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Shrinkage stress and mechanical properties of photoactivated composite resin using the argon ion laser

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Abstract The objective of this study was to verify the influence of photoactivation with the argon ion laser on shrinkage stress (SS), followed by evaluation of Vickers microhardness (VM), percentage of maximum hardness (PMH), flexural strength (FS), and flexural modulus (FM) of a composite resin. The study groups were: L1-laser at 200 mW for 10 seconds; L2-laser at 200 mW for 20 seconds; L3-laser at 250 mW for 10 seconds; L4-laser at 250 mW for 20 seconds; H-halogen light at 275 mW for 20 seconds. Data were analyzed by ANOVA/Tukey's test ($\alpha = 5\%$). The values of SS (MPa) were statistically lower for the group L3 (1.3)c, followed by groups L1 (2.7)b, L4 (3.4)a, b, L2 (3.7)a, and H (4.5)a. There was no difference in the values of VM when the same time of photoactivation was used, with respective values being L1 = 70.1a, L2 = 78.1b, L3 = 69.9a, L4 = 78.1b and H = 79.9b. All groups showed a PMH of at least 80%. Only the group L1 showed differences in FS (MPa) and FM (GPa), the respective values of 86.2 and 5.4 being lower. Therefore, the use of argon ion laser had influenced the composite resin polymerization. The L3 group presented adequate mechanical properties and minimum SS, reducing the clinical working time for photoactivation of restorations with the tested resin by 50%.

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1 Introduction

The polymerization process of a composite resin occurs by the conversion of the monomer molecules of the matrix into a polymer chain, followed by a reduction of the space between these molecules, which then occupy a smaller volume than the initial. This reduction in the total volume of the material is known as the shrinkage of polymerization. Although the composite resin is considered the best esthetic direct restorative material, excessive shrinkage of polymerization is one of the main factors that contribute to the failure of restorations [1, 2]. This occurs because the contraction of dental composite during polymerization generates stress within the material which is transferred to the interfacial bond between the tooth and the restorative system. The magnitude of the stress is influenced by several factors, including the overall shrinkage, the curing rate, the elastic modulus, and the stress relieving characteristics of the composite restoration [3].

To photoactivate the composite resin, halogen light is the main used source, but the heat generated by the wavelengths that are not used can reduce the average life of the lamp

Table 1 Material used in this study (information provided by the manufacturer)

Composite	Composition	Manufacturer	Description	Batch
Filtek Z-250	Bis-GMA, UDMA, Bis-HEMA, Zirconium, Silex	3M ESPE (St Paul, MN, USA)	Hybrid resin A3 color	6FB

and other components, and cause damage to the pulp tissue [4]. In an attempt to minimize these effects, new devices with argon ion laser sources were proposed. The argon ion laser emits light energy in the blue and green bands of visible light. The wavelength of blue light is 488 nm and is capable of photoactivating resin composites, a characteristic that no other laser has. The wavelength of green light is 514.5 nm and is used for procedures in soft tissue and blood coagulation [5]. These characteristics of its monochromatic coherent and collimated light, promote greater penetration in depth, generating less heat [6].

Unlike the self-curing resin which polymerizes in a uniform way, provided that the mixture is efficient, the light-curing resins polymerize only where the light reaches them. This means that the light is absorbed, scattered and, consequently, attenuated during its passage through the mass of resin [7]. This is the main cause of conflict between the two sources of light because the laser emits collimated light which results in the same power density independently of the distance, in contrast to the halogen light, power density of which decreases with distance [8].

Although the best mechanical properties of composites are obtained with the argon ion laser [6, 9, 10], the values of microhardness obtained with this new source of light are still very controversial in the literature [8, 11–13]. Furthermore, there are no studies evaluating the SS of the composite resin photoactivated with the argon ion laser, or studies linking this stress with the mechanical properties of composite resin.

This way, considering the constant development of dental materials and the improvement of techniques for evaluation of their properties, studies are always necessary to consolidate new concepts and assess their likely clinical performance, evaluating possible alternatives to improve them. Therefore, analyzing the SS, followed by an evaluation of VM, PMH, FS and FM, can be an alternative to defining the real effectiveness of photoactivation of a composite resin with the argon ion laser.

