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Effect of Argon Plasma Surface Treatment on Bond Strength of Resin Composite Repair

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Clinical Relevance

The use of argon plasma application as a surface treatment did not improve the bond strength of composite repairs. The traditional protocol combining sandblasting, silanization, and the use of a hydrophobic bonding adhesive was the most reliable method.

SUMMARY

Objectives: This study evaluated the effect of argon plasma treatment (PLA) and its combination with sandblasting (SAN), silanization (SIL), and hydrophobic bonding resin (HBR) application on the micro-shear bond strength of water-aged restorative resin composite to a newly placed composite, simulating restoration repair.

Methods and Materials: Forty-five light-cured composite plates (20-mm long × 20-mm wide × 4-mm thick) were fabricated using a hybrid composite and stored at 37°C in distilled water for six months. The aged composite surfaces were treated according to the following experimental groups, varying both treatment and order of application: 1) SAN + SIL + HBR (control), 2) SAN + PLA for 30 seconds + SIL + HBR, 3) SAN + SIL + PLA + HBR, 4) PLA + SIL + HBR, 5) PLA + SIL, 6) PLA + HBR, 7) SIL + PLA + HBR, 8) SIL + PLA, and 9) PLA. After the surface treatments, four fresh resin composite cylinders (1.5-mm high × 1.5-mm diameter) of the same composite were built on each aged composite surface using a silicone mold. After water storage for 24 hours or one year, the specimens were submitted to shear bond strength testing. Data were statistically analyzed by two-way analysis of variance and Tukey's test (5%).

Results: Groups 1, 2, and 4 presented significantly higher bond strength means at 24 hours, although group 4 did not differ from group 7. Groups 5, 8, and 9 demonstrated significantly lower means than the other groups. Even

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though groups 1 and 2 had a significant bond strength reduction after 1 year, they still demonstrated higher bond strength at one year of storage.

Conclusions: While PLA application combined with surface treatment methods demonstrated high bond strength results, this treatment alone was not as beneficial as other methods that included SAN, SIL and HBR.

INTRODUCTION

Resin composite restoration repairs are common procedures in general practice. The repair can be performed in both recently placed restorations and old ones.[1-5] In just-placed restorations, the repair is indicated to correct and adjust color, anatomy, lack of margin sealing, or early small composite fractures. In these cases, the prognosis of success is high if the composite restoration is recently placed and unreacted monomers are still available for chemical bonding with the fresh composite increment.[6-8]

Conversely, for older composite restorations, the unreacted monomers are leached out, and there are no chemical bonds available for bonding with a fresh composite. To overcome this lack of chemical bonding between the old and new composites, sandblasting with aluminum oxide has been recommended to increase the superficial roughness and create micromechanical retention at the surface of the old resin composite.[9-11] In addition, silane application is recommended to bond the monomers from the fluid adhesive resin layer and composite to the exposed glass filler of the old composite through a chemical reaction.[9-17]

Atmospheric pressure plasma (PLA) has been used to chemically destabilize surfaces and bond to another matter. The PLA is formed by reactive species that are created by the interaction between ions and electrons of argon PLA; thus, when the PLA micro-atmosphere reaches the sample surface, the reactive species breaks the stabilized bonds and forms polar groups at the surface. In summary, PLA improves the reactive level of surfaces by opening up the chemical sites to future bonds.[18-22]

Argon PLA applied as a surface treatment of old composites may have the ability to enhance the composite restoration repair and improve its longevity.[1,4] Heretofore, no study has been performed to investigate advantages in using argon PLA for resin composite repair. The purpose of this study was to evaluate the effect of argon PLA and its combination with sandblasting (SAN), silanization (SIL), and hydrophobic bonding resin (HBR) treatments on the micro-shear bond strength of water-aged restorative resin composite to a newly placed composite, simulating restoration repair, after 24 hours and one year of storage. The hypothesis tested was that PLA used as a surface treatment would improve the bond strength of repaired composites.

