Bond strength of artificial teeth to thermoplastic denture base resin for injection molding

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This research was conducted to investigate the bond strength between artificial teeth and a thermoplastic denture base resin for injection molding with different surface preparations for use in flexible resin removable partial dentures. Composite resin denture teeth and acrylic denture resin teeth were bonded to three types of thermoplastic denture base resins for injection molding (polyamide, polyester, and polycarbonate) and a conventional heat-polymerized polymethyl methacrylate (PMMA) resin (control). The ridge lap surfaces of the artificial teeth were classified into four groups based on the type of ridge lap surface treatment applied ($n=10$): no treatment, ethyl acetate, small T-shaped tunnel, and large T-shaped tunnel. The specimens were tested for bond strength. The results showed that the ethyl acetate treatment was ineffective for enhancing the bond strength ($p>0.05$) between the artificial teeth and thermoplastic denture base resin for injection molding, whereas the T-shaped tunnel was quite effective in this regard ($p<0.05$).

Keywords: Thermoplastic resin for injection molding, Artificial teeth, Bond strength, Flexible resin removable partial dentures, Surface preparation

INTRODUCTION

Conventional removable dental prostheses are fabricated using acrylic resin, such as polymethyl methacrylate (PMMA), owing to its favorable handling properties and appropriate physical and aesthetic properties[1)]. Recently, removable partial dentures (RPDs) that avoid the use of metal clasps have become an alternative treatment option for patients disliking the metal clasps of conventional RPDs for aesthetic reasons. In 2014, the Japan Prosthodontics Society (JPS) approved the use of flexible resin RPDs[2,3)]. The denture bases and retentive arms of flexible resin RPDs are fabricated using thermoplastic resins suitable for injection molding, including polyamide, polyester, and polycarbonate. These thermoplastic resins are inherently flexible during insertion and can help easily remove the RPDs from the abutment teeth, in addition to their excellent aesthetic properties owing to the gingival shade color[4-6)].

In RPD treatment, artificial teeth, such as composite resin denture teeth, acrylic resin denture teeth, or porcelain denture teeth, are used to restore the occlusion to the missing teeth along with the denture base resin. Although porcelain denture teeth exhibit superior resistance to wear compared to other artificial teeth, tooth debonding from the denture base is oftentimes observed[7-11)]. In contrast, composite resin denture teeth and acrylic resin denture teeth can chemically bond to conventional PMMA denture resins[7,8,10,11)]. Nevertheless, in most flexible resin RPDs, artificial teeth do not chemically bond to the thermoplastic denture base resin, and therefore can easily detach from the denture base[2,3)].

To minimize the need for repairs, various studies have been conducted on the bond strength of conventional denture base resin to composite resin denture teeth and acrylic resin teeth. Organic solvents, such as ethyl acetate and dichloromethane, have been applied for the micromechanical retention of the ridge lap surface of artificial teeth[10,12-14)]. Ethyl acetate has recently been used for surface preparation, considering the carcinogenic nature of dichloromethane[14)]. Modifying the ridge lap of artificial teeth is effective in improving the mechanical retention with the denture base[15-18)]. In flexible resin RPDs, it is indispensable to provide mechanical retention for artificial teeth to enhance the bond strength between artificial teeth and thermoplastic resins[3)]. In particular, the maxillary incisor artificial teeth are easily affected by loads applied in the labial direction during function[18)]. However, no guidelines have been given for the efficient design of mechanical retentions for the ridge lap range of artificial teeth used in flexible resin RPDs. The use of mechanical retentions, such as vertical or horizontal grooves, can improve the bond strength to the denture base[16,17)]. Therefore, T-shaped tunnels combined with vertical and horizontal grooves (Fig. 1) may produce a more reliable and firm mechanical retention between the artificial teeth and thermoplastic resins. Furthermore, T-shaped tunnels are currently used clinically, and ready-made artificial composite resin teeth with T-shaped channels are also available.

