The influence of temperature on sheet lamination process when fabricating mouthguard on dental thermoforming machine

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Abstract: Laminated custom-made mouthguards tend to delaminate with use; this is a problem in clinical use. Insufficient bonding strength causes delamination, and bonding strength is strongly affected by heating temperature during lamination. The present study aimed to evaluate the influence of heating temperature on the sheet lamination process. Seven mouthguard sheet products were laminated together at different heating temperatures. To evaluate the bonding strength, a delamination test (n = 6) was performed, and the fracture patterns were inspected visually. To evaluate the shock absorption capability, a falling impact test (n = 5) was performed, and the specimen thicknesses were measured. All recorded values were analyzed using two-way analysis of variance and Tukey’s Honest Significant Difference Test (P < 0.05). The present study confirmed that bonding strength was dependent on heating temperature: In ethylene-vinyl acetate (EV A) copolymer products, the bonding strength was almost constant at 130°C and the specimen thicknesses were measured. All recorded values were analyzed using two-way analysis of variance and Tukey’s Honest Significant Difference Test (P < 0.05). The present study confirmed that bonding strength was dependent on heating temperature: In ethylene-vinyl acetate copolymer products, the bonding strength was almost constant at 130°C and above, and it was constant at 110°C and above in polyolefin products. The thickness of every specimen decreased and, in some specimens, the shock absorption capability decreased with increasing heating temperature. The present study concludes that the heating temperature during the sheet lamination process when laminated custom-made mouthguards are fabricated may not be less than 120°C in ethylene-vinyl acetate copolymer products and 110°C in polyolefin products.

Keywords: heating condition, lamination process, mouthguard

Introduction

Mouthguards (MGs) reduce the incidence and severity of sports-related oral injuries, especially in contact sports [1]. Custom-made mouthguards (CMGs), which are fabricated on a cast prepared from dental arch impressions taken by a dentist, are very effective in trauma prevention [2].

Appropriate CMG design should afford both high trauma-prevention efficacy and comfort to users. An important factor in the design of CMGs is controlling the thickness. Specifically, the necessary surface, such as anterior teeth, must be sufficiently thick while the other surfaces are kept as thin as possible. MG sheets should be 3-4 mm thick to sufficiently reduce the impact force [3], but excessively thick MG on the palatal side may cause discomfort and even result in speech difficulty [4]. CMGs are classified as single-layer-type CMG (S-CMG) and laminated-type CMG (L-CMG), which consist of two or more layers. L-CMGs can keep the necessary thickness where it is required, thus ensuring the shock absorption capability of the CMG. Therefore, L-CMGs are superior to S-CMGs.

Both the ethylene-vinyl acetate copolymer (EVA) and polyolefin (PO) have been used as CMG materials. Over the past two decades, the material properties, shock absorption capabilities, and thickness after forming of MG materials have been widely studied, but most of the studies have considered S-CMGs only [5,6]. Although there have been a few reports on L-CMGs, L-CMGs have been fabricated using the optimal conditions for S-CMG fabrication through the sheet lamination process. However, L-CMGs tend to delaminate with use as the L-CMG spreads, which is a problem for their clinical application. The cause of delamination is insufficient bonding strength (BS) between two sheets. The heating temperature is regarded as an important factor affecting the BS of thermoplastic materials. Studies on S-CMG fabrication have provided a lot of information regarding the appropriate forming duration but not enough information on attaining a high BS. The optimal temperatures required to realize a high BS and to prevent delamination of L-CMGs probably differ from the conditions required to form S-CMGs.

The optimal heating conditions in the case of S-CMGs have been extensively reported [7,8], but there have been few reports on the BS of L-CMGs. Yamada et al. suggested that the suitable temperature range for EVA forming was 80-120°C as indicated by the forming capability index [9]. However, L-CMG fabrication requires both good forming and bonding; therefore, the optimal heating temperature needs to be further investigated.