METHODS AND MATERIALS

Specimen Preparation

Forty-five composite (Charisma, Heraeus Kulzer, Germany, shade A2, lot No. 010611) plates (20-mm long × 20-mm wide × 4-mm thick) were fabricated using silicon molds and light activated with a polywave light-curing unit (886 mW/cm^2 irradiance) for 40 seconds (Valo, Ultradent Product Inc, South Jordan, UT, USA). The composite plates were kept in distilled water at 37°C for six months. After aging, samples were polished with 600-grit silicon carbide paper (Norton, Vinhedo, SP, Brazil) and submitted to ultrasonic cleaning (USC 1400, Unique Ind Com Prod Eletr Ltda, Indaiatuba, SP, Brazil) with distilled water for five minutes.

Experimental Groups

Composite plates were divided into nine groups (n=5), varying surface treatments and application order, as described in Table 1. The PLA equipment used was the Surface Plasma Tool Model SAP–Lab applications (Surface-Engineering and Plasma Solution LTDA, Campinas, SP, Brazil). Application time of the argon PLA was 30 seconds as the working gas (Praxair 4.8, White Martins Gases Ind SA, Rio de Janeiro, RJ, Brazil), with an output of 1.0 L/min.

Groups that were sandblasted received the following protocol: air abrasion with 50 μm aluminum oxide particles using a sandblasting unit (Microetcher, Danville Materials, San Ramon, CA, USA) for 10 seconds, 10 mm away from the surface at 60 psi. Afterward, the plates were submitted to an ultrasonic bath (Unique Ind Com Prod Eletr Ltda) in distilled water for five minutes, followed by uniform air drying of the samples using an air syringe for 30 seconds.

The silane-coupling agent used was the Ceramic Primer (lot No. N522201, 3M ESPE, St Paul, MN, USA), and the HBR was the adhesive (Adper Scotchbond Multi-Purpose, lot No., N5154423M ESPE). A drop of the silane was deposited in a mixing well, from which the silane was collected by a micro-brush for application on the composite plates. A uniform layer of liquid silane was applied to the

Table 1: *Experimental Groups According to Surface Treatment and Order of Application*

Group	Treatment
1	SAN + SIL + HBR (control)
2	SAN + PLA + SIL + HBR
3	SAN + SIL + PLA + HBR
4	PLA + SIL + HBR
5	PLA + SIL
6	PLA + HBR
7	SIL + PLA + HBR
8	SIL + PLA
9	PLA

Abbreviations: PLA, plasma treatment; SAN, sandblasting; SIL, silanization; HBR, hydrophobic bonding resin.

plates, which formed a thin layer of the silane on the surface of the sample after drying and evaporation of water and other solvents. Silane was applied, kept undisturbed for 15 seconds, and air dried for 10 seconds. HBR application consisted of a uniform coating of adhesive applied with a micro-brush, followed by light activation for 10 seconds.

Micro-shear Bond Strength Test

Silicone molds (Aquasil Ultra Putty, Dentsply Caulk, Milford, DE, USA) were positioned over the treated, aged composite plate, and the fresh composite (same commercial composite and lot number) was inserted into the mold (1.5-mm high × 1.5-mm diameter). The composite was light activated for 20 seconds, and the mold was carefully removed. Four composite cylinders were manufactured onto each plate. Samples were stored in water at 37°C for 24 hours or one year.

For micro-shear bond strength testing, each composite plate was placed with cyanoacrylate glue (Model Repair II Blue, Sankin Industry Co, Tokyo, Japan) to a jig attached to a universal testing machine (EZ Test, Shimadzu Corp, Kyoto, Japan) and subjected to a bond strength test at a crosshead speed of 0.5 mm/min. Two resin cylinders of each plate were tested after 24 hours and the two remaining after one year. The shear load was applied at the base of the resin cylinders with a loop wire (0.2-mm diameter). Bond strength data were calculated using the peak of loading failure divided by specimen surface area, and means were obtained in MPa. A single failure stress value for each composite plate was calculated by averaging the values of the two resin cylinders from the same plate and evaluation time. Bond strength data were expressed in MPa and statistically analyzed by two-way analysis of variance (ANOVA; repeated-measures approach) and Tukey post hoc test (with a preset alpha of 0.05).

The fractured surfaces were gold coated (MED 010, Balzers, Balzer, Liechtenstein) and examined using a scanning electron microscope (JSM-5600LV, JEOL Inc, Tokyo, Japan) at 35× and 200× magnifications (voltage, 15 kV; beam width, 25-30 nm; working distance, 10-20 mm). The failure mode of each specimen was classified as follows: 1) adhesive failure at the old-new composite interface and 2) cohesive failure within old composite.