The purpose of this study was to analyze a variety of surface preparations applied to the ridge lap of artificial teeth by determining the bond strength of the maxillary incisor artificial teeth to thermoplastic denture base resins for injection molding. The null hypothesis was that the bond strength of artificial teeth to thermoplastic

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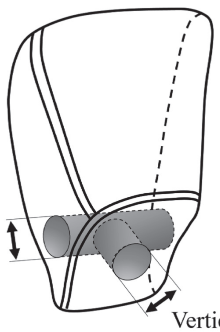

Fig. 1 Diagram of a T-shaped tunnel applied to the ridge lap surface of artificial teeth.

resins for injection molding would not be affected by the type of artificial teeth, type of surface preparation, and types of thermoplastic resin.

MATERIALS AND METHODS

Preparation of the ridge lap of artificial teeth

The composite resin teeth (Endura, Shofu, Kyoto, Japan, Lot; 011801) and acrylic resin teeth (Real Crown, Shofu, Lot; 012801) (AR) were bonded to three types of thermoplastic resins for injection molding and a heat-polymerized conventional PMMA denture base resin as the control (Table 1). One hundred and thirty maxillary central incisor artificial teeth were classified into four

Table 1 Thermoplastic resin for injection-molding used in this study

Constituent	Product	Code	Manufacturer	Processing method	Lot number
Polyamide	Valplast	V	UniVal, Tokyo, Japan	Technique of injection molding; after heat treating at 215°C for 20 min, injection at 1 MPa pressure and cooling for 30 min	170904
Polyester	EstheShot Bright	E	i-Cast, Kyoto, Japan	Technique of injection molding; after heat treating at 280°C for 20 min, injection at 1 MPa pressure and cooling for 30 min	7L2909970
Polycarbonate	Reigning N	R	Toushinyoukou, Niigata, Japan	Technique of injection molding; after heat treating at 320°C for 30 min, injection at 1 MPa pressure and cooling for 30 min	15B2X00011
Polymethyl methacrylate (conventional PMMA)	Acron	A	GC, Tokyo, Japan	Heat polymerized, compression molding technique; molded at 5 MPa pressure, heat treated at 70°C for 90 min, afterwards 100°C for 30 min and cooling for 30 min	Powder; 1801101 Liquid; 1712131

Table 2 Surface treatment groups used in the thermoplastic resins

Materials	Group	Surface treatment
Polyamide (Valpast)	Group 1V	No surface treatment
	Group 2V	Ethyl acetate treatment
	Group 3V	Small T-shaped tunnel (1.6 mm)
	Group 4V	Large T-shaped tunnel (2.1 mm)
Polyester (EstheShot Bright)	Group 1E	No surface treatment
	Group 2E	Ethyl acetate treatment
	Group 3E	Small T-shaped tunnel (1.6 mm)
	Group 4E	Large T-shaped tunnel (2.1 mm)
Polycarbonate (Reigning N)	Group 1R	No surface treatment
	Group 2R	Ethyl acetate treatment
	Group 3R	Small T-shaped tunnel (1.6 mm)
	Group 4R	Large T-shaped tunnel (2.1 mm)
Polymethyl methacrylate (Acron)	Group 1A	No surface treatment

groups, and four kinds of surface preparations applied to the ridge lap area (n=10) (Table 2):

Group 1: No further surface treatment was carried out.

Group 2: Ethyl acetate treatment (Wako Pure Chemical Industries, Ltd., Osaka, Japan, lot DCM0986) was applied on the ridge lap surface for 120 s immediately before injection molding.

Group 3: A small T-shaped tunnel with mesiodistal and vertical holes was made by reshaping the ridge lap with a 1.6-mm-diameter round bur (Maillefer Carbide Bur Round No. 5, DENTSPLY-Sirona, Tokyo, Japan).

Group 4: A large T-shaped tunnel with mesiodistal and vertical holes was made by reshaping the ridge lap with a 2.1-mm-diameter round bur (Maillefer Carbide Bur Round No. 7, DENTSPLY-Sirona).

All artificial teeth in groups 3 and 4 were fixed using a milling machine (Milling Machine F1, Degussa, Frankfurt, Germany), and similar T-shaped tunnels were reshaped by the round bur in the mesiodistal direction and the tooth axis direction. The smallest possible diameter to fill the thermoplastic resin into the T-shaped tunnel is 1.6 mm. The largest possible diameter to reshape within the ridge lap surface region of the artificial teeth with a round bur is 2.1 mm.

