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Original

Radiopacity of conventional, resin-modified glass ionomer, and resin-based luting materials

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Abstract: **The purpose of the present study was to evaluate the radiopacity of currently available dental luting materials. Five conventional cements, six resin-modified glass ionomers (RMGIs), two methyl methacrylate (MMA)-based acrylic resins (eight shades), and nine composite luting materials were evaluated. Radiographs of the specimens were taken together with tooth slices and aluminum step wedges. The density of the specimens was determined with a densitometer and was expressed in terms of the equivalent thickness of aluminum per 2.0-mm unit thickness of specimen. The radiopacity values for human enamel and dentin were 4.3 and 2.3 mm Al/2.0 mm specimen, respectively. The values for materials ranged from 5.1 to 12.9 for conventional luting materials, from 3.4 to 6.3 for RMGIs, from less than 0.5 to 7.3 for MMA resins, and from 2.3 to 9.9 for the composite luting materials. A zinc phosphate cement showed the highest value (12.9), whereas five shades of MMA resin resulted in the lowest value (less than 0.5). Two RMGIs and three composite luting materials exhibited radiopacity values between those of enamel (4.3) and dentin (2.3). It can be concluded that the radiopacity value of luting materials varies considerably, and that care must be taken when selecting luting materials, considering the material composition of restorations. (J Oral Sci 51, 223-230, 2009)**

Keywords: aluminum; cement; densitometer; luting material; radiopacity.

Introduction

Dental luting materials are used for cementing restorations and fixed partial dentures to abutments and cavity preparations. In addition, they are sometimes substituted for base, core foundation, and transitional restorative materials. Radiopacity is one of the prerequisites for luting materials especially when they are applied for seating ceramic restorations and indirect composite restoratives. The advantages of radiopaque over radiolucent materials are easy detection of recurrent dental caries as well as observation of the radiographic interface between the materials and tooth substrates (1).

A number of studies focusing on the optimal radiopacity of dental materials have been reported. Prevost et al. (2) stated that application of materials less radiopaque than dentin should be avoided as bases or liners. Goshima and Goshima (3) reported that luting material should have a minimal radiopacity at least equal to the same thickness of aluminum, to help in accurate radiologic discrimination. Application of semi-radiopaque restorations with radiopacity slightly exceeding that of enamel has also been recommended (1). El-Mowafy and Benmergui (4) concluded that materials having radiopacity values greater than or equivalent to the radiopacity of enamel are suitable for use as inlay cements. In relation to these papers, the ISO 4049:2000(E) document (5) claims that the radiopacity of a radiopaque luting material should be equal to or greater than that of the same thickness of aluminum.

A previous report has indicated that the radiopacity of a glass ionomer luting cement is equivalent to that of enamel (2.2 mm Al/1 mm cement) (6). Prevost et al. (2) reported that the radiopacity of zinc phosphate cement was

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far greater than that of enamel, whereas that of glass ionomers was less than that of dentin. The radiopacity of eight zinc phosphates, seven polycarboxylates, and two glass ionomers exceeded that of enamel, whereas the radiopacity of a glass ionomer was inferior to that of dentin (7). Skartveit and Halse (8) also reported that glass ionomers had insufficient radiopacity. In the 1990s, the radiopacity of indirect composites was higher than that of their accompanying luting cements (9). Since then, the radiopacity characteristic of luting composites has improved (4).

A growing number of luting materials categorized as acrylic resins as well as resin-modified glass ionomers have recently been introduced because of their improved bonding characteristics. However, only limited information is available about the radiographic properties of currently available luting materials (10-12). In this study we determined the radiopacity values of various luting materials, and compared them with those of enamel and dentin, in order to improve the diagnostic accuracy of dental radiographs taken together with existing luting materials.

Materials and Methods

Five conventional cements, six resin-modified glass ionomers (RMGIs), two methyl methacrylate (MMA)-based acrylic resins (with three and five shades respectively), and nine composite luting materials were used. Information on the materials is summarized in Table 1. Step wedges made of 99.99% aluminum (2.0-20.0 mm in thickness, Seico Inc., Hiroshima, Japan) as well as extracted human teeth were also employed. This experiment was conducted with the approval of the Ethics Committee of Nihon University School of Dentistry (approval No.: 2007-5).

