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Analysis of 3D Printed Materials for Permanent Restorations

By Dr. Russell Giordano, DMD

INTRODUCTION

With the change in the ADA definition of a ceramic to include printed materials with at least 50% inorganic filler, numerous materials were promoted for use as permanent full contour restorations. As part of the mission of the Department of Biomaterials to analyze new materials and systems, many of these materials were investigated with respect to mechanical and physical properties. There are several of ADA and ISO standards that apply to conventional denture materials and polymer containing dental restorations. However, none yet specifically address printed restorations. Efforts are currently underway to revise these standards to include CAD/CAM fabricated restorations and prosthetic devices. In leu of this, the most relevant standards were used as part of the analysis of properties of these materials.





FILLER CONTENT DETERMINATION

In order to meet the revised ADA definition for ceramic restorations, printed materials for permanent restorations should have an inorganic filler content of 50% or more. A simple ash method (polymer component was burned off) was used to determine filler content of several printed full contour restorative materials. The polymer component was burned off, leaving the inorganic filler behind, Figure 1.



Figure 1. Weight % of Inorganic Content Determined by Ash Method



FLEXURAL STRENGTH TESTING

Flexural strength is one property that the dental community often uses as a criterion for material selection. While this might be important, other properties such as fracture toughness, elastic modulus, material wear, and color stability all factor into a comprehensive evaluation of new materials for clinical selection.



THREE-POINT BEND TEST

In this study, two different methods determining flexural strength were used. One followed ISO 4049 "Dentistry – Polymer-based restorative materials" to measure three-point flexural strength that involves testing bars of materials. Another used biaxial flexure strength that uses discs of materials. The biaxial test usually gives higher values but also is generally easier to conduct, and many manufacturers reference biaxial strength of their materials. However, there currently is no biaxial strength standard for polymer-containing materials; therefore, ISO 6872 on Dental Ceramics was used as a guideline for the biaxial flexure strength specimens.

BIAXIAL FLEXURE STRENGTH TEST

A three-point bend test following ISO 4049 methodology was used. Bars were printed for each test material per manufacturer's recommendations. Parts were washed using isopropyl alcohol and cured using an Otoflash system under Nitrogen. The bars were tested on an Instron 5566A at a crosshead speed of 1.0 mm/min. Load at break was used to calculate flexural strength in MPa. A clinically successful composite resin block material was included for comparison in the strength tests. Results are shown in Figure 2.





Figure 2. Three Point Bend Strength (MPa)

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A biaxial flexure strength test following ISO 6872 methodology was used. Discs were printed for each test material per manufacturer's recommendations. Parts were washed using isopropyl alcohol and cured using an Otoflash system under Nitrogen. The discs were tested on an Instron 5566A at a crosshead speed of 1.0 mm/min. Load at break was used to calculate flexural strength in MPa. Results are shown in Figure 3.



Figure 3. Biaxial Flexure Strength (MPa)



FLEXURAL MODULUS

Flexural Modulus was determined from the three-point bend test. Modulus values are important in evaluating a material's resistance to deformation under stresses developed during clinical use. Several years ago, a polymer-based material with a low modulus of about 2 GPa was widely used for crowns; failure rates were very high due to excessive wear sensitivity and debonding. Thus, we have some idea of what might be problematic with respect to modulus values. These values are presented in Figure 4.





FRACTURE TOUGHNESS

While strength is often used as the primary selection criterion for restorative materials, fracture toughness may be more important in correlating with clinical success. Fracture toughness is a measure of a material's resistance to crack propagation. Ideally, we would like restorative materials to be able to sustain damage and remain intact and function even after the damage has been sustained. This is one advantage of materials like 3Y mol% zirconia. Fracture toughness was determined using a single edge notched beam on materials fabricated according to the manufacturer's recommendations. Methodology for the polymer-based materials used ISO 20795 "Denture base materials" as ISO 4049 does not include fracture toughness testing. E.max machinable ceramic was included for comparison with fracture toughness determined as described in ISO 6872 shown in Figure 5.