Bond strength testing

For evaluating the bond strength of the specimens, 130 wax blocks were fabricated using an adaptation of the Japanese Industrial Standard (JIS) methodology (JIS T 6506:2005)[19]. The artificial teeth were placed on the beveled surface of a rectangular wax block, and the long axis of the artificial tooth was aligned 45° to the basal plane of the rectangular wax block. The mesiodistal surface of the cingulum of the artificial tooth was embedded in 3 mm of wax from the ridge lap surface. For the thermoplastic resins, the specimens were prepared and placed in a metal flask with the injection unit (MIS-

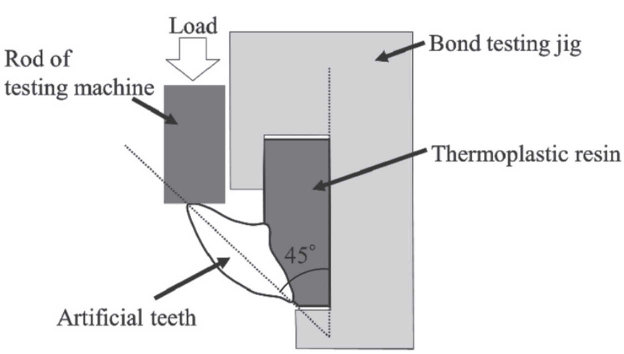

Fig. 2 Cross-sectional diagram of a specimen on a bond testing machine.
The long axis of the artificial tooth is oriented 45° to the basal surface of the wax block.

II, i-CAST, Tokyo, Japan) with gypsum investment (Advastone, GC, Tokyo, Japan), and a heat-polymerized PMMA resin in a conventional metal flask. The denture base resins were fabricated according to their respective manufacturers' instructions (Table 1). Referring to past research, after deflasking, the specimens were soaked in water at 37ºC for 10 days before bond testing[10,12]. The bond strengths in Newtons (N) were measured using a universal testing machine (Autograph AGS-J, Shimadzu, Kyoto, Japan) at 45° from the long axis of the artificial tooth on the palatal plane at a crosshead speed of 1 mm/min until fracture (Fig. 2). The bond strength of the specimens from which the artificial tooth was debonded after deflasking was set to 0 N.

Statistical analysis

The data were statistically processed using a statistical software (IBM SPSS Statistics version 26.0, IBM, NY, USA). All data were analyzed with the Kolmogorov-Smirnov test to evaluate the normality, with the Levene test to evaluate the homoscedasticity, and with analysis of variance (ANOVA) and the Newman-Keuls *post hoc* comparison test to determine significant differences between the artificial teeth, surface preparations, and the thermoplastic resins for injection molding at a significance level of 5%. The data for the heat-polymerized conventional PMMA resin group were excluded from ANOVA to compare the result with only that for the thermoplastic resins for injection molding.

Observation of fracture pattern

For all the specimens after the bond testing, the debonded surface of the ridge lap was studied under a stereo microscope (SZ61, Olympus, Tokyo, Japan) at ×30 magnification. The failure mode was categorized into three modalities: adhesive failure at the thermoplastic resins for injection molding/artificial teeth interface, cohesive failure 1 within the thermoplastic resins for injection molding, and cohesive failure 2 within the artificial teeth.

Scanning electron microscopy analysis

To observe at a magnification of 5,000× the morphological changes on the ridge lap surfaces of the artificial teeth after ethyl acetate treatment, representative scanning electron microscopy (SEM) micrographs were acquired after ion sputter coating with gold using a scanning electron microscope (JSM-6330F; JEOL, Tokyo, Japan) at an accelerating voltage of 5 kV.

RESULTS

The three-way mixed model ANOVA results are shown in Table 3, and Table 4 lists the mean bond strengths of the specimens, standard deviations (S.D.), and failure modes. The data were analyzed as having normality and homoscedasticity. The three-way ANOVA results showed significant differences between the artificial teeth, thermoplastic denture base resins, and surface preparations (p<0.05). There were also differences in

Table 3 Result of three-way ANOVA for bond strength data with artificial teeth (AT), thermoplastic denture base resin (DB) and surface preparation (SP) factors ($p<0.05$)