In addition, it is necessary to consider how the change in heating temperature influences the shock absorption capability and the thickness after forming; this is essential for the MG function. In the case of S-CMGs, these factors have been extensively investigated, whereas there have been a few reports on the lamination process for L-CMGs. The relationship between shock absorption capability and the MG thickness has only been reported in the case of S-CMGs [10,11]. In the case where the BS is considerably weak, the laminated interface is not completely integrated. Therefore, the BS of this interface is thought to influence the L-CMG’s shock absorption capability.

The purpose of the present study was to evaluate the influence of heating temperature on the sheet lamination process when L-CMGs are fabricated based on the following factors: 1) BS, 2) thickness after forming, and 3) shock absorption capability.

Materials and Methods

Mouthguard materials

Seven MG sheet products were selected (Table 1). All sheets were selected to be 2 mm thick and transparent; these are common characteristics in the lamination process.

Measuring the bonding strength

Sheets of the same MG products were laminated together using a vacuum forming machine in a vacuum tank (Erkofor 3d, Erkodent, Pfalzgrafenweiler, Germany). The instrument was equipped with a touchless infrared temperature sensor, which measured the temperature at the center of the other side of the heated surface of the MG sheet loaded on the heating holder of the vacuum forming machine. The temperature at the position was confirmed using a calibrated alunel-chronel thermocouple sensor (CT-1310D, CUSTOM, Tokyo, Japan). The first layer MG sheet was cut into a 75 mm × 60 mm rectangular specimen and placed on a suction table. Then, the second layer was cut into a circle with a radius of 60 mm. This circular specimen was set in an MG sheet heating holder and heated at dif-
ferent heating temperatures: 90°C, 100°C, 110°C, 120°C, 130°C, 140°C, 150°C, and 170°C. For the formation process, MG sheets are not suitable at 180°C and above [9]. When each setup temperature was reached, the second layer was bonded to first layer while being suctioned for 3 min. After lamination, the test specimens were stored at room temperature (23°C) for 24 h. The laminated sheet was cut with a dumbbell-shaped cutter according to ISO 37 (2011) type 2 [12] so that the adhesive area was at the center of the narrow segment and sectioned at the center of this narrow segment as the final specimen for delamination testing. The adhesion area was controlled to be 4 × 7.5 mm (n = 6).

The width of the specimen was measured with a digital micrometer (Digimatic 293-421-20, Mitsutoyo, Kawasaki, Japan) before the delamination test. The BS was calculated as the maximum load divided by the width of the specimen. To evaluate the BS, a delamination test, namely a T-peel test, was performed based on previous research results [13-15] using a universal test machine (Model 1123, Instron, Norwood, MA, USA) at a cross head speed of 50 mm/min with a load cell capacity of 50 kg. A jig was used to grip the specimen firmly. The load and displacement were varied until specimen breakage was recorded by the material testing software (Series IX, Instron). All specimens were tested at room temperature (23°C).

The fracture patterns of the specimen after the delamination test were inspected visually and classified as material failure or interface failure, depending on the location of failure.

Measuring thickness after forming and shock absorption capability
Sheets of the same MG products (2 mm thickness) were laminated together using a vacuum forming machine (Erkoform 3d plus, Erkodent) at 90°C, 130°C, and 170°C.

Each of the obtained laminated specimens was round in shape and had a diameter of 50 mm (n = 5). Then, the specimen thicknesses were measured at three different points near the center of MG sheets using the digital micrometer, and the mean value was taken as the thickness of the MG sheets after lamination.

The free-falling ball impact test was selected to measure the specimens’ shock absorption capability because it is a convenient method that yields results that correspond closely to the actual impact strength [6]. This test was performed using a modified IM-201 impact testing machine (Tester Sangyo Co., Miyoshi, Japan; Fig. 1). A free-falling stainless steel ball (weight: 32.6 g, diameter: 20.0 mm), a 10 mm thick steel platform, and a stainless steel base plate (180 × 180 × 10 mm) was used in the test. The ball was released from 740 mm above the specimen, and the impact force generated was measured using a load cell sensor system. The control load without the specimen was set to 673 N, because the fracture load of the human upper incisor is 607-894 N [16,17]. Three dynamic LMB-A-2KN compression load cells with a rated capacity of 2 kN (Kyowa Electronics Instruments Co., Chofu, Japan) were located 120° apart on the base plate of the impact tester, and the stainless steel platform was placed upon the load cells. The change in force during the impact test was recorded with the three load cells, and the sum of the measured force values was calculated. After impact, the maximum of the sum of the measured forces was taken as the maximum impact force (MIF). All specimens were tested at room temperature (23°C).