Monomer liquid, initiator, if available, and powdered MMA resins were mixed, poured into acrylic molds (10.0 mm in diameter by 2.3 mm height) and cured between two glass plates at 25°C. Other products were spatulated or cured in accordance with the manufacturers' instructions, and 2.3-mm-thick specimens were prepared. After 24 h, all the specimens were ground with #600 silicon-carbide paper to obtain 2.0-mm-thick specimens. Extracted human molars were sectioned mesiodistally with a rotary cutting machine (Isomet, Buehler, Lake Bluff, IL, USA). Slices were wet-polished, and 2.0-mm-thick specimens were prepared. Each specimen was placed together with tooth slices and aluminum step wedges on an occlusal radiographic film (Ultra-Speed Dental Film DF-50 Occlusal, Eastman Kodak Co., Rochester, NY, USA). Two radiographs were taken with a dental X-ray source (DFW-20, Asahi Roentgen Ind., Kyoto, Japan) using exposure factors of 0.6 s at 60 kVp, 15 mA with a target-film distance of 35 cm. The total filtration on the X-ray beam was 2 mm of aluminum. The films were processed in an automatic developing machine (Dent-X 9000, AFP Imaging Co., Elmsford, NY, USA) under normal conditions, i.e., 27°C for 6 minutes.

The radiographic density of the films was measured with a transmission densitometer (PDA-15, Konika-Minolta Inc., Tokyo, Japan). The radiopacity values of the specimens were expressed in terms of the equivalent thickness of aluminum per 2.0 mm unit thickness of material. For each condition, the mean value and standard deviation of ten (luting materials) or 20 (molars) replications were calculated. One reading value was equivalent to the average value obtained from five points within an identical disk. Kolmogorov-Smirnov test was performed for evaluation of the distribution for each of the categories. The radiopacity value of each material was thereafter compared with that of enamel or dentin by Steel's comparison, setting the value of statistical significance at $P = 0.01$ (Kyplot 4.0, KyensLab, Tokyo, Japan). The radiopacity value of either enamel or dentin was considered as the control value.

Results

The radiopacity values of the 2.0-mm-thick specimens, enamel and dentin are presented in Table 2. Results of

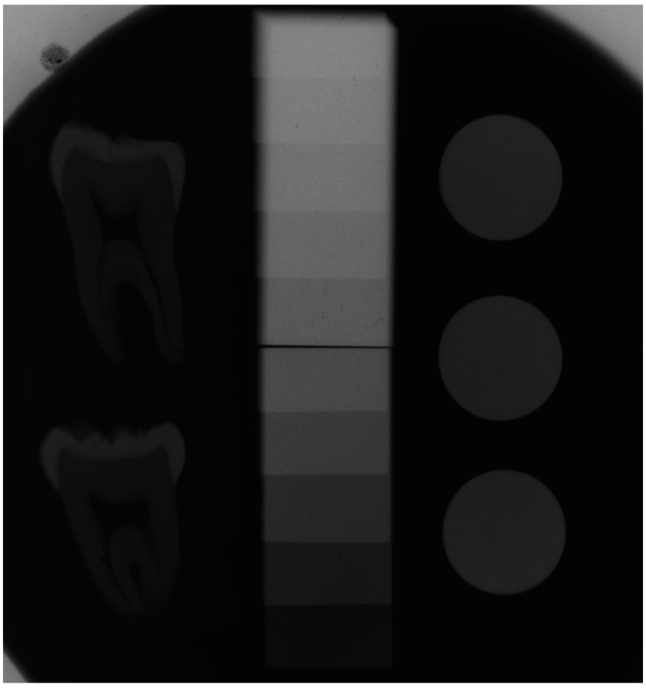

Fig. 1 Radiograph of the Linkmax luting material taken together with molar slices and aluminum step wedges.

statistical analysis are summarized in Tables 3 and 4. Fig. 1 shows a typical radiograph taken together with molar slices and aluminum step wedges. Five shades of two MMA resins showed radiopacity values of less than 0.5. These were judged as radiolucent materials and excluded from statistical analysis. Kolmogorov-Smirnov test run on the radiopacity data did not reveal a normal distribution for each of the categories. Therefore the radiopacity data

