

Effects of CO₂, Er:YAG, and Nd:YAG laser treatments on shear bond strength of resin cement to zirconia

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Abstract

Objective: This study aimed to evaluate the effect of different lasers at various settings on the shear bond strength (SBS) of resin cement to zirconia.

Methods: In this in vitro study, 165 zirconia discs were divided into 11 groups (n=15). The groups underwent the following treatments: a control group with no treatment, a sandblasted group with 50 µm Al₂O₃, three groups treated with a CO₂ laser (4 W, 5 W, 6 W), three groups treated with an Er:YAG laser (3 W, 4 W, 6 W), and three groups treated with a Nd:YAG laser (2 W, 2.5 W, 3 W). Composite cylinders were bonded to the zirconia using resin cement. After 24 hours, the samples were thermocycled and SBS was measured using a universal testing machine. Data were analyzed by the Welch test and the Games-Howell test ($\alpha=0.05$).

Results: The sandblasted group exhibited the highest SBS, while the control group showed the lowest. A significant difference in SBS was observed between the groups ($P<0.001$). Pairwise comparisons revealed no significant differences between the sandblasted group and the Nd:YAG laser groups (2 W, 2.5 W, 3 W), the CO₂ laser group (6 W), and the Er:YAG laser group (6 W) ($P > 0.05$).

Conclusions: Sandblasting with Al₂O₃ particles yielded the highest bond strength of resin cement to zirconia. Alternatively, Nd:YAG at all tested parameters, and Er:YAG, and CO₂ lasers at specific settings could be used to enhance bond strength to zirconia. Among lasers, the Nd:YAG laser at 2.5 W achieved the best performance.

Keywords: CO₂ laser, Er:YAG laser, Nd:YAG laser, Shear bond strength, Zirconia, Surface treatment

Introduction

The success of zirconia ceramics in dental restorations largely depends on their cementation bond strength (1-5). Zirconia ceramics have gained widespread use in dentistry due to their biocompatibility, high aesthetic appeal, compressive strength, and thermal expansion coefficient similar to that of natural teeth (6-9). However, a significant challenge with zirconia restorations, compared to glass-ceramic restorations, is achieving adequate bonding to resin cement (4,5). This difficulty arises mainly from the absence of a glass phase in zirconia, which limits the effectiveness of traditional acid etching. Consequently, alternative methods are

required to enhance adhesion when using adhesive or silane-containing resin cements (10,11).

Adhesive cement bonds to ceramic surfaces through micromechanical interlocking (12). To achieve this micromechanical bond, the surface of zirconia ceramics must be roughened, increasing the contact area for resin penetration (13-16). Various techniques have been proposed for preparing the internal surface of zirconia restorations, including diamond bur abrasion, sandblasting with aluminum oxide particles, silica coating, and laser irradiation. These surface modifications improve micromechanical adhesion by enhancing surface energy, wettability, and roughness (16-20).

In dentistry, lasers are widely utilized for multiple applications such as soft tissue surgery, removing decayed dentin, surface preparation of enamel, and surface treatment of indirect restorations (21). Laser irradiation on zirconia ceramics induces surface abrasion and roughness, potentially improving the micromechanical adhesion of resin cement (22,23).

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Although multiple studies have investigated the effects of various lasers on the shear bond strength (SBS) of zirconia ceramics (24-26), limited information is available regarding the comparison of different types of lasers on this parameter. Parameters such as irradiation time, pulse energy, frequency, and power may play a crucial role in the outcome. Therefore, this study aimed to evaluate and compare the SBS of Panavia F 2.0 resin cement to zirconia ceramics after surface preparation with sandblasting and different lasers including erbium-doped yttrium-aluminum-garnet (Er:YAG) laser, neodymium-doped yttrium aluminum garnet (Nd:YAG) laser, and carbon-dioxide (CO₂) laser, at varying power levels. The null hypothesis of this study was that there would be no significant difference in SBS between the sandblasted group, laser-treated groups, and the control group.

Materials and methods

A total of 165 multilayer zirconia discs (5Y-TZP, Kuraray Noritake Dental Inc., Aichi, Japan) with a diameter of 10 mm and thickness of 3 mm were fabricated using a CAD/CAM system (Ceramill Motion 2, Amann Girrbach AG, Koblach, Austria). The discs were polished with 600-grit silicon carbide abrasive paper (Matador 991A, Soflex Stracke, Melle, Germany) and mounted on 4.5 × 2.1 cm molds using quick-setting acrylic resin (Acropars, Tehran, Iran).