Source	Sum of squares	Degrees of freedom	Mean squares	F	*p* value
AT	3,974.355	1	3,974.355	105.734	0.000
DB	31,929.638	2	15,964.819	424.729	0.000
SP	674,244.285	3	224,748.095	5,979.210	0.000
AT * DB	80.508	2	40.254	1.071	0.345
AT * SP	8,188.070	3	2,729.357	72.612	0.000
DB * SP	24,623.078	6	4,103.846	109.179	0.000
AT * DB * SP	720.877	6	120.146	3.196	0.005
Error	8,119.064	216	37.588	—	—
Total	1,442,602.167	240	—	—	—

Table 4 Mean and standard deviation values of bond strength (N) and type of failure mode

Denture base resins	Surface treatment	Bond strength (N) Mean±S.D.		Failure mode (number) Cohesive 1/Cohesive 2/Adhesive	
		Composite resin teeth	Acrylic resin teeth	Composite resin teeth	Acrylic resin teeth
Polyamide (Valplast)	Group 1V	0.60±0.21[f]	0.90±0.31[f]	0/0/10	0/0/10
	Group 2V	0.75±0.33[f]	0.98±0.31[f]	0/0/10	0/0/10
	Group 3V	67.1±2.83[d,e]	62.44±3.31[e]	9/1/0	10/0/0
	Group 4V	111.85±8.92[b]	86.41±3.41[c]	4/6/0	4/6/0
Polyester (EstheShot Bright)	Group 1E	2.25±0.93[f]	1.05±0.55[f]	0/0/10	0/0/10
	Group 2E	3.04±0.94[f]	1.86±0.51[f]	0/0/10	0/0/10
	Group 3E	85.24±3.04[c]	74.98±1.32[c,d]	10/0/0	10/0/0
	Group 4E	130.89±7.86[a]	104.44±0.85[b]	4/6/0	4/6/0
Polycarbonate (Reigning N)	Group 1R	5.31±2.67[f]	3.60±1.32[f]	0/0/10	0/0/10
	Group 2R	4.18±1.77[f]	4.62±1.73[f]	0/0/10	0/0/10
	Group 3R	109.25±13.86[b]	114.50±5.86[b]	4/6/0	3/7/ 0
	Group 4R	172.12±8.02	139.17±11.12[a]	1/9/0	0/10/0
PMMA (Acron)	Group 1A	201.82±26.04	184.60±42.80	0/10/0	0/10/0

S.D.: standard deviation.
The same superscripts indicate that the groups were not significant differences ($p>0.05$).

the artificial teeth–surface preparation interaction, thermoplastic denture base resin–surface preparation interaction, and artificial teeth–thermoplastic denture base resin–surface preparation interaction ($p<0.05$). However, there were no differences in the artificial teeth–thermoplastic denture base resin interaction ($p>0.05$). Therefore, the data for the bond strengths were analyzed by one-way ANOVA and the Newman-Keuls *post hoc* comparison test. The one-way ANOVA result differed significantly between each group ($p<0.05$).

For the thermoplastic denture base resins, the artificial teeth in groups 1 and 2 yielded significantly lower bond strength values than those in groups 3 and 4 ($p<0.05$). The bond strength value of the composite resin denture teeth in group 4R was highest (172.12 N), followed by those of the acrylic resin denture teeth in group 4R (139.17 N), composite resin teeth in group 4E (130.89 N), and composite resin teeth in group 4V (111.85 N). Group 4 yielded significantly higher bond strength values than group 3 ($p<0.05$). When two artificial teeth were compared, the composite resin teeth in group 4 exhibited a significantly higher strength than that of acrylic resin teeth ($p<0.05$), although no significant differences existed between groups 1, 2, and

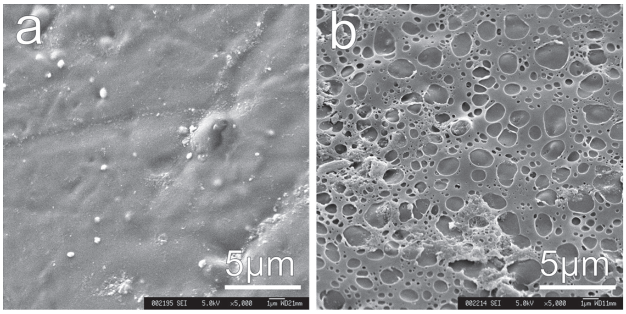

Fig. 3 SEM micrographs at ×5,000 magnification of the ridge lap surface of the composite resin denture teeth: (a) before ethyl acetate treatment and (b) after ethyl acetate treatment.