Figure 5. Fracture Toughness Using Single Edge Notched Beam (MP.m^{0.5})



WEAR TESTING

METHODOLOGY

Rapid wear of restorative materials may create numerous clinical problems, as proper occlusion that was created with the restoration may rapidly change due to excessive wear of the material. Although this is an important property, there are no standardized wear tests. There are very complex chewing machines as well as more basic wear tests using a pin on disc two or three body wear device. One of the most widely used methods follows one developed at the University of Alabama-Birmingham. Our laboratory fabricated a wear device based on this design and it has been used to determine wear rates for a variety of materials and even was accepted by the FDA to support wear kindness claims about fine particle veneering porcelains.

TEST PROCEDURE AND RESULTS

In this wear test, a pin of the test material is fabricated and then this is run against a plate of Vita MKII that has been shown clinically and in lab testing to have similar wear properties as tooth enamel. Plates of the tooth analog material are on the bottom of the device and pins of the test material are on the top and weighted. A continuous flow of water is used to remove wear debris. Weight and height loss are measured for the pin. The pins are bonded to metal rods that are weighted with a 400-gm load. The pins are moved on a 39 mm path across the plates. A linear wear rate was determined for the test materials and weight and height loss per million cycles. Conventional composite resin as well as denture teeth were used for comparison. Height and weight loss per million cycles are shown in Figures 6 and 7.

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Figure 7. Wear Test: Weight Loss



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COLOR STABILITY

Initial tests on color stability were performed following an initial ISO standard (ISO 7491) on color change. Specimens were printed as discs 20 mm x 2 mm thick according to manufacturer's recommendations. These were polished using a Buehler Ecomet polisher in sequence finishing with a 1-micron diamond paste. Materials were subjected to toluidine blue dye and UV light. The Lab^{*} values and the change in ΔE values were determined before and after treatment using an X-Rite I7 spectrophotometer. Specimens were also stored in de-ionized water and in the dark (control group) for 7 days. Clinically, color differences are seen by most people when there is a difference of 2 to 4 ΔE (Figure 8).

Figure 8. Color Change of Test Materials Post Treatment Color Stability Control set **More Color Change** Water T.B DYE ISO Standard 7491 🔲 UV liaht · Blue Dve Ultra Violet Light Delta-E • 37° C Water Storage Perceptibility 3 37° C Dry, Dark Control Change in L*a*b* color n Spectrophotometer Rodin Rodin Rodin Teliocad Flexcera Sculpture 1.0 Sculpture 2.0 Titan

CONCLUSION

In summation, many materials do not meet the ADA definition for a ceramic due to low filler content. Overall, the material properties of several of the printed materials compare favorably with machinable composite resins and conventional ceramics with respect to mechanical and physical properties.





ABOUT DR. GIORDANO



Dr. Russell Giordano, DMD received specialty training in prosthodontics at Harvard School of Dental Medicine and performed research at the Ceramics Processing Research Laboratory at the Massachusetts Institute of Technology leading to a D.M.Sc. degree in 1991 and a Certificate in Prosthodontics. Dr. Giordano's research involved novel ceramics processing techniques as well as analysis of stress developed during finishing procedures. His research paper won the Arthur R. Frechette Award presented by International Association of Dental Research Prosthodontic Group. Before being appointed at Boston University, Dr. Giordano was an Instructor at Harvard in the Department of Prosthodontics where he served as Associate Director of Complete Denture Prosthodontics and Course Director of Dental Materials.

Giordano presently has several private and federally funded projects. A major research focus is ceramics and ceramic matrix composites. Projects include the testing of current ceramic restorative systems as well as the development of ceramic matrix composites with improved resistance to fracture and higher toughness. Evaluation of new dental materials systems is also an ongoing part of his research activity. Evaluation of the effects of surface finish on strength of ceramics has involved the application of novel machining systems such as the CEREC CAD-CAM system and the Celay copy milling system and 3d printing systems and materials as well as the effects of polishing, fine grinding, glazing and etching.



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