The samples were divided into 11 groups of 15 each. No surface treatment was performed in the control group (group 1). In group 2, zirconia surfaces were sandblasted with 50 µm Al₂O₃ particles at 3 bar pressure for 10 seconds from a 10 mm distance.

Groups 3-5 were treated with a CO₂ laser (Smart US-20D, Deka, Florence, Italy) operating at a wavelength of 10,600 nm. The laser was operated in pulsed mode at a frequency of 100 Hz, and a total irradiation time of 35 seconds. The settings varied among these groups. Group 4 received 4 W power, 178.34 J/cm² energy density, and 1 ms pulse duration; group 5 was irradiated at 5 W power, 221.92 J/cm² energy density, and 1.3 ms pulse duration; and group 6 was exposed to 6 W power, 267.51 J/cm² energy density, and 1.5 ms pulse duration.

Groups 6-8 were treated with an Er:YAG laser (Pluser, Doctor Smile, Lambda SpA, Vicenza, Italy) at a wavelength of 2940 nm for 30 seconds at a frequency of 20 Hz. In group 6, pulse energy was 150 mJ (3 W power) resulting in 29.86 J/cm² energy density. Group 7 was irradiated with 200 mJ pulse energy (4 W power) and 39.8 J/cm² energy density, and group 8 with a pulse

energy of 300 mJ (6 W power) and an energy density of 59.7 J/cm².

Finally, groups 9-11 were treated with a 1064 nm Nd:YAG laser (LightWalker ATS, Fotona, Ljubljana, Slovenia) at a frequency of 20 Hz, 300 µs pulse duration, and irradiation time of 45 seconds. Group 9 was irradiated with 100 mJ energy (2 W power) and 142.88 J/cm² energy density; group 10 with 125 mJ energy (2.5 W power) and 178.57 J/cm² energy density; and group 11 with 150 mJ energy (3 W power), and 214.28 J/cm² energy density.

The laser tip was positioned perpendicular to the ceramic surface at a 1-mm distance in all laser groups.

After surface treatment, the samples were cleaned in an ultrasonic cleaner (Elmasonic-S60H, Elma, Singen/Htw, Germany) with 96% isopropanol alcohol for three minutes. Surface morphology after treatment was evaluated in one sample from each group using a scanning electron microscope (SU3500; Hitachi High Tech Corp., Tokyo, Japan) at 5000× magnification.

Bonding procedure

Transparent plastic tubes with an inner diameter of 3 mm and a height of 4 mm were filled with resin composite (shade A3.5; Filtek Z250, 3M ESPE, St. Paul, MN, USA). The composites were then polymerized using a light-curing device (Heliolux DLX; Ivoclar Vivadent, Schaan, Liechtenstein) with a minimum intensity of 600 mW/cm² for 40 seconds from two directions. This process led to the preparation of composite cylinders for bonding to the zirconia surface. Zirconia and composite cylinders were etched with 37% phosphoric acid for five seconds, rinsed, and dried thoroughly.

The zirconia surfaces were prepared with a ceramic primer (Clearfil Ceramic Primer Plus, Kuraray Noritake Dental, Okayama, Japan) according to the manufacturer's instructions. The ED Primer II (Panavia F2.0, Kuraray, Okayama, Japan) was applied to the composite cylinders. Dual-cure resin cement (Panavia F2.0) was mixed and applied to the zirconia surface. The composite cylinders were positioned and pressed onto the zirconia discs using a Gilmore needle with a weight of 435.6 g. Initial curing was performed for 10 seconds, after which excess cement was removed. Oxyguard II gel (Panavia F2.0) was then applied to prevent the formation of an oxygen-inhibited layer, followed by final curing for 40 seconds in four directions.