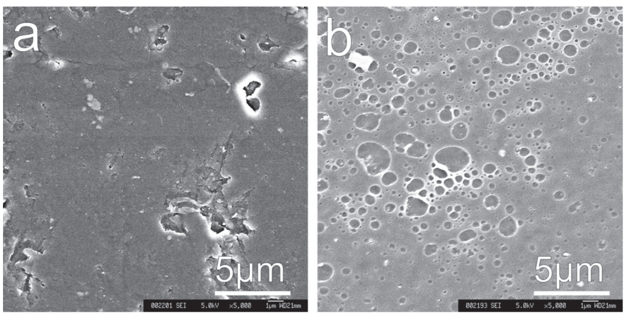

Fig. 4 SEM micrographs at ×5,000 magnification of the ridge lap surface of the acrylic resin denture teeth: (a) before ethyl acetate treatment and (b) after ethyl acetate treatment.

3 (p>0.05). For the conventional PMMA denture base resin, the bond strengths were 184.60 and 201.82 N for each artificial tooth, which were approximately 132 and 117% of those in group 4R, respectively.

For the failure mode, groups 1 and 2 showed complete adhesive failures without group 1A. No adhesive failure was observed in groups 3 and 4. Group 4R showed predominantly cohesive 2 failures, whereas cohesive 1 failure was observed for groups 3V and 3E.

The SEM images of the ridge lap surfaces after ethyl acetate treatment (Figs. 3 and 4) show several small pores in both the composite resin denture teeth and acrylic denture resin teeth.

DISCUSSION

This study was aimed at evaluating the bonding characteristics of artificial teeth to thermoplastic resins for injection molding in terms of the effect of ethyl acetate and T-shaped tunnel preparations. The results showed significant differences in the bond strengths regardless of the dental base resin, artificial teeth materials, or the contact surface treatment method. Therefore, the null hypothesis was rejected.

Surface preparation with ethyl acetate can lead to swelling of the surface of PMMA resins, promoting the formation of interpenetrating polymer networks (IPNs), which help improve the bond strength to PMMA[20-22]. The SEM evaluation revealed a significant modification of the ridge lap surface by the ethyl acetate treatment (Figs. 3 and 4). However, the application of ethyl acetate did not improve the bond strength to a clinically acceptable level, while the conventional PMMA resin showed a high bond strength even with no surface treatment. It is speculated that the methyl methacrylate monomer of the conventional PMMA resin penetrated the ridge lap surface to the artificial teeth and formed an IPN zone, thereby improving the bond strength[20,23]. However, the thermoplastic denture base resins contain polymerized polymer without any monomer. Therefore, during injection molding, it is unlikely that the thermoplastic resin for injection molding formed an IPN layer of the artificial teeth[20,23].

The T-shaped tunnel exhibited a statistically superior bond strength in all the thermoplastic resin types. The combined mechanical retention of vertical and horizontal holes created a strong mechanical bond between the artificial teeth and thermoplastic resins for injection molding, thus dislodging from the gingival cuff of the denture base[16-18]. The first advantage of the T-shaped tunnel is that the tunnels of the artificial teeth become pathways for the thermoplastic resins to fill the connected channels from the ridge lap surface to the mesiodistal area. Consequently, the defects of injection molding rarely occur. The second advantage is that there are three mechanical jointing points between the artificial teeth and the thermoplastic resins, resulting in high bond strength. In this study, there was a significant difference in the diameter (between 1.6 and 2.1 mm). For the bond strength of the artificial teeth prepared with a T-shaped tunnel, the hole diameter must be adequate. If the diameter of the T-shaped tunnel is too small, the bond strength of the artificial teeth may be too low to stress the junction area of the ridge lap of the artificial teeth. The bond strengths of group 4R (Reigning N), group 4V (Valplast), and group 4E (EstheShot Bright) fulfilled the requirement of JIS T 6506, *i.e.*, 110 N[19]. The large T-shaped tunnel (2.1 mm) preparation would also meet the clinical functional requirements with respect to the bond strength to the thermoplastic resins for injection-molding.

