, and changing the ratio between the base and the activator, could modify the shrinkage rate.[21] The contraction of four groups of master dies obtained

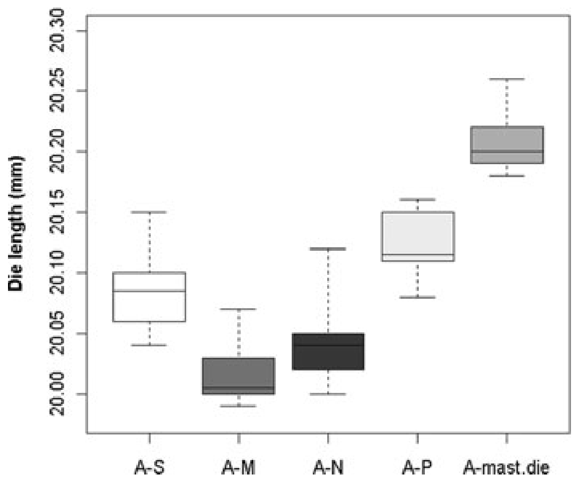

Figure 4 Plot comparing the die length of side A, for resins S, M, N, P, and the master die. Top and bottom of boxes show 75th and 25th percentiles. Top and bottom of graph depict maximum and minimum values. The horizontal line inside the boxes is the median value. Tukey's multiple comparisons test ($p < 0.05$) revealed that resin M is statistically similar to resin N, resin S to resin N, and resin S to resin P.

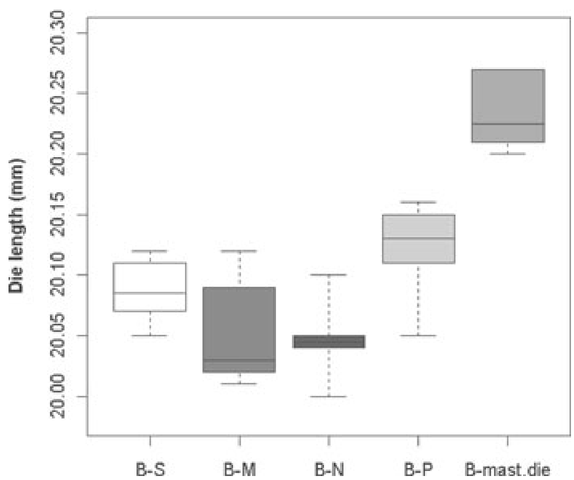

Figure 5 Plot comparing the die length of side B, for resins S, M, N, P, and the master die. Tukey's multiple comparisons test ($p < 0.05$) revealed that resin M is statistically similar to resin N, resin S to resin M, and resin S to resin P.

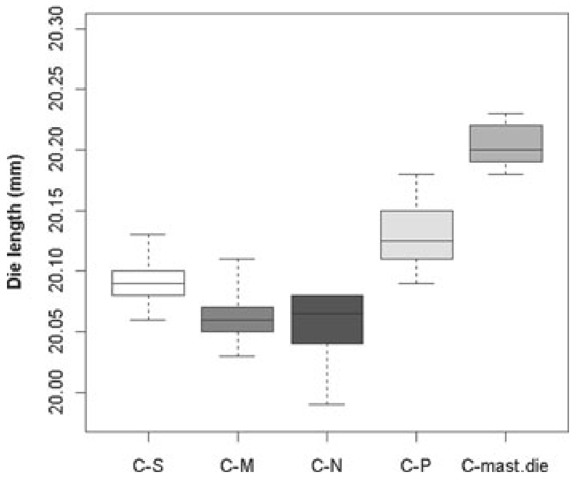

Figure 6 Plot comparing the die length of side C, for resins S, M, N, P, and the master die. Tukey's multiple comparisons test ($p < 0.05$) revealed that resin M is statistically similar to resin N, and resin S to resin M.

with the same epoxy die material was investigated: in group S, a standard base activator ratio was used; in groups M, N, and P, altered manufacturer's instructions were used, retarding and manipulating the setting material. The four groups of the epoxy resin die material contracted in varying degrees, all different from the stainless steel master die on each side. Side A and side B were equivalent for groups S and P; side C of group S contracted more than the same side of group P. Group P had the smallest dimensional change; this result can be considered statistically homogeneous between sides A, B, and C, even though the significance is minor.

