**Purpose:** This study investigated the effect of different surface treatments and the effect of silane heat treatment with laser on the shear bond strength (SBS) of a nanoceramic composite to repaired hybrid CAD/CAM blocks.

**Materials and Methods:** 60 hybrid CAD/CAM specimens (Cerasmart, GC) were prepared and randomly divided into six groups according to the different surface treatments (n = 10): group ER: Er:YAG laser+silane (Monobond Plus, Ivoclar Vivadent); group ER+SHT: Er:YAG laser+silane heat treatment; group B: bur+silane; group B+SHT: bur+silane heat treatment; group HF: hydrofluoric acid+silane; group HF+SHT: hydrofluoric acid+silane heat treatment. Afterwards, a universal adhesive (Universal Bond Quick, Kuraray) was applied, and nanoceramic resin composite (Zenit, President) cylinders were bonded to the Cerasmart specimens. They were thermocycled for 10,000 cycles (5–55°C) and subjected to SBS testing using a universal testing machine. Failure modes were examined with a stereomicroscope (15X). Scanning electron microscopy (SEM) was used to evaluate the surface topography (n = 2). The data were statistically analyzed using the Mann-Whitney U-test and the Kruskal-Wallis test (p < 0.05).

**Results:** Regarding the surface treatments, group ER showed significantly lower SBS than groups B and HF (p < 0.05). Regarding the presence of silane heat treatment by laser, groups ER+SHT and B+SHT showed significantly lower SBS than group HF+SHT (p < 0.05). In addition, group B+SHT showed significantly lower SBS than did group B (p < 0.05).

**Conclusion:** Er:YAG laser treatment for repairing hybrid CAD/CAM blocks was not as effective as bur roughening or hydrofluoric acid etching. Silane heated by Er:YAG laser was incapable of significantly increasing the bond strength to repaired hybrid CAD/CAM blocks.

**Keywords:** shear bond strength, CAD/CAM, silane heat treatment, Er:YAG laser.
performed as surface treatments to ensure a durable bond between repair material and CAD/CAM blocks. Nevertheless, there is limited data on the efficacy of universal dental adhesives in combination with various surface treatments of resin nanoceramic and hybrid ceramic CAD/CAM block materials.3,40

It is reported that erbium:yttrium aluminum garnet (Er:YAG) laser irradiation increases the bond strength of dental restorative materials by creating microretentive surfaces.39 Additionally, the use of a silane coupling agent is recommended to enhance the wettability of the surface to create effective adhesion. A silane heating procedure can improve the silane condensation reaction with the help of durable covalent bonds.26 To the authors’ knowledge, the literature contains only limited data regarding the effect of laser applications for silane heat treatment (SHT) on the adhesion of CAD/CAM hybrid materials.7,24

Further research is required, as previous studies used various laser parameters and reported conflicting results.24 The results of laser-heated silane procedures are also unclear. Thus, the purpose of this study was to investigate the effect of different surface treatments and laser heat treatment of silane on the shear bond strength (SBS) of a nanoceramic composite to repaired CAD/CAM hybrid blocks. The null hypotheses of this study were: 1. Different surface treatments would not affect the shear bond strength of the nanoceramic composite to repaired CAD/CAM hybrid blocks. 2. Laser heat treatment would not affect the shear bond strength of the nanoceramic composite to repaired CAD/CAM hybrid blocks.

**MATERIALS AND METHODS**

**Specimen Size Calculation**

A power analysis was performed to establish specimen size according to a previous study.24 In this study, for each group, a minimum of 10 specimens was required to gain a medium effect size (d = 0.50) with 90% power and a 5% type-1 error rate.

**Specimen Preparation and Restorative Procedures**

The chemical compositions and brands of the restorative materials used are summarized in Table 1. Sixty hybrid CAD/CAM blocks (Cerasmart, GC; Tokyo, Japan) were cut with a diamond-coated disk (Dimos, Ø125, Metkon; Bursa, Turkey) into bars with dimensions of 5 x 10 x 10 mm² and embedded in acrylic molds. The surfaces were prepared by polishing with silicon carbide papers (400, 600, 800, and 1000 grit). They were then randomly divided into six groups for different surface treatments (n = 12). Ten specimens from each group were evaluated with shear bond strength testing, and two specimens from each group were examined using SEM (Hitachi S-4800 FEG Scanning Electron Microscope, Hitachi; Tokyo, Japan). Specimen preparation is illustrated schematically in Fig 1.