In group 4 of the thermoplastic resins for injection molding, the polycarbonate (Reigning N) exhibited a significantly higher bond strength than the other thermoplastic resins (polyamide and polyester). The type of denture base resin affects the bonding ability of artificial teeth to denture base resins[12]. A previous report showed that the ultimate flexural strength of polycarbonate is higher than that of polyamide and polyester[24]. Further, the ultimate flexural strength of polyamide is lower than that of polycarbonate and polyester. Additionally, the elastic modulus and proportional limit of polycarbonate are higher than those of polyamide and polyester. Therefore, polycarbonate is rigid and fairly inflexible, whereas polyamide is soft and highly flexible. The mechanical property of polyester is between that of polycarbonate and polyamide[24]. Furthermore, all specimens were soaked in water for 10 days in this study as it is possible for the mechanical properties to change because of water absorption. Water molecules acting as plasticizers interfere with the entanglement of the polymer chains. Therefore, the molecular structure may be related to water absorption. When this state occurs, polymer chains generally become more mobile, and mechanical properties change[1]. Polyamide, polyester, and polycarbonate absorbs water rapidly for 10 days, and then their water absorption rate decreases. Therefore, the water immersion period of this study was set to 10 days[25].

The bond strength of artificial teeth prepared with a T-shaped tunnel may be influenced by the mechanical properties of the thermoplastic resin. This might indicate that the higher the mechanical strength of the thermoplastic resin, the higher the bond strength of the artificial teeth prepared with a T-shaped tunnel and the lower the percentage of cohesive failure within the thermoplastic denture base resin. Accordingly, in clinically scenarios, it is effective to apply T-shaped connected tunnels with a diameter of 2.1 mm to the artificial teeth of the polycarbonate (Reigning N).

Comparing the composite resin denture teeth with the acrylic resin denture teeth, the composite resin denture teeth in group 4 have a higher bond strength than those of acrylic resin denture teeth. The composite resin teeth have better physical and mechanical properties, such as strength, wear resistance, and surface hardness, than the acrylic resin denture teeth, owing to the inorganic composition in addition to the highly cross-linked structure[26,27]. In the preparation of large T-shaped tunnels (2.1 mm), the ridge lap portion of the artificial teeth should be eliminated. In summary, the mechanical properties of the artificial teeth affect the values of bond strength of artificial teeth prepared with large T-shaped tunnels.

During the preparation of a large T-shaped tunnel with a diameter of 2.1 mm, a significant portion of the artificial teeth material is withdrawn, thus reducing the mechanical strength of the teeth themselves. Therefore, the bond strength in these cases is determined mainly by the artificial teeth strength. This is supported by the observations of the cohesive type 2 breaking mode within the teeth (Table 4).

Polycarbonate (Reigning N) and artificial teeth made of polycarbonate resin can be thermally welded owing to its high glass transition temperature[3]. However, in this study, the bond strength of polycarbonate (Reigning N) without surface preparation did not meet the requirement of JIS T 6506:2005[19]. Irrespective of the type of thermoplastic resin for injection molding of flexible resin RPDs, it is indispensable to prepare the mechanical retention of the artificial teeth to bond the artificial teeth and thermoplastic resin for the regular clinical use of flexible resin RPDs. The preparation of T-shaped tunnels for artificial teeth requires a design that takes adequate clearance with opposing teeth in the missing area. In patients with inadequate clearance, the flexible resin RPDs may cause problems such as loss of tooth, tooth cracking, or tooth fracture. For clinical applications of flexible resin RPDs, it is important to improve the clinical design of flexible resin RPDs by further conducting basic and clinical studies. Furthermore, clinically, when artificial teeth need to be ground in cases where there is a narrow denture space, it is difficult to apply the T-shaped tunnel. In addition, when the anterior artificial teeth are arranged tightly, it may be difficult to fill thermoplastic resin into the T-shaped tunnel of the artificial teeth.

CONCLUSIONS

The following conclusions can be drawn from this study:

1. Pre-preparing the ridge lap surface with ethyl acetate is ineffective for improving the bond strength between artificial teeth and thermoplastic resins for injection molding.
2. T-shaped tunnels may be an effective mechanical retention design for achieving a good bond strength value of artificial teeth to thermoplastic resins for injection molding.

CONFLICT OF INTEREST

The authors have no conflict of interest to declare.

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