The contraction across the three measured sides suggested that epoxy resin die materials might produce inaccurate master casts. In addition, the results regarding this type of epoxy resin are not consistent with Paquette et al's article,[21] which showed that it was possible to reduce the shrinkage by manipulating the manufacturer's instructions. The use of Paquette et al's technique is recommended with caution, and a preliminary test of the used epoxy resin die materials is advisable when possible.

The shrinkage variability of an epoxy resin might be an important clinical disadvantage. The clinical success and relative fit of a cast restoration depend on die materials that have to be precise and strong, have predictable setting, good detail reproduction, and are abrasion resistant. In laboratory procedures for fixed prosthodontics, unpredictable setting characteristics of die materials cannot be compensated for during other steps of the construction process, such as wax contraction, investment material expansion, and metal contraction. This will determine the inaccuracy in transferring the spatial position of teeth or implants from the oral cavity to the master cast: the laboratory technician may fabricate a restoration that requires more corrective procedures during the clinical phase by the restoring dentist. This may be particularly true in implant prosthodontics: the ability of the resin material to correctly reproduce distances is a key factor if a passively fitting structure has to be realized on multiple implants. Gypsum products that meet ADA specification No. 25[5] are still the most popular materials in dentistry for constructing master casts for fixed prostheses. In this study only horizontal measurements were made; oblique dimensional changes from the metal master could not be detected. We should also note that in this study we used an electric centrifuge to mix the resin material: this was related to the necessity to compare this study with other previous investigations where the step of centrifuging the epoxy resin was reported.[10,14,21] However, it is doubtful that dental laboratories and/or clinicians would use this type of material or equipment. Further controlled trials are needed to investigate the clinical significance of these findings; different brands of epoxy resins should also be evaluated, since they may be different from each other, due to the chemical structure or the percentage of chemical ingredients.

Table 2 The percentage dimensional changes (shrinkage) of the resins for each dimension

Sides	S	M	N	P
A	0.59	0.94	0.79	0.43
B	0.72	0.91	0.93	0.54
C	0.54	0.69	0.73	0.36

Conclusions

From this in vitro study the following conclusions may be drawn:

(1) Retarding the setting time and changing the manufacturer's instructions did not reduce contraction of the tested epoxy resin die material.
(2) When the manufacturer's instructions were manipulated, modified resin groups with retarded setting time contracted more.
(3) Group P epoxy resin die material showed the smallest dimensional change that might be considered statistically homogeneous between sides A, B, and C.

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References

1. Vigolo P, Millstein PL: Evaluation of master cast techniques for multiple abutment implant prostheses. Int J Oral Maxillofac Implants 1993;8:439-446
2. Dilts WE, Podshadley AG, Sawyer HF, et al: Accuracy of four removable die techniques. J Prosthet Dent 1971;83:1081-1085
3. Myers M, Hembree JH Jr: Relative accuracy of four removable die systems. J Prosthet Dent 1982;48:163–165
4. Anusavice KJ: Phillips' Science of Dental Materials (ed 10). Philadelphia, PA, Saunders, 1996, p. 186
5. American National Standards Institute/American Dental Association: Specification No. 25 for Dental Gypsum products, 2000