Table 1 Materials assessed

Material	Manufacturer	Lot	Composition
Conventional cements			
HY-Bond Carboxylate Cement	Shofu Inc., Kyoto, Japan	1006	P: ZnO, MgO, tannin-fluoride preparation L: copolymer of acrylic and tricarboxylic acids, tartaric acid, distilled water
HY-Bond Zinc Oxide Eugenol Cement	Shofu Inc.	P010669 L070670	P: ZnO, rosin, zinc acetate, aluminum silicate, tannin-fluoride preparation L: eugenol liquid
HY-Bond Zinc Phosphate Cement	Shofu Inc.	P070659 L070686	P: ZnO, MgO, tannin-fluoride preparation L: phosphoric acid, $Al(OH)_3$, ZnO, distilled water
Ketec Cem Easy Mix	3M ESPE, St. Paul, MN, USA	256551	P: fluoro-alumino silicate glass, copolymer of acrylic and maleic acids L: distilled water, tartaric acid
Ketec Cem Radiopaque	3M ESPE	40614	P: fluoro-alumino silicate glass, copolymer of acrylic and maleic acids L: distilled water, tartaric acid
Resin-modified glass ionomer (RMGI) luting materials			
Fuji Bond LC	GC Corp., Tokyo, Japan	502041	P: fluoro-alumino silicate glass L: polyacrylic acid, HEMA, proprietary ingredient, 2,2,4-trimethyl hexamethylene dicarbamate, TEGDMA, distilled water
G-Cem	GC Corp.	61221	P: fluoro-alumino silicate glass, initiator, pigment L: UDMA, dimethacrylate, 4-MET distilled water, phosphoric acid ester monomer, SiO_2, initiator, inhibitor
HY-Bond Resiglass	Shofu Inc.	1106	P: fluoro-alumino silicate glass, tannin-fluoride preparation L: polyacrylic acid, HEMA, distilled water
Ionotite F	Tokuyama Dental Corp., Tokyo, Japan	K34106	P: calcium-alumino silicate glass, SiO_2, BPO L: HEMA, UDMA, MTU-6, phosphate monomer
RelyX Luting Cement	3M ESPE	2006919	P: silane treated glass, potassium persulfate L: distilled water, copolymer of acrylic and itaconic acids, HEMA
Xeno Cem Plus	Dentsply Sankin Industry Co. Ltd., Japan	415-030	P: fluoro-alumino silicate glass, BPO L: UDMA, HEMA, polyacrylic acid
MMA based luting materials			
M-Bond	Tokuyama Dental Corp.		
Clear		517Y6	P: PMMA, BPO
Ivory		41586	P: PMMA, BPO
Dentin		204Y6	P: PMMA, BPO
Opaque Dentin		302286	P: PMMA, BPO, ZrO_2
Opaque Ivory		103B04	P: PMMA, BPO, ZrO_2
		517Y6	L: MMA, MAC-10, amine
Super Bond C&B	Sun Medical Co. Ltd., Moriyama, Japan		
Clear		MR11	P: PMMA
Teeth Color		MS1	P: PMMA, TiO_2
Radiopaque		MR1	P: PMMA, TiO_2, ZrO_2
		MR11	L: MMA, 4-META
Catalyst		ML41	C: TBB, TBB-O, hydrocarbon