RESULTS

Bond strength results for 24 hours and one-year storage are presented in Table 2. The two-way ANOVA results indicated that treatment ($p<0.0001$) and evaluation time ($p<0.0001$) factors significantly influenced bond strength values, with significant interaction between the factors

Table 2: *Bond Strength (SD) of New Composite to Old for All Experimental Groups (MPa)*[a]

Group	Treatment	Evaluation Time	
		24 Hours	1 Year
1	SAN + SIL + HBR (control)	32.7 (1.7) Aa	23.0 (3.2) Ba
2	SAN + PLA + SIL + HBR	34.6 (2.8) Aa	20.3 (4.3) Bab
3	SAN + SIL + PLA + HBR	19.7 (4.2) Ac	18.3 (4.0) Aabc
4	PLA + SIL + HBR	30.2 (3.2) Aab	15.9 (2.1) Bbcd
5	PLA + SIL	8.9 (3.2) Ad	5.1 (0.4) Ae
6	PLA + HBR	20.6 (1.7) Ac	13.8 (3.4) Bcd
7	SIL + PLA + HBR	24.1 (1.2) Abc	11.8 (2.3) Bd
8	SIL + PLA	7.0 (0.7) Ad	2.8 (1.8) Ae
9	PLA	4 6 (0.9) Ad	1.8 (0.6) Ae

Abbreviations: PLA, plasma; SAN, sandblasting; SIL, silanization; HBR, hydrophobic bonding resin.
[a] *Means followed by different lowercase letters (column: comparing groups within the same evaluation time) and different uppercase letters (row: comparing evaluating times within the same group) represent significant statistical differences according to Tukey test ($p \leq 0.05$)*

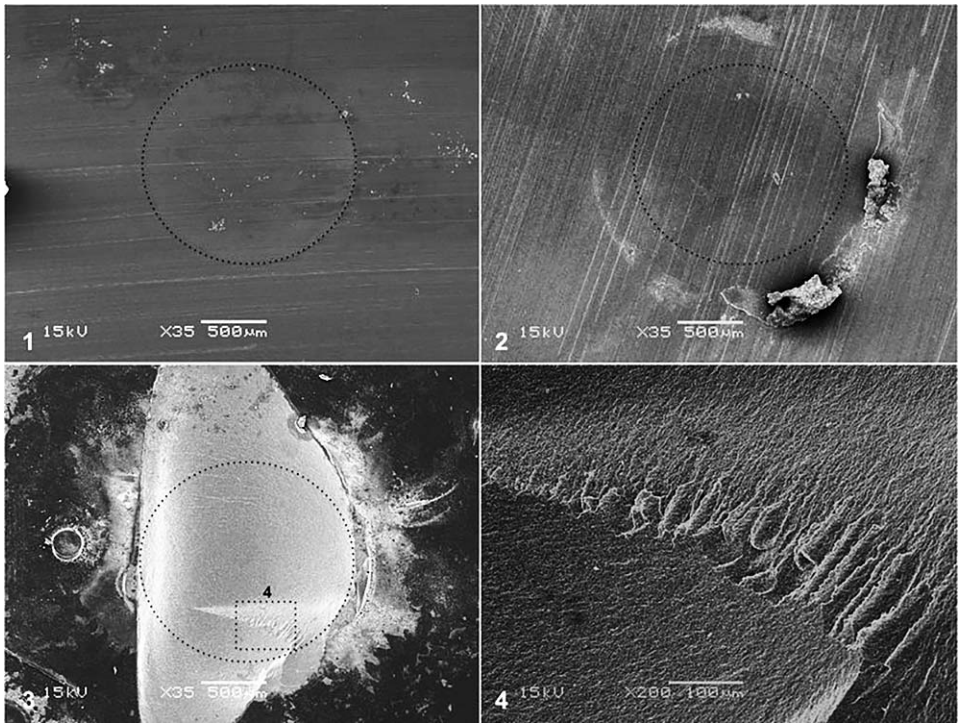

Figure 1. *Scanning electron microscopy photomicrograph illustrating an adhesive failure along the aged composite surface for group 9 (PLA), tested after 24 hours of water storage. Original magnification 35×.*