- **Group HF** (hydrofluoric acid etching + silane): An Er:YAG laser (AT Fidelis, Fotona; Ljubljana, Slovenia) was applied to the hybrid block surface for 30 s in non-contact mode (20 Hz, long pulse, 5 W and 250 mJ).15 Silane (Monobond Plus, Ivoclar Vivadent, Liechtenstein) was then applied and allowed to dry for 60 s.
- **Group ER+SHT** (Er:YAG laser etching + silane heat treatment): In addition to surface pretreatment procedures in group ER, the silane was heated with Er:YAG laser irradiation according to the parameters in group ER.
- **Group B** (bur roughening + silane): The hybrid block surfaces were abraded with a coarse-fissure diamond bur (green band, 125–150 µm) (Le Blond A&M Instruments; Alpharetta, GA, USA), and then silane was applied as described above.
- **Group B+SHT** (bur roughening + silane heat treatment): After the hybrid block surfaces were abraded with a coarse-fissure bur, silane was applied and then heated by Er:YAG laser irradiation according to the parameters in group ER.
- **Group HF** (hydrofluoric acid etching + silane): The hybrid blocks were washed and dried after 90 s of hydrofluoric acid etching (Ultradent Porcelain Etch, Ultradent; South Jordan, UT, USA), then the silane procedure was performed as described above.

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**Table 1** The restorative materials used, their compositions, and brand numbers

<table>
<thead>
<tr>
<th>Brand names</th>
<th>Manufacturer</th>
<th>Composition</th>
<th>Lot number</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cerasmart</td>
<td>GC; Tokyo, Japan</td>
<td>Bis-MEPP, UDMA, DMA with 71 wt% silica and barium glass nanoparticles</td>
<td>2007281</td>
</tr>
<tr>
<td>Monobond Plus</td>
<td>Ivoclar Vivadent; Schaan, Liechtenstein</td>
<td>MPTMS, 10-MDP; disulfide dimethacrylate, ethanol</td>
<td>Z00WXC</td>
</tr>
<tr>
<td>Clearfil Universal Bond Quick</td>
<td>Kuraray Noritake; Tokyo, Japan</td>
<td>Bis-GMA, HEMA, ethanol, 10-MDP. hydrophilic aliphatic dimethacrylate, colloidal silica, CQ, silane coupling agent, accelerators, initiators, water</td>
<td>780219</td>
</tr>
<tr>
<td>Zenit</td>
<td>President; Munich, Germany</td>
<td>Glass filler, pyrogenic silica, agglomerated nanoparticles, diurethane dimethacrylate, butanediol dimethacrylate, isopropylidene- bis [2(3)-hydroxy-3(2)- (4-phenoxy) propyl] bis-methacrylate</td>
<td>2019009884</td>
</tr>
</tbody>
</table>

Bis-GMA: bisphenol A glycidyl methacrylate; bis-MEPP: bis-methacryloxyethoxy phenyl propane; DMA: dodecyl dimethacrylate; HEMA: 2 hydroxyethyl methacrylate; MDP: methacryloyloxydecyl dihydrogen phosphate; MPTMS: γ-methacryloxypropyl trimethoxysilane; UDMA: urethane dimethacrylate; TEG-DMA, triethylene glycol dimethacrylate; wt%, weight percentage.
Group HF+SHT (hydrofluoric acid etching + silane heat treatment): After 90 s of hydrofluoric acid etching, the hybrid blocks were washed and dried, silane was applied and then heated by silane Er:YAG laser irradiation to the parameters in group ER.

Prior to the shear bond strength test, a thin layer of universal adhesive (Clearfil Universal Bond Quick; Kuraray Noritake, Japan) was applied to the specimens (n = 10), and after waiting for 10 s, they were polymerized with a light-emitting diode (LED) light-curing unit (LCU) (1000 mW/cm²) (Valo, Ultradent) for 20 s. A nanoceramic resin composite (Zenit, President; Munich, Germany) was filled into a cylindrical teflon mold (2 mm height, 2.4 mm diameter), light cured for 20 s with the LED LCU, and bonded to the hybrid CAD/CAM material. All restorative procedures were performed by a single operator (C.D.) according to the manufacturers’ instructions.