Table 1 Materials assessed

Composite luting materials			
Bistite II	Tokuyama Dental Corp.	73306	A: silica-zirconia, NPGDMA, Bis-MPEPP
		73306	B: silica-zirconia, MAC-10, BPO, *dl*-camphorquinone
Chemiace II	Sun Medical Co. Ltd.	MG1	P: ZrO_2, SiO_2, copolymer of methacrylates, aromatic amine, 3-methacryloxypropyl trimethoxysilane
		MK1	L: HEMA, 4-META, diacrylate of polycondensate of bisphenol A glycidyl ether, dimethacrylate of polycondensate of bisphenol A and glycol, TEGDMA
Clearfil Esthetic Cement	Kuraray Medical Inc., Tokyo, Japan	0002AA	A: silanated barium glass, SiO_2, Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, accelerators and others
		0001AA	B: silanated silica, silanated barium glass, SiO_2, Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, *dl*-camphorquinone, initiators, accelerators, pigments and others
Imperva Dual	Shofu Inc.	080612	P: SiO_2, barium sulfate, 5-MSBA fluoro-alumino silicate glass,
		100629	L: UDMA, TEGDMA, BPO, 4-AET, HEMA, photo-initiator
Linkmax	GC Corp.	611152	A: fluoro-alumino silicate glass, UDMA, HEMA, SiO_2, *dl*-camphorquinone, amine
		611152	B: fluoro-alumino silicate glass, UDMA, HEMA, SiO_2, BPO
Maxcem	Sds Kerr Corp., Orange, CA, USA	B448242	A: UDMA, fluoro-alumino silicate glass, *dl*-camphorquinone
		C448242	B: Bis-GMA, TEGDMA, GPDM, fluoro-alumino silicate glass, barium glass, silicate glass
Panavia F 2.0	Kuraray Medical Inc.	4620	A: silanated silica, SiO_2, bisphenol A polyethoxy dimethacrylate, MDP, hydrophobic dimethacrylate, hydrophilic dimethacrylate, BPO, *dl*-camphorquinone
			B: silanated barium glass, silanated titanium oxide, sodium fluoride, SiO_2, bisphenol A polyethoxy dimethacrylate, hydrophobic dimethacrylate, hydrophilic dimethacrylate, *N,N'*-diethanol-*p*-toluidine, sodium 2,4,6-triisopropyl benzene sulfinate
RelyX ARC	3M ESPE	20060807	A: silane treated ceramic, TEGDMA, Bis-GMA
		20060807	B: silane treated silica, functionalized dimethacrylate polymer silane treated ceramic, TEGDMA, Bis-GMA, silane treated silica, functionalized dimethacrylate polymer
Variolink II	Ivoclar Vivadent, Schaan, Liechtenstein	B J13724	A: Ba-Al-F-Si-glass, Bis-GMA, TEGDMA, UDMA, BPO
		C J09824	B: dimethacrylates, inorganic fillers, ytterbium trifluoride, Bis-GMA, BPO, *dl*-camphorquinone

4-AET, 4-acryloxyethyl trimellitate; Bis-GMA, bisphenol A diglycidyl methacrylate; Bis-MPEPP, 2,2-bis[4(methacryloxyethoxy) phenyl] propane; BPO, benzoyl peroxide; GPDM, glycerol dimethacrylate dihydrogen phosphate; HEMA, 2-hydroxyethyl methacrylate; MAC-10, 11-(methacryloyloxy) 1,1-undecane dicarboxylic acid; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; 4-MET, 4-methacryloyloxyethyl trimellitate; 4-META, 4-methacryloxyethyl trimellitate anhydride; MTU-6, 6-methacryloyloxyhexyl 2-thiouracil-5-carboxylate; 5-MSBA, 5-mono-substituted barbituric acid; NPGDMA, neopentyl glycol dimethacrylate; PMMA, poly(methyl methacrylate); TBB, tri-*n*-butyl borane; TBB-O, partially oxidized tri-*n*-butyl borane; TEGDMA, triethyleneglycol dimethacrylate; UDMA, urethane dimethacrylate; P, Powder; L, Liquid; A, A paste; B, B paste; C, Catalyst

Table 2 Radiopacity of dental luting materials, enamel, and dentin (mm Al/2 mm specimen)

Material	Median	Mean	SD
Enamel	4.3	4.3	0.2
Dentin	2.4	2.3	0.2
Conventional cements			
Ketec Cem Easy Mix	5.2	5.1	0.2
Ketec Cem Radiopaque	6.1	6.1	0.1
HY-Bond Zinc Oxide Eugenol Cement	8.8	8.7	0.1
HY-Bond Carboxylate Cement	10.5	10.5	0.1
HY-Bond Zinc Phosphate Cement	12.9	12.9	0.1
RMGI luting materials			
Ionotite F	3.4	3.4	0.2
G-Cem	3.6	3.6	0.2
Xeno Cem Plus	4.4	4.5	0.3
HY-Bond Resiglass	4.6	4.6	0.3
RelyX Luting Cement	4.6	4.7	0.2
Fuji Bond LC	6.3	6.3	0.1
MMA based luting materials			
Super Bond C&B Radiopaque	7.0	7.0	0.1
M-Bond Opaque Ivory	7.2	7.2	0.1
M-Bond Opaque Dentin	7.2	7.3	0.2
Composite luting materials			
Panavia F 2.0	2.3	2.3	0.1
Imperva Dual	2.6	2.6	0.1
Clearfil Esthetic Cement	3.5	3.6	0.2
Chemiace II	4.6	4.6	0.2
RelyX ARC	4.6	4.7	0.5
Maxcem	4.8	4.9	0.5
Linkmax	5.5	5.5	0.3
Bistite II	6.2	6.3	0.2
Variolink II	9.9	9.9	0.4