Figure 2. *Scanning electron microscopy photomicrograph illustrating an adhesive failure along the aged composite surface for group 5 (PLA + SIL), tested after 24 hours of water storage. Original magnification 35×.*

Figure 3. *Scanning electron microscopy photomicrograph illustrating a cohesive failure within the aged composite for group 1, tested after one year of water storage. Original magnification 35×.*

Figure 4. *Higher magnification of Figure 3 showing the aged composite cohesive fracture. Original magnification 200×.*

($p<0.0001$). At 24 hours, groups 1 (control, SAN + SIL + HBR), 2 (SAN + PLA + SIL + HBR), and 4 (PLA + SIL +HBR) presented higher bond strength (between 34.6 and 30.2 MPa), although group 4 did not differ statistically from group 7 (SIL + PLA + HBR). Groups 5 (PLA + SIL), 8 (SIL + PLA), and 9 (PLA), exhibited the lowest bond strength means (between 4.6 and 8.9 MPa), being statistically different from all other groups. Groups 3 (SAN + SIL + PLA + HBR), 6 (PLA + HBR), and 7 exhibited intermediate bond strength values, which were not significantly different from each other.

After 1 year, when storage time is compared, groups 3 and 5 did not demonstrate significant statistical reduction in bond strength. Even though groups 1 and 2 had a significant bond strength reduction after 1 year, they still demonstrated a higher bond strength at one year of water storage (20.3 and 23.0 MPa, respectively). However, group 2 itself did not differ statistically from groups 3 and 4. Groups 5, 8, and 9 maintained significantly lower bond strength means after one year (between 1.8 and 5.1 MPa) compared with the other groups.

Representative images of the failure modes are presented in Figures 1 to 4. Groups 5 (PLA + SIL), 6 (PLA + HBR), and 9 (PLA) had 100% adhesive failures at both composite interfaces, whereas groups 2 (SAN + PLA + SIL + HBR) and 4 (PLA + SIL + HBR) presented 100% cohesive failure within the old composite at both times (Table 3). An increase in adhesive failures was observed for group 7 (SIL + PLA + HBR; from 40% to 100%), whereas increases in cohesive fractures (within the old composite) were noted for groups 3 (SAN + SIL + PLA + HBR / from 40% to 80%) and 8 (SIL + PLA / from 60% to 80%) after one year of storage. The control group (SAN + SIL + HBR) presented 20% adhesive failures and 80% cohesive fractures within the old composite, which did not change after one year.

DISCUSSION

The hypothesis that PLA used as a surface treatment would improve the bond strength of repaired composites was rejected. PLA application alone or combined with SAN, SIL, and HBR did not result in higher bond strength when compared with the control (SAN + SIL + HBR). Nevertheless, the PLA treatment associated with SIL and HBR demonstrated similar results to control at 24 hours, despite the use of SAN or not (groups 2 and 4). Only after one-year of storage was the use of SAN combined with PLA shown to be relevant (group 2), obtaining similar results to the control (group 1).

This gold standard control composite repair technique was used after carrying out a bibliographical survey on this subject, which indicated that the technique and materials used have obtained the best

Table 3: *Failure Modes (%) Among Experimental Groups*

Group	Treatment	Evaluation Time	
		24 Hours AD/CO	1 Year AD/CO
1	SAN + SIL + HBR (control)	20/80	20/80
2	SAN + PLA + SIL + HBR	0/100	0/100
3	SAN + SIL + PLA + HBR	60/40	20/80
4	PLA + SIL + HBR	0/100	0/100
5	PLA + SIL	100/0	100/0
6	PLA + HBR	100/0	100/0
7	SIL + PLA + HBR	40/60	100/0
8	SIL + PLA	40/60	20/80
9	PLA	100/0	100/0

Abbreviations: AD, adhesive failure; CO, cohesive failure; PLA, plasma; SAN, sandblasting; SIL, silanization; HBR, hydrophobic bonding resin.