All specimens were thermocycled (SD Mechatronik Thermocycler THE-1100, Mechatronik; Feldkirchen-Westerham, Germany) for 10,000 cycles between 5°C and 55°C (30 s dwell time, 10 s transfer time). They were then subjected to a notch-edge SBS test using a universal testing machine (AGS-X, Shimadzu; Kyoto Japan) with a crosshead speed of 1 mm/min. The load was directly applied on the resin composite/repaired CAD/CAM block interface until fracture occurred. The diameter of the bonded resin composite cylinder was the same as the notched-edge crosshead. Shear bond strength (SBS) was converted to MPa by dividing the failure load (N) by the bonding area (mm²). One operator (Z.O.) who was blinded to the surface treatments used in this study performed all SBS tests.

**Failure Mode Analysis**
The failure modes of the specimens were determined using a stereomicroscope (SMZ 1000, Nikon; Tokyo, Japan) under 15X magnification. The mode of failure was determined as “adhesive” if the fracture appeared along the junction of the resin composite and the repaired CAD/CAM block, and “cohesive” if the fracture occurred in the resin composite or repaired CAD/CAM block. Finally, if the fracture appeared along the junction of the resin composite and repaired CAD/CAM block as well as in the composite resin or repaired CAD/CAM block, the mode of failure was determined as “mixed”. One operator (B.O.) who was blinded to the surface treatments used in this study performed all failure mode analyses.

**SEM Analysis**
After surface treatments, two specimens from each group were evaluated by SEM. The specimens were gold sputter-coated for an examination of the repaired CAD/CAM block surfaces at an accelerating voltage of 10 kV in secondary mode. The micromorphology of representative surfaces was achieved at 500X and 1000X magnification. One operator (E.E.D.) who was blinded to the surface treatments used in this study performed all SEM procedures.

**Statistical Analysis**
Statistical analysis was performed with SPSS 22.0 software. First, the normality of variables was tested by the Shapiro-Wilk test, and the data were then analyzed with the Levene test to determine the homogeneity of variances. Nonparametric tests were used, since they did not satisfy parametric test assump-
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Failure Mode Analysis
Table 3 illustrates the failure modes of all groups. The predominant failure mode for most of the groups was adhesive. Group B+SHT exhibited the highest frequency (80%) of adhesive failures, while group HF+SHT exhibited the lowest frequency (10%). Furthermore, group HF+SHT predominantly showed mixed failure mode in repair materials (80%). No cohesive failures were detected for group ER or group ER+SHT. Finally, group B presented cohesive and mixed failure modes in restorative materials at equal rates (20%).

SEM Analysis
Figure 2 displays representative SEM images of hybrid blocks treated with different surface treatments. Different surface topographies were observed depending on the surface treatment. For example, after bur roughening, parallel scratches and grooves were detected (Fig 2c), while laser-etched groups revealed smoother surface alterations (Fig 2a). Furthermore, surface irregularities were observed for HF-etched groups (Fig 2e), and melted areas were detected with silane heat treatment in the bur-roughened group (Fig 2d).

Table 2  SBS means ± SD and median values (1st-3rd quarter) of all tested groups in MPa (n = 10)

<table>
<thead>
<tr>
<th>Treatment</th>
<th>ER</th>
<th>B</th>
<th>HF</th>
</tr>
</thead>
<tbody>
<tr>
<td>Without SHT</td>
<td>6.53 ± 0.61&lt;sup&gt;a&lt;/sup&gt;</td>
<td>12.35 ± 2.24&lt;sup&gt;b&lt;/sup&gt;</td>
<td>9.40 ± 1.18&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>With SHT</td>
<td>6.81 ± 0.95&lt;sup&gt;a&lt;/sup&gt;</td>
<td>7.05 ± 1.41&lt;sup&gt;a&lt;/sup&gt;</td>
<td>9.51 ± 2.07&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>p</td>
<td>0.541</td>
<td>&lt;0.001</td>
<td>0.549</td>
</tr>
</tbody>
</table>

<sup>*Different superscript lowercase letters indicate difference within columns. SHT, silane heat treatment; ER: Er:YAG laser treatment; B: bur roughening; HF: hydrofluoric acid etching.</sup>

Table 3  Failure mode analysis of fractured surfaces (%)