Enamel and dentin, $n = 20$; Luting agents, $n = 10$. Five MMA based materials were radiolucent.

were analyzed by Steel's comparison. The radiopacity values for human enamel and dentin were 4.3 and 2.3 mm Al/2.0 mm specimen, respectively. The values of materials ranged from 5.1 to 12.9 for the conventional luting materials, from 3.4 to 6.3 for the RMGIs, from 7.0 to 7.3 for the three radiopaque MMA resins, and from 2.3 to 9.9 for the composite luting materials. The HY-Bond zinc phosphate cement showed the highest value (12.9) among the materials assessed.

Table 3 compares the significance of differences in radiopacity values between the luting materials and human enamel. Five conventional cements, two RMGIs, three MMA-based materials, and five composite materials were more radiopaque than enamel, whereas two RMGIs and three composite materials were less radiopaque than enamel ($P < 0.01$). The radiopacity values of Xeno Cem Plus and HY-Bond Resiglass RMGIs as well as RelyX ARC composite were not significantly different from that of enamel (4.3, $P > 0.01$). Table 4 shows the significance of differences in radiopacity values between luting materials and human dentin. The radiopacity value of Panavia F 2.0 was not significantly different from that of dentin (2.3, $P = 0.9749$). Other materials were more radiopaque than dentin.

Table 3 Statistical difference between luting material and enamel analyzed by Steel's test

Material	Statistic value	*P*-value
Conventional cements		
Ketec Cem Easy Mix	-4.4	< 0.001
Ketec Cem Radiopaque	-4.4	< 0.001
HY-Bond Zinc Oxide Eugenol Cement	-4.4	< 0.001
HY-Bond Carboxylate Cement	-4.4	< 0.001
HY-Bond Zinc Phosphate Cement	-4.4	< 0.001
RMGI luting materials		
Ionotite F	4.4	< 0.001
G-Cem	4.4	< 0.001
Xeno Cem Plus	-1.0	0.8778*
HY-Bond Resiglass	-2.7	0.0356
RelyX Luting Cement	-3.6	< 0.001
Fuji Bond LC	-4.4	< 0.001
MMA based luting materials		
Super Bond C&B Radiopaque	-4.4	< 0.001
M-Bond Opaque Ivory	-4.4	< 0.001
M-Bond Opaque Dentin	-4.4	< 0.001
Composite luting materials		
Panavia F 2.0	4.4	< 0.001
Imperva Dual	4.4	< 0.001
Clearfil Esthetic Cement	4.4	< 0.001
Chemiace II	-3.5	< 0.01
RelyX ARC	-2.2	0.1957*
Maxcem	-3.3	< 0.01
Linkmax	-4.4	< 0.001
Bistite II	-4.4	< 0.001
Variolink II	-4.4	< 0.001

Radiopacity value of enamel was used as the control. *Not significant ($P > 0.01$).

Discussion

The original definition of cement was hydrated inorganic compounds based on alumina and silica. However, currently available dental luting materials consist of various elements and compounds. Metallic elements usually show high radiopacity and are easily detected on radiographs. Polymers, on the other hand, are substantially radiolucent, and it is difficult to detect them on radiographs. Radiopaque elements or compounds have therefore been added to polymeric and composite materials to make them radiopaque.