Thermocycling process and bond strength testing

The bonded specimens were stored in distilled water at 37°C for 24 hours, followed by 2000 thermal cycles

Table 1. Mean and standard deviation (SD) of shear bond strength (SBS) for the study groups, reported in MPa

Group	Definition	Mean $\pm$ SD
1	Control	3.79 $\pm$ 0.97 ^a
2	Sandblasting	11.14 $\pm$ 3.89 ^c
3	CO ₂ 4 W	3.90 $\pm$ 0.76 ^a
4	CO ₂ 5 W	6.32 $\pm$ 1.85 ^b
5	CO ₂ 6 W	9.55 $\pm$ 3.77 ^{b, c}
6	Er:YAG 3 W	6.51 $\pm$ 1.20 ^b
7	Er:YAG 4 W	6.71 $\pm$ 1.20 ^b
8	Er:YAG 6 W	7.88 $\pm$ 2.48 ^{b, c}
9	Nd:YAG 2 W	9.62 $\pm$ 2.43 ^c
10	Nd:YAG 2.5 W	10.87 $\pm$ 3.02 ^c
11	Nd:YAG 3 W	10.38 $\pm$ 2.48 ^c
P-value		<0.001

The p-value represents the results of the Welch test.

Different lowercase superscript letters indicate statistically significant differences between groups at $P < 0.05$, based on pairwise comparisons.

between 5°C and 55°C, with a dwell time of 20 seconds in each bath and a transfer time of 10 seconds. Shear bond strength (SBS) was tested using a universal testing machine (Bongshin, DBBP-2t, Seongnam, Korea) at a crosshead speed of 0.5 mm/min.

Failure analysis

After SBS testing, failure modes were assessed under a stereomicroscope (Carl Zeiss Inc., Oberkochen, Germany) at 40 $\times$ magnification. Failures were categorized as adhesive failure, occurring at the ceramic-resin interface; cohesive failure, occurring within the resin cement; or mixed failure, identified by the presence of resin cement or composite remnants on the ceramic surface.

Statistical analysis

Data were analyzed using IBM SPSS software (version 22; IBM Inc., Armonk, NY, USA). The Shapiro–Wilk test was used to confirm normal data distribution and the results confirmed that the SBS data for different surface preparation methods followed a normal distribution ($P > 0.05$). Welch's analysis of variance was performed to assess SBS differences among eleven groups, with pairwise comparisons made using the Games-Howell post hoc test. The chi-square test was employed to compare failure types between groups. Statistical significance was set at $P < 0.05$.

Results

Table 1. presents the mean and standard deviation (SD) of SBS values for all groups, reported in megapascals (MPa). The Welch test showed a statistically significant difference in SBS among the 11 groups ($P < 0.001$). Pairwise comparisons using the Games-Howell post hoc test revealed that there was no significant difference in SBS between the control group and the CO₂ laser group at 4 W ($P > 0.05$). Furthermore, the SBS of the sandblast group was not significantly different from the CO₂ laser group at 6 W, the Er:YAG laser group at 6 W, and all Nd:YAG laser groups ($P > 0.05$; Table 1).

Significant differences were observed between the sandblast group and several other groups. The SBS value of the sandblast group was significantly greater than those of the control, the CO₂ laser group at 4 W and the CO₂ laser group at 5 W ($P < 0.05$; Table 1). Significant differences were also found between the sandblast group and the Er:YAG laser group at 3 W as well as the Er:YAG laser group at 4 W ($P < 0.05$; Table 1).

The distribution of failure modes is presented in Table 2. The chi-square test indicated a significant difference in failure modes among the groups ($P < 0.001$). Adhesive failure was predominantly observed in the control group (78.6%), and Er:YAG and Nd:YAG laser groups at low power settings including Er:YAG laser at 3 W (100%), Er:YAG laser at 4 W (85.7%), and Nd:YAG laser at 2 W (71.4%). In contrast, mixed-type failures were most prevalent in the sandblast group (57.1%), all CO₂ laser groups (ranging from 64.3% to 92.9%), Nd:YAG laser

groups at 2.5 W and 3 W (64.3% and 57.1%, respectively), and Er:YAG laser group at 6 W (57.1%). No case of cohesive failure was detected in any group.

Morphological changes in zirconia surface structures were evident in all study groups except for the control group, with the sandblast group exhibiting the most pronounced alterations (Figures 1-4).

Discussion

This study evaluated the effect of different laser wavelengths and settings on the shear bond strength of resin cement to zirconia ceramic and compared the results with control (no treatment) and sandblasted specimens. The results of this study indicated that surface preparation methods, including sandblasting and most laser treatments, significantly enhanced the bond strength of zirconia ceramic.