Statistical analysis
All recorded values were entered into JMP11 (SAS Institute Inc., Cary, NC, USA) for statistical analysis. Tabled values are presented as mean values plus or minus the standard deviation. Statistical comparisons of the

| Code | Composition | Commercial name | Manufacturer | Lot number |
|------|-------------|-----------------|--------------|------------|
| EVA  | ethylene-vinyl acetate copolymer | Mountguard | Yamahachi Dental MFG. Co. Gamagori, Japan | 05046 |
| DRA  | ethylene-vinyl acetate copolymer | Drufosoft | Dreve DentaMid GmbH Unna, Germany | 20160998 |
| ERK  | ethylene-vinyl acetate copolymer | Erkoflex | Erkodent Pfalzgrafenweiler, Germany | 581220 |
| BIO  | ethylene-vinyl acetate copolymer | Bioplast | Schau Dental Technology Islohn, Germany | 170731 |
| CAP  | ethylene-vinyl acetate copolymer | Capture Sheet | Shofu Inc. Kyoto, Japan | 011801 |

| PO M21 | ethylene/ε-polymers | MG21 | CGK Corp. Hiroshima, Japan | 061170301 |
| CAP-P | polyolefin* | Capture Sheet Pro | Shofu Inc. | 021701 |

* polyethylene/polypropylene/ethylene propylene copolymer

![Fig. 1](image1.png) Modified IM-201 impact testing machine (Tester Sangyo Co., Miyoshi, Japan): (a) a free-falling stainless steel ball (weight, 32.6 g; diameter, 20.0 mm) was released from 740 mm above the specimen. (b) the generated impact force was measured using a load cell sensor system (Kyowa Electronics Instruments Co., Chofu, Japan).

![Fig. 2](image2.png) Change in the temperature measured by an infrared temperature sensor and a thermocouple.
BS, thickness after forming, and MIF were carried out, and two-way analysis of variance (ANOVA) and Tukey’s honest significant difference were performed to detect significant differences among combinations of heating temperature and product, because the data were normally distributed and homoscedastic (Shapiro-Wilk normal test, $P = 0.08$ and Levene’s test, $P = 0.56$). The significance level was set at $P < 0.05$.

**Results**

Figure 2 illustrates the temperature measured by the thermocouple plotted against the temperature measured by the infrared temperature sensor. All discrepancies between the two measurements were within 5°C. Figure 3 presents the BS results of the delamination test for each product. Overall, the BS increased with the heating temperature. The results of the two-way ANOVA were as follows: heating temperature: degrees of freedom (df) = 7, $F = 330$, $P < 0.001$; products: df = 6, $F = 49$, $P < 0.001$; and heating temperature × products interaction: df = 42, $F = 140$, $P < 0.001$. The ANOVA revealed that the heating temperature and product and the effects of their interaction on the BS were significant. Next, the effects of the temperature on each sheet were statistically analyzed separately in post-hoc tests. Within the product, each BS at 110°C and above was significantly higher than that at 90°C, and the BSs at 130°C and above were not significantly different from one another. The BSs of all EVA sheets at 120°C and above were significantly higher than those at 90 and 100°C, and for the PO sheets, the BSs at 110°C and above were significantly higher than those at 90°C. However, the BSs of all EVA sheets at 130°C and above were not significantly different from one another, and regarding the PO sheets, BS values from 110°C to 170°C were not significantly different.