Of the materials we assessed, conventional luting materials exhibited high radiopacity values as compared with enamel. In particular, three cements that contain zinc oxide recorded radiopacity values of more than 8.0 mm Al/2.0 mm specimen. Considering that the atomic numbers of aluminum, silicon, and calcium are 13, 14 and 20, respectively, the high radiopacity value of zinc oxide-based cements is probably derived from the considerable content of elemental zinc (atomic number 30). High radiopacity values of zinc oxide-based cements have been reported (7,10,13), and the present results are in accord with this.

The radiopacity value of RMGIs varied from a maximum of 6.3 to a minimum of 3.4. The composition of two conventional glass ionomers and six RMGIs shown in Table 1 indicates that all glass ionomers contain alumino silicate glass. This is the basis of their definition as glass

Table 4 Statistical difference between luting material and dentin analyzed by Steel's test

Material	Statistic value	*P*-value
Conventional cements		
Ketec Cem Easy Mix	-4.5	< 0.001
Ketec Cem Radiopaque	-4.5	< 0.001
HY-Bond Zinc Oxide Eugenol Cement	-4.5	< 0.001
HY-Bond Carboxylate Cement	-4.5	< 0.001
HY-Bond Zinc Phosphate Cement	-4.5	< 0.001
RMGI luting materials		
Ionotite F	-4.5	< 0.001
G-Cem	-4.5	< 0.001
Xeno Cem Plus	-4.5	< 0.001
HY-Bond Resiglass	-4.5	< 0.001
RelyX Luting Cement	-4.5	< 0.001
Fuji Bond LC	-4.5	< 0.001
MMA based luting materials		
Super Bond C&B Radiopaque	-4.5	< 0.001
M-Bond Opaque Ivory	-4.5	< 0.001
M-Bond Opaque Dentin	-4.5	< 0.001
Composite luting materials		
Panavia F 2.0	0.9	0.9749*
Imperva Dual	-3.6	< 0.01
Clearfil Esthetic Cement	-4.5	< 0.001
Chemiace II	-4.5	< 0.001
RelyX ARC	-4.5	< 0.001
Maxcem	-4.5	< 0.001
Linkmax	-4.5	< 0.001
Bistite II	-4.5	< 0.001
Variolink II	-4.5	< 0.001

Radiopacity value of dentin was used as the control. *Not significant ($P > 0.01$).

ionomer materials (ASPA; alumino silicate poly-acrylate). Thus, glass ionomer cements show optical translucency. Unfortunately, however, incorporation of alumino silicate glass alone makes glass ionomer materials radiolucent (2,6,7). The manufacturers currently employ various radiopaque glasses, i.e., barium (atomic number 56) or strontium (atomic number 38) glass, as powder components of glass ionomers (11). The glass ionomer materials evaluated in this study contain alumino silicate with barium or strontium, and the amount of these radiopaque elements may affect the radiopacity value of the material (11).

Eight shades of two MMA-based materials exhibited varied radiopacity. Specifically, three shades of two materials that contained zirconium oxide showed radiopacity values of more than 7.0. However, the other five shades of the two materials were radiolucent, although one material contained titanium oxide. The improved radiopacity value of radiolucent acrylic resins may be derived from the incorporation of oxide of zirconium (atomic number 40). However, incorporation of the oxide of titanium (atomic number 22) did not particularly improve the radiopacity of the resin material. In order for MMA resins to achieve radiopacity, they need to incorporate a considerable amount of high-atomic-number compound(s). As a result, the color of the material will change from transparent to a whitish or metallic color.

Increasing the radiopacity value of a composite material is not very difficult because such materials consist mainly of large proportions of inorganic filler. Radiopaque elements can be added to the filler particles as oxides or other compounds. Among the material ingredients shown in Table 1, zirconia, silica-zirconia, barium glass, barium sulfate, and ytterbium trifluoride are candidates for radiopaque additives. In particular, ytterbium has a large atomic number of 70 and a molecular weight of 173. It is considered that the high radiopacity value of Variolink II composite is attributable to incorporation of ytterbium trifluoride (YbF_3) into the B paste.

As revealed in the present study, a number of resin-based materials showed radiopacity values below that of enamel. These values will be sufficient to allow detection in some cavity preparations. However, the use of low-value radiopaque materials may lead to incorrect diagnosis. Therefore, a radiopacity value equal to or slightly greater than that of enamel is desirable for dental luting materials.

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