results.[9-11,14,17,23-25] However, the comparison of the results must be done carefully when the studies have used different types of composites, as well as surface treatment protocols (mechanical and chemical) and materials (conditioners, adhesives, and silanes).[7,8,12,13,15,16,26,27]

To obtain better clinical outcomes, some authors have suggested that knowing the composition of the composite to be repaired is important for the success of the technique. In addition, the different types of composites (microhybrid, nanohybrid, or nanofilled) seem to produce different results according studies.[7,13,15] The presence of fillers in a polymer network can greatly affect water uptake and dissolution, possibly in direct relation to its proportion as it reduces the overall volume of the absorbing polymer.[28] Another factor that is critical to the success of the repair technique is the age of the restoration. Thus, better repair adhesion between the placed restoration and the new composite is found in recently placed restorations.[6,8,11]

In the current study, samples were aged by immersion in distilled water for six months before repairing and testing, which was the same time used by Rathke and others[25] and Celik and others.[12] Artificial aging in water was performed to leach unreacted monomer layer formed due to inhibition by oxygen at the surface of the composite and to simulate oral conditions. Sorption and solubility may serve as precursors to a variety of chemical and physical processes that create biological concerns as well as produce deleterious effects on the structure and function of the dental composite. These effects may include volumetric changes such as swelling, physical changes such as plasticization and softening, and chemical changes such as oxidation and hydrolysis, all processes that help to degrade the material.[28] Other studies have aged the samples for shorter times, such as seven days in saline solution,[16] seven days or one month in water,[8,29] and three months in artificial saliva,[26] while Staxrud and Dahl,[17] in 2015, repaired composite samples that were six years old. Accelerated aging of 300 hours in a weathering tester,[11] 5,000 thermocycles,[13,15] and a combination of thermocycling and water storage also have been used.[27]

The air abrasion promoted by SAN with aluminum oxide particles creates micro retention and a uniformly rough surface, which increases the superficial area that interacts with a bonding agent and the new composite increment.[9,10,14,16,25] According to different studies, in the repair technique, SAN must be used to treat composites, corroborating the results of this study. Actually, for 24-hour testing, the use of SAN was not primordial for achieving high bond strength. Group 4, which used only PLA + SIL + HBR, had similar results to the application of the same protocol using SAN with or without PLA treatment. Nevertheless, after one year of water storage, the group that did not use SAN demonstrated inferior outcomes compared with these same groups. Summarizing the results, when SAN was used in combination with PLA, SIL, and HBR, regardless of the sequence of the use of PLA, SIL, and HBR, higher bond strengths were observed after one year. This behavior demonstrates that the chemical bonding created by PLA and SIL was initially adequate, but after hydrolytic degradation of this interface, the mechanical approach of SAN was necessary. However, in consideration of the safety of the patient and professionals during intraoral sandblasting, an aspirator device and rubber dam isolation must be used, which limits its clinical use.[30] As an alternative, rotary instruments may also be able to roughen the old composite restoration surface, and studies have shown significant improvement in bond strength following surface treatment with diamond burs.[11,16]

The argon PLA application proposed in this study modifies the hydrophilicity and the reactivity of the surfaces but does not promote mechanical changes.[18-21] The reactive species produced by PLA with the purpose of enhancing the reactive level of the composite surface was not enough to improve the bond strength when used alone (group 9). The use of PLA in combination with SAN, SIL, and HBR (groups 2 and 4) did not interfere with bond strength results; however, when PLA was used with only SIL and HBR (groups 5 and 6), lower bond strengths

were observed compared with SAN, SIL, and HBR used together. Substituting SAN (group 1) with PLA treatment (group 4) did not generate a significant difference at 24 hours; however, the bond strength was reduced significantly in approximately 50% of group 4 and 25% of the control group after one year; these results were statistically different, as previously discussed.