The lowest bond strength was observed in the control group (3.79 ± 0.97 MPa). The only group that showed no significant difference from the control group was the CO₂ laser at 4 W (3.90 ± 0.76 MPa). The sandblasting group exhibited the highest shear bond strength among the study groups (11.14 ± 3.89 MPa), followed by Nd:YAG laser at 2.5 W (10.87 ± 3.02 MPa), Nd:YAG laser at 3 W (10.38 ± 2.48 MPa), Nd:YAG laser at 2 W (9.62 ± 2.43 MPa), CO₂ laser at 6 W (9.55 ± 3.77 MPa), and Er:YAG laser at 6 W (7.88 ± 2.48 MPa). These groups were statistically comparable and showed significantly higher bond strength than most other groups. Although laser treatments at specific parameters improved

zirconia's bonding properties, they did not show a significant superiority over the sandblasting method. Because of simplicity and cost-effectiveness, sandblasting remains a more reasonable option for enhancing bond strength in the clinical setting.

The micromechanical interlocking mechanism plays a key role in enhancing the bonding quality of resin cement to zirconia ceramics. Greater roughness on zirconia restorations facilitates resin cement penetration into the surface, thereby improving the bond strength. Notably in this study, the sandblasting group achieved the highest SBS for zirconia ceramics, significantly outperforming the control, CO₂ laser at 5 W, CO₂ laser at 4 W, Er:YAG laser at 3 W, and Er:YAG laser at 4 W groups. This is consistent with previous research demonstrating that techniques such as diamond bur abrasion and sandblasting are highly effective in eliminating surface contaminants, increasing surface area, and improving wettability—key factors for achieving optimal bonding (16-21).

Although lasers offer an alternative approach to surface preparation, there is no standard method for laser application on zirconia. A wide variability is observed in the laser parameters employed in the literature (27). The laser output power (or the combination of pulse energy and frequency) and energy density are crucial factors influencing the bond strength between resin cement and zirconia ceramics (16). Therefore, in this study, various laser wavelengths were applied at different power settings to achieve a better

Table 2. Distribution of failure types across the study groups

Groups	Definition	Failure type [Number (%)]		
		Adhesive	Cohesive	Mixed
1	Control	11 (78.6%)	0 (0%)	3 (21.4%)
2	Sandblasting	6 (42.9%)	0 (0%)	8 (57.1%)
3	CO ₂ 4 W	4 (28.6%)	0 (0%)	10 (71.4%)
4	CO ₂ 5 W	5 (35.7%)	0 (0%)	9 (64.3%)
5	CO ₂ 6 W	1 (7.1%)	0 (0%)	13 (92.9%)
6	Er:YAG 3 W	14 (100%)	0 (0%)	0 (0%)
7	Er:YAG 4 W	12 (85.7%)	0 (0%)	2 (14.3%)
8	Er:YAG 6 W	6 (42.9%)	0 (0%)	8 (57.1%)
9	Nd:YAG 2 W	10 (71.4%)	0 (0%)	4 (29.6%)
10	Nd:YAG 2.5 W	5 (35.7%)	0 (0%)	9 (64.3%)
11	Nd:YAG 3 W	6 (42.9%)	0 (0%)	8 (57.1%)
P-value		<0.001		

The p-value represents the results of the chi-square test.

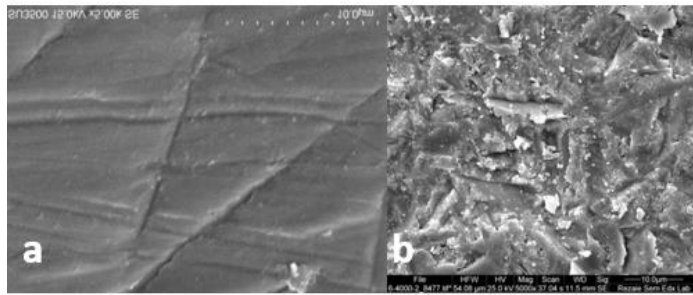


Figure 1. SEM images of zirconia surfaces at 5000× magnification for the control group with no surface treatment (a), and sandblast-treated group (b).