6. Martignoni M, Schonenberger A: Precision Fixed Prosthodontics: Clinical and Laboratory Aspects. Chicago, IL, Quintessence, 1990, pp. 167-222
7. Heshmati RH, Nagy W, Wirth CG, et al: Delayed linear expansion of improved dental stone. J Prosthet Dent 2002;88:26-31
8. Duke P, Moore BK, Haug S, et al: Study of the physical properties of type IV gypsum, resin-containing, and epoxy die materials. J Prosthet Dent 2000;83:466-473
9. Kenyon BJ, Hagge MS, Leknius C, et al: Dimensional accuracy of 7 die materials. J Prosthodont 2005;14:25-31
10. Derrien G, Le Menn G: Evaluation of detail reproduction for three die materials by using scanning electron microscopy and two-dimensional profilometry. J Prosthet Dent 1995;74:1-7
11. Derrien G, Sturzt G: Comparison of transverse strength and dimensional variations between die stone, die epoxy resin, and die polyurethane resin. J Prosthet Dent 1995;74:569-574
12. Schaffer H, Dumfahrt H, Gausch K: Distance alterations of dies in sagittal direction in dependence of the die material. J Prosthet Dent 1989;61:684-688
13. Yaman P, Brandau HE: Comparison of three epoxy die materials. J Prosthet Dent 1986;55:328-331
14. Moser JB, Stone DG, Willoughby GM: Properties and characteristics of a resin die material. J Prosthet Dent 1975;34:297-304
15. Vermilyea SG, Huget EF, Wiskoski J: Evaluation of resin die materials. J Prosthet Dent 1979;42:304-307
16. Nomura GT, Reisbick MH, Preston JD: An investigation of epoxy resin dies. J Prosthet Dent 1980;44:45-50
17. Aiach D, Malone WF, Sandrick J: Dimensional accuracy of epoxy resins and their compatibility with impression materials. J Prosthet Dent 1984;52:500-504
18. Bailey JH, Donovan TE, Preston JD: The dimensional accuracy of improved dental stone, silverplated, and epoxy resin die materials. J Prosthet Dent 1988;59:307-310
19. Chaffee NR, Bailey JH, Sherrard DJ: Dimensional accuracy of improved dental stone and epoxy resin die materials: Part I. Single die. J Prosthet Dent 1997;77:131-135
20. Chaffee NR, Bailey JH, Sherrard DJ: Dimensional accuracy of improved dental stone and epoxy resin die materials: Part II. Complete arch form. J Prosthet Dent 1997;77:235-238
21. Paquette JM, Taniguchi T, White SN: Dimensional accuracy of an epoxy resin die material using two setting methods. J Prosthet Dent 2000;83:301-305
22. Goll GE: Production of accurately fitting full-arch implant frameworks. Part I: clinical procedures. J Prosthet Dent 1991;66:377-384
23. Hecker DM, Eckert SE: Cyclic loading of implant-supported prostheses: changes in component fit over time. J Prosthet Dent 2003;89:346-351
24. Hsu CC, Millstein PL, Stein RS: A comparative analysis of the accuracy of implant transfer techniques. J Prosthet Dent 1993;69:588-593
25. Aramouni P, Millstein P: A comparison of the accuracy of two removable die systems with intact working casts. Int J Prosthodont 1993;6:533-539
26 American National Standards Institute/American Dental Association: Specification No. 19 for nonaqueous elastomeric dental impression materials. J Am Dent Assoc 1977;94:733-741; addendum 1982:105-686
27. Tjan AHL, Nemetz H, Nguyen LTP, et al: Effect of tray space on the accuracy of monophasic polyvinylsiloxane impressions. J Prosthet Dent 1992;68:19-28
28. Schnell RJ, Phillips RW: Dimensional stability of rubber impressions and certain other factors affecting accuracy. J Am Dent Assoc 1958;57:39-48
29. Ciesco JN, Malone WF, Sandrik JL, et al: Comparison of elastomeric impression materials used in fixed prosthodontics. J Prosthet Dent 1981;45:89-94
30. de Araujo PA, Jorgensen KD: Effect of material bulk and undercuts on the accuracy of impression materials. J Prosthet Dent 1985;54;791-794
31. Eames WB, Sieweke C, Wallace WG, et al: Elastomeric impression materials: effects of bulk on accuracy. J Prosthet Dent 1979;41:304-307
32. Saunders WP, Sharkey SW, Smith GM, et al: Effect of impression tray design and impression technique upon the accuracy of stone casts produced from a putty-wash polyvinyl siloxane impression material. J Dent 1991;19:283-289
33. Bomberg TJ, Goldfogel MH, Hoffman W Jr., et al: Consideration for adhesion of impression materials to impression trays. J Prosthet Dent 1988;60:681-684
34. Assif D, Marshak B, Nissan J: A modified impression technique for implant-supported restoration. J Prosthet Dent 1994;71:589-591
35. Wee AG: Comparison of impression materials for direct multi-implant impressions. J Prosthet Dent 2000;83:323-331
36. Vigolo P, Majzoub Z, Cordioli G: Evaluation of the accuracy of three techniques used for multiple implant abutment impression. J Prosthet Dent 2003;89:186-192
37. Vigolo P, Fonzi F, Majzoub Z, et al: An evaluation of impression techniques for multiple internal connection implant prostheses. J Prosthet Dent 2004;92:470-476
38. Samet N, Shohat M, Livny A, et al: A clinical evaluation of fixed partial denture impressions. J Prosthet Dent 2005;94:112-117