Because there is a low amount of unreacted monomers in the surface of aged composites, another chemical reaction for creating a chemical bonding mechanism is important for composite repair techniques. Silanes consist of a silanol group (or alkoxy group) that chemically reacts with hydroxyl groups from silica-based filler particles and the methacrylate group that co-polymerizes with the resin matrix of the new composite increment.[31,32] Some studies have indicated use of silane in an attempt to improve the bond strength of the composite repair; however, the silanization efficacy depends on the type of filler particles and the amount silica available at the surface.[9,17,24,33]

The use of bonding agent has been recommended for composite repair especially because when the composite surface is air abraded or roughened, the fluid adhesive resin is able to penetrate into formed micro-porosities. After light activation, the bonding resin remains attached and interlocked to the surface.[6,15] In this study, for groups 5, 8, and 9, in which HBR was not used, the lowest bond strength values were found at both evaluation times. Various studies have recommended the use of a bonding agent[14,16,23,25-27,29,33]; however, they used different types and generations of adhesive systems. More hydrophobic fluid adhesive resins are preferred, because more hydrophilic adhesives tend to result in early degradation of the adhesive layer with decreasing of bond strength even after short-term evaluation.[14]

The long-term storage time of this study was one year, which was fundamental for analyzing the behavior of different treatments over time. The groups that obtained higher bond strength values (between 34.6 and 20.6 MPa) at 24 hours (groups 1, 2, 4, 6, and 7) showed a bond strength reduction after one year. Although groups 2 (SAN + PLA + SIL + HBR) and control (group 1: SAN + SIL + HBR) presented significant bond strength reduction after long-term storage, they still demonstrated higher bond strength at one year (20.3 and 23.0 MPa, respectively). Group 3 (SAN + SIL + PLA + HBR/ 18.3 MPa), for which no bond strength reduction was observed, did not differ from groups 1 and 2 at one year and had the best outcomes in this study. Two other studies that evaluated the influence of surface treatments on repair bond strength of aged composites showed that the use of aluminum oxide sandblasting and adhesive application yielded stable bond strength after six months.[14,16] Conversely, Staxrud and Dahl,[27] in 2011, reported that the thermocycling method used for aging of the samples affected the bond strength of fresh composite to one year of age, which could compromise the durability of composite repair.[17]

Water storage for one year influenced the failure pattern for only three groups (3, 7, and 8), with an increase in the incidence of adhesive or cohesive failures, depending on the experimental group (Table 3). Groups 5, 6, and 9, which were not treated by sandblasting but rather with PLA, showed 100% adhesive failure. The cohesive failures within the aged composite resulted in part from the strong interaction between materials and the micro-shear bond strength test design, which favors this type of fracture at the adherent structure.[34-36]

This study used a shear bond strength test with a circular bonding area of 1.77 mm^2, which is considered a micro-shear method because its bonded area is lower than 3 mm^2 and is more favorable than a "macro-size" one.[37-39] Bond strength tests such as shear and tensile present advantages, disadvantages, and limitations, and no single bond strength methodology provides a strong clinical correlation.[34,39-41] The micro-tensile method is preferred to evaluate the bond strength of adhesive/composite resins in enamel and dentin, but in studies of resin cement adhesion in zirconia, other types of ceramics, indirect resin, as well as composite resin repair studies, the shear test is still used.[7,11,17,27,29,42-44] For the micro-tensile test, the load is more uniformly distributed at the interface, which is the main advantage of this test, whereas some authors have reported that the shear bond strength method generates a complex stress field at the interface, and adhesive failure occurs predominantly as a result of tensile stresses induced by bending moments.[34-41]

Some studies have discussed the teaching, feasibility, and durability of repairs of composite restorations,[2,4,9] and the reported results showed that the repair technique can increase the clinical longevity of the restorations.[3,5] However, others have criticized the durability of composite restoration repairs, following the unsuitable results obtained and the lack of a specific protocol.[1,13,24] As this study also demonstrated a significant bond strength reduction

between new and old composite materials for most of the groups after one year, perhaps additional macroretentions in the cavity preparation may be necessary to ensure longer repaired restoration durability, especially in the cases of older composite restorations.

CONCLUSIONS

Within the limitation of the shear bond strength method, PLA used alone as a surface treatment for resin composite repair was not as beneficial as other protocols that included SAN, SIL, and HBR in combination or not with PLA. When SAN was substituted by PLA as surface treatment, it presented similar outcomes when associated with SIL and HBR after 24 hours of storage; however, after a long storage period, SAN was demonstrated to be essential to the bond strength of repaired resin composites. The bond strength stability of the interface between aged and fresh composite was not determined in this study, at least for the protocols that presented acceptable initial bond strength values.

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Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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