Table 2 presents the classification of fracture patterns of the specimens after the delamination test. As indicated in Table 2, from 90°C to 130°C, the fracture patterns were observed only at the interface, and at 140°C, the material started to fail. The post-forming thickness of every specimen decreased as the heating temperature increased (Fig. 4). The results of the two-way ANOVA were as follows: heating temperature: df = 2, $F = 615$, $P < 0.001$; products: df = 6, $F = 89$, $P < 0.001$; and heating temperature × products interaction: df
= 12, F = 10, P < 0.001. The ANOVA revealed that the two main effects and their interaction were significant. Next, the main simple effects were statistically analyzed in post-hoc tests, the results of which are illustrated in Fig. 4. Broadly, the thicknesses after forming at 90°C, 130°C, and 170°C (except in case of CAP-P at 130°C and 170°C) were significantly different.

Figure 5 presents the results for the MIF of each product. The results of the two-way ANOVA were as follows: heating temperature: df = 2, F = 30, P < 0.001; products: df = 6, F = 23, P = 0.001; and heating temperature × products interaction: df = 12, F = 3, P = 0.004. ANOVA revealed that the two main effects and their interaction were significant. Hence, the simple main effects were statistically analyzed in post-hoc tests, revealing that for BIO, M21, and CAP-P, MIFs between 90°C and 170°C were significantly different (Fig. 5).

**Discussion**

For the lamination process in the present study, a vacuum forming machine for clinical use was employed, and particular attention was paid to the lamination process to control the experimental conditions. The conditions of the MG sheet surface had to be kept as clean as possible before forming. Lamination preparation procedures were carried out using new sheets to avoid contamination, because Mihindukulasuriya et al. reported the influence of contaminants on BS [18]. In the present study, no surface treatment agents, e.g., cleaner and adhesive, were used before forming, because preliminary experiments revealed that the surface treatment agents interacted with the MG sheets, and in some cases, this interaction inhibited lamination. Previous reports on the use of adhesives to laminate EVA sheets have had mixed results: One study reported that the use of adhesives was effective [19]; another study used an adhesive without comparison to no use of adhesive [13]; and a third study did not use adhesive agents [14].

The BS of the commercialized MG sheet used in the present study ranges from about 0 to 8 N/mm, consistent with values reported in other studies [14,15]. The BS of all products at 90°C was almost 0 N/mm, and commercial MG sheets did not adhere to each other. There were concerns that L-CMG would easily peel apart, or delaminate, due to daily use. The present study confirmed that the BS was dependent on the heating temperature: BS increased with the heating temperature. Within the same product, each BS at 110°C and above was significantly higher than that at 90°C, and the BSs at 130°C and above were not significantly different from each other. In each EVA sheet, the BS increased from 90°C to 110°C and was almost constant at 120°C and above. Statistically, the BS of each EVA sheet at 120°C and above was significantly higher than that at 90 and 100°C, whereas that at a temperatures from 130°C to 170°C was not significantly different from that at 90°C and 100°C. There were subtle differences between products, such as the peak of the BS and the temperature at maximum BS. When the BS became almost constant, it was approximately 4-7 N/mm, which indicated that it was not easy to delaminate MG materials. In PO sheets, BS was constant at 110°C and above, and there was no significant difference between the BS values from at temperatures from110°C to 170°C. Each product had somewhat different results, likely due to the difference in each product’s meltability and compounding ratio of the plasticizer; therefore, the heating temperature should be carefully considered for fabricating CMGs depending on the product used. Although the required BS for LMG is still unknown, results suggested that the in vitro heating temperature for LMGs should be higher than those conventionally used for SMGs.