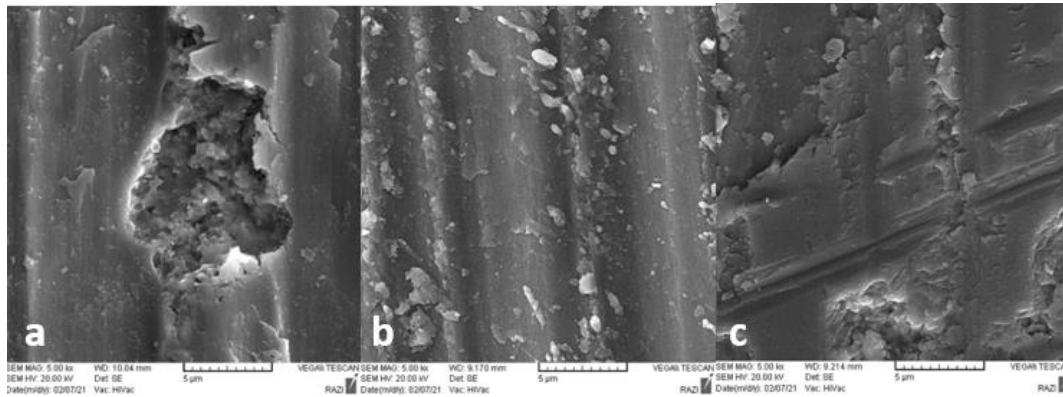


Figure 2. SEM images of zirconia surfaces prepared with CO₂ laser at 5000× magnification, with power settings of 4 W (a), 5 W (b), and 6 W (c).

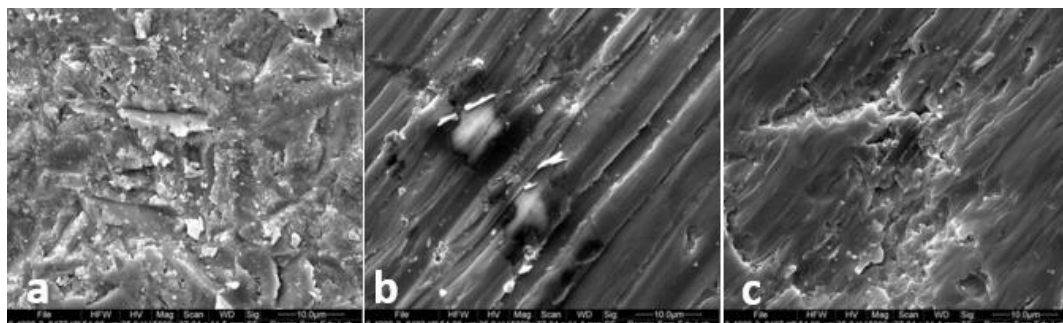


Figure 3. SEM images of zirconia surfaces prepared with Er:YAG laser at 5000× magnification, with power settings of 3 W (a), 4 W (b), and 6 W (c).

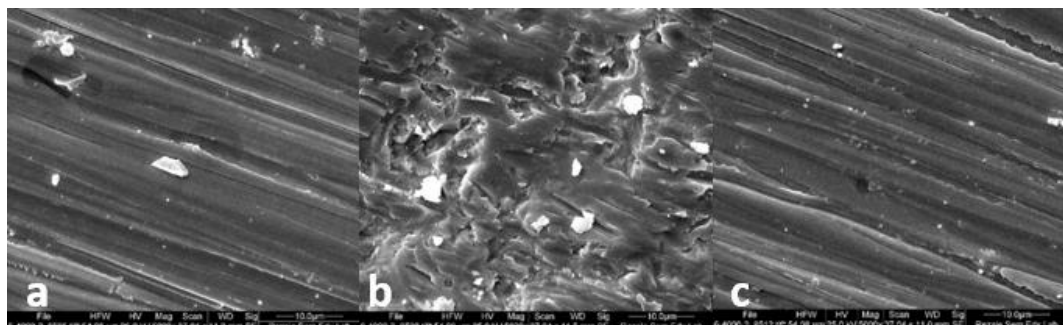


Figure 4. SEM images of zirconia surfaces prepared with Nd:YAG laser at 5000× magnification, with power settings of 2 W (a), 2.5 W (b), and 3 W (c).

conclusion about the optimal parameters for the treatment of zirconia ceramic.