<table>
<thead>
<tr>
<th>Group</th>
<th>Adhesive (%)</th>
<th>Cohesive (%) (CAD/CAM block)</th>
<th>Cohesive (%) (resin composite)</th>
<th>Mix (%) (CAD/CAM block)</th>
<th>Mix (%) (Resin composite)</th>
<th>Total (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ER</td>
<td>60</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>40</td>
<td>100</td>
</tr>
<tr>
<td>ER+SHT</td>
<td>70</td>
<td>0</td>
<td>0</td>
<td>10</td>
<td>20</td>
<td>100</td>
</tr>
<tr>
<td>B</td>
<td>60</td>
<td>20</td>
<td>0</td>
<td>20</td>
<td>0</td>
<td>100</td>
</tr>
<tr>
<td>B+SHT</td>
<td>80</td>
<td>0</td>
<td>10</td>
<td>0</td>
<td>10</td>
<td>100</td>
</tr>
<tr>
<td>HF</td>
<td>60</td>
<td>10</td>
<td>0</td>
<td>0</td>
<td>30</td>
<td>100</td>
</tr>
<tr>
<td>HF+SHT</td>
<td>10</td>
<td>10</td>
<td>0</td>
<td>0</td>
<td>80</td>
<td>100</td>
</tr>
</tbody>
</table>

ER: Er:YAG laser treatment; SHT: silane heat treatment; B: bur roughening; HF: hydrofluoric acid etching.

RESULTS
The mean SBSs ± SD of all tested groups are presented in Table 2. Regarding the surface treatments, group ER showed significantly lower SBSs than group B and group HF (p < 0.05), while no significant differences in SBSs were determined between groups B and HF (p > 0.05). Furthermore, groups ER+SHT and B+SHT demonstrated significantly lower SBSs than group HF+SHT (p < 0.05), and no significant differences in SBSs was observed between groups ER+SHT and B+SHT (p > 0.05). Regarding the silane heat treatment, group B+SHT showed significantly lower SBSs than did group B (p < 0.05). No significant differences were found for the other tested groups (p > 0.05).
This study evaluated the effects of different surface treatments and the laser heat treatment of silane on the SBSs of a nano-cremic composite to repaired CAD/CAM hybrid blocks. The first null hypothesis, which stated that different surface treatments would not affect the SBS to repaired CAD/CAM hybrid blocks, was rejected because irrespective of silane heat treatment, the Er:YAG laser etching of CAD/CAM blocks led to lower SBSs than did HF etching. The second null hypothesis, which stated that laser heat treatment would not affect the shear bond strength to repaired CAD/CAM hybrid blocks, was rejected because the silane heat treatment negatively affected the SBSs to bur-roughened hybrid blocks, while it did not significantly affect the SBSs to the Er:YAG laser-treated and HF-etched groups.

Repairing the fractured site of dental CAD/CAM restorations is more cost-effective and preserves more remaining dental tissue than replacing a restoration completely. The repair procedure includes surface pretreatment of the restoration plus silane and adhesive application. The repair procedures currently used involve chemical and micromechanical bonding, and resin-ceramic adhesion is based on the surface treatment of the ceramic. Surface pretreatments, such as diamond bur abrasion, acid etching and laser etching, have been investigated by simulating the intraoral repair process in the laboratory. In earlier approaches, diamond burs were used to create grooves and undercuts for macromechanical bonding. A silane coupling agent increases the wettability of the ceramic surface and creates effective chemical adhesion between composite resin and ceramic. Silane has a dual function of bonding with both the methacrylate groups of the resin and the silicon dioxide groups of the ceramic. Universal dental adhesives have been marketed with a multi-purpose formulation that may adhere to metal, ceramic, and com-

Fig 2 Representative SEM images of all tested groups at 500X and 1000X.

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Conclusions regarding surface treatments, without silane heat treatment, the Er:YAG laser resulted in significantly lower SBSs than did bur and HF. With silane heat treatment, the Er:YAG and bur presented significantly lower SBSs compared to HF. Regarding the presence of the silane heat treatment, using a bur with silane heat treatment resulted in significantly lower SBSs than did a bur without silane heat treatment.

This study employed an Er:YAG laser at a 20 Hz repetition rate at 5W for 30 s to pretreat surfaces. Thus, further studies should focus on the effect of different pulse frequency, duration and output powers of the Er:YAG laser on the bond strength to repaired hybrid CAD/CAM blocks with the protocol of both silane heat treatment and surface treatment methods.

Acknowledgments

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materials when hydrofluoric acid and Er:YAG laser were 
used as surface pretreatment methods. When surfaces 
are pretreated with bur roughening, silane heat treatment 
may not be advisable.