Observations of fracture patterns reveal that if the BS is greater than the material strength, material breakdown occurs at the location of failure. In the present study, even though the BS of all EVA sheets at 130-170°C and all PO sheets at 110-170°C was not significantly different, fracture patterns of the specimen observed by visual inspection after the delamination test indicated material failure starting at 140°C in the case of DRU, ERK, and CAP and 150°C in the case of M21, but YAM and CAP-P did not indicate material failure. Material failures were seen only on the second layer’s side which was heated at the forming machine to over 140°C when observed in the delamination test. The second layer may have undergone heat-induced deterioration and/or thickness reduction due to overheating. Material failure can indicate the beginning of degradation, as well as the appropriate BS, and the temperature just before material failure can be applied for forming laminated mouthguards. Insight into this aspect will be necessary to produce reliable L-CMGs.

It is well-established that after forming, the MG thickness decreases [20], and this decrease is influenced by many factors, including the sheet material, sheet color, forming methods, and heating temperature [21]. Thickness decrease reduces the protection afforded by the CMG, especially to absorb, attenuate, and disperse shock. Reza et al. reported that the maximal load to mouthguard material test piece decreased with an increase in the MG thickness [11].

In the current study, the thickness of every specimen decreased with increasing heating temperature (Fig. 4). The thicknesses after forming between 90°C, 130°C, and 170°C (except in cases of CAP-P at 130°C and 170°C) significantly decreased as the heating temperature increased, indicating that higher temperatures lead to thinner specimens. During heat-softening, the increase in the sagging distance of the MG sheet from the vacuum forming machine caused a decrease in thickness. The temperature and heating time affect the sagging distance. To maintain the MG thickness, a requisite minimum sagging distance of MG sheets and a lower heating temperature may be important to ensure that the BS and other physical properties are not adversely affected. When CMGs are fabricated, attention should be paid to the heating temperature and the initial thickness of the MG sheets.

The shock absorption capability is one of the most important properties for MGs to prevent dental trauma, but few reports exist on the effects of the fabrication process of laminated MGs. In particular, in the case where the BS is considerably weak, the laminated interface is not completely integrated. Therefore, the BS of this interface is thought to influence the L-CMG’s shock absorption capability. Furthermore, thermal deterioration is also thought to influence this capability. In the present study, the MIF range was 381-464 N, which reduced the control load by 56.6-69.1%. These values are consistent with those obtained in other studies [5,21]. The result of BSs at 90°C in the present study indicated that integration of lamination had little influence on the shock absorbing capability of L-CMG. Thus, the bonding condition (almost unadhered interface failure and material failure) had negligible effect on MIF.

The MIFs of BIO, M21, and CAP-P between 90°C and 170°C were significantly different. The results of this experiment are similar to those of previous studies [10,11], and the thickness reduction after forming mouthguards led to a decrease in the shock absorbing capability. Therefore, when forming L-CMGs, the heating temperature should not be too high, because a high heating temperature causes thickness reduction of MG sheets and may subsequently decrease their shock absorption capability.

In conclusion, the results indicated that the suitable conditions for L-CMG fabrication differ from the suitable conditions for S-CMG fabrication. The results also suggest the heating temperature when fabricating L-CMGs may not be less than approximately 120°C in EVA products and 110°C in PO products; this is higher than the heating temperature that has long been employed for S-CMG fabrication. By contrast, the thickness after forming and the shock absorption capability both decreased with increasing heating temperature. To ensure the MG’s thickness after forming and its shock absorption capability, the optimal heating temperature when fabricating L-CMGs should not be higher than that required for lamination. In clinical use, formability is one of the most important points for one to evaluate during thermoforming of MGs [9]. In the current study, the MG sheets twisted, and the formability deteriorated at 150°C or higher, because formability is one of the most important properties in clinical use [9]; this result provides more evidence against using a temperature higher than that necessary for lamination.

The results of this research are experimental data by only one type of machine from immediately after forming onward. The present study does not consider saliva, clenching of the jaw, body temperature conditions over time, or the oral anatomical form and environment. Some materials, such as EVA, have high water absorption, which affects MG properties. For future research, experiments should be conducted under conditions that more closely mimic actual use.

**Acknowledgments**

The author would like to thank Associate Professor Meiko Oki for her encouragement and advice and Editage (www.editage.jp) for English language editing.
Conflict of interest
The authors declare no conflict of interest.

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