The findings of this study align with Akyil et al. (28), who evaluated the SBS of yttrium-stabilized tetragonal zirconia (Y-TZP) ceramics to resin cement after various surface treatments. They found that air abrasion and

silica coating provided the highest SBS. However, CO₂ and Er:YAG laser irradiation alone, or Nd:YAG laser irradiation following air abrasion, could serve as alternative methods for enhancing the bond strength between resin cement and Y-TZP ceramics (28). Kasraei et al. (29) reported that after thermocycling and six

months of water storage, the SBS of the control group was significantly lower than that of the laser-treated groups, confirming the effectiveness of CO₂ and Nd:YAG laser treatments in enhancing the SBS of resin cement to zirconia ceramics.

In contrast to the outcomes of this study, Hatami et al. (30) identified Er:YAG laser as the most effective method for enhancing SBS to zirconia, which showed comparable results to sandblasting technique. In the present study, specimens treated with Er:YAG laser at 6 W demonstrated lower but comparable SBS to the sandblasting group, while lower power settings (i.e., 3 W and 4 W) resulted in significantly lower SBS. Tabatabai et al. (31) indicated that treatments with Nd:YAG and Er:YAG lasers resulted in the lowest SBS values, which were comparable to the control. Kasraei et al. (32) evaluated the impact of CO₂ and Er:YAG lasers on SBS and surface roughness of zirconia disks. They concluded that both lasers enhanced these properties, with the CO₂ laser outperforming the Er:YAG laser. A meta-analysis conducted by Bitencourt et al. (24) concluded that the Er:YAG laser did not cause a significant improvement in bond strength between Y-TZP zirconia disks and resin cement, compared to the control. These different results are mainly due to variations in laser parameters as well as differences in sample dimension and adhesive systems applied between the studies.

SEM analysis demonstrated that both the increase in laser power and the use of sandblasting substantially altered the surface morphology of zirconia. These changes included enhanced surface roughness and distinct textural modifications, which are known to improve micromechanical interlocking and thereby strengthen the bond between zirconia and resin cement.

The adhesive failure mode was significantly different among the groups. Adhesive failures were predominantly observed in the control group and Er:YAG and Nd:YAG laser groups at low power settings including Er:YAG laser at 3 W, Er:YAG laser at 4 W, and Nd:YAG laser at 2 W. In contrast, mixed-type failures were most prevalent in the sandblasting group, all CO₂ laser groups, Nd:YAG laser groups at 2.5 W and 3 W, and Er:YAG laser group at 6 W. No case of cohesive failure was detected in any group. Kasraei et al. (32) observed that the adhesive failure predominantly occurred in the control group, and the mixed failure patterns in laser-treated groups, which were consistent with the present findings. Hatami et al. (30) also found that the control group predominantly exhibited adhesive failure. Moreover, the laser-treated groups, particularly those

using Nd:YAG and Er:YAG lasers, displayed an increasing proportion of mixed failures as SBS improved.

This study is limited by the in vitro design and the lack of variation in resin cements and zirconia types, which may restrict the generalizability of the findings. Future research should explore the survival of laser-treated and sandblasted surfaces in clinical settings.

Conclusions

According to the outcomes of this study:

- 1- Sandblasting with 50 µm aluminum oxide particles provided the highest SBS for zirconia ceramics, significantly outperforming the control, CO₂ laser at 5 W, CO₂ laser at 4 W, Er:YAG laser at 3 W, and Er:YAG laser at 4 W groups.
- 2- Increasing the power of both CO₂ and Er:YAG lasers to 6 W resulted in the highest SBS values within their respective groups, which were not significantly different from the sandblasting group.
- 3- Among the laser treatments, Nd:YAG laser was most effective in enhancing SBS, showing no significant difference in all settings compared to the sandblasting group.

Acknowledgments

None to report.

Conflict of interest

There is no conflict of interest.

Authors' contributions

All authors contributed to the study conception and design. Material preparation, data collection and analysis were performed by R.G., A.J., M.M., A.M. and S.T. All authors read and approved the final manuscript.

Ethical approval

This study was approved by the ethics committee of Shahid Beheshti University of Medical Sciences under the ethical code IR.SBMU.DRC.REC.1399.085.

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