2 Materials and methods

The samples were made from a hybrid resin (Filtek^{TM} Z-250, 3M ESPE, St Paul, MN, USA). Table 1 presents the material used in this study. The samples were divided into 5 groups, with 5 samples for each variable (Table 2).

Table 2 Groups of study

Groups $n = 5$	Light source	Time of exposure
L1	Argon ion laser at 200 mW	10 seconds
L2	Argon ion laser at 200 mW	20 seconds
L3	Argon ion laser at 250 mW	10 seconds
L4	Argon ion laser at 250 mW	20 seconds
H	Halogen light	20 seconds

Table 3 Parameters used in this study

	L1	L2	L3	L4	H
Power (mW)	200	200	250	250	275
Time (s)	10	20	10	20	20
Intensity (mW/cm^2)	714.28	714.28	892.85	892.85	550
Dose (J/cm^2)	7.14	14.28	8.92	17.84	11

For photoactivation, the argon ion laser (AccuCure 3000, LaserMed, Salt Lake, UT, USA) and halogen light (Degulux Soft-Start, Degussa-Hulls, Dusseldorf, Germany) were utilized with the parameters described in Table 3. The intensity was measured with a power meter PD300-SH (Ophir Optronics LTD., Israel) before photoactivating each sample with argon ion laser, and with a radiometer (Demetron, Kerr, USA) in the case of halogen light, to avoid any possible decays of the source of light or the influence of other compounds on the results.

2.1 Shrinkage stress

The end of a Pyrex rod (5 mm in diameter and 13 mm in length) was polished with alumina (250 μm), treated with a silane ceramic primer and 1 layer of unfilled resin (Scotchbond Multipurpose, 3M ESPE, St Paul, MN, USA) and photoactivated for 20 seconds with halogen light. One of the glass rods had its lateral surface protected by Scotch tape; it was then attached to a chuck connected to the load cell of a universal testing machine (Instron model 5565, Canton, MA, USA). The other rod was attached to a steel fixture

that was connected to the other end of the testing machine; the steel fixture allowed the tip of the light-curing unit to be kept in contact with the opposing side of the glass rod. The composite was placed on the prepared surfaces between 2 rods, using a 1 mm fixed distance. The ratio of bonded to unbounded surface area (configuration factor) was 2.5 with this arrangement. An extensometer (Instron, Canton, MA, USA) was attached directly to the glass rods in order to keep the sample height constant, simulating a low compliance situation. Photoactivation was performed in contact with the polished surface of the lower glass rod. The SS was recorded for 5 minutes. Maximum force was divided by the cross-sectional area to calculate the average axial stress. Five samples were tested for each experimental group.

2.2 Vickers microhardness

The test samples were made using circular black polypropylene matrixes with a 4 mm diameter cavity and a height of 2 mm [7]. Each matrix was supported on a glass slide to obtain a smooth surface on the resin composite. A piece of black cardboard was placed under the glass slide in order to avoid light reflection from the bottom. The resin was inserted in a single increment, completely filling the matrix cavity. To smooth the surface where the light was applied, a strip of polyester was used with a glass slide over it. The glass slide was removed prior to photoactivation. In all groups, the photoactivation was done with the active center of the tip of the apparatus positioned as close as possible to the sample, coinciding with its center and without moving it [14]. When the polymerization was complete, each sample was kept dry and was stored in a black receptacle at 37°C for seven days. After storage, each sample, while still in the matrix, was taken to a microhardness instrument (HMV 2000, Shimadzu, Kyoto, Japan), where the irradiated surface (upper) and the opposite surface to the irradiated (lower) were examined. Five samples of each group experimental were prepared. Each sample received five VM measurements: one central (defined by the location of light application) and the other four at a distance of approximately 100 μm from the central location under a 50 gf load for 15 seconds. For the statistical analysis, the averages from 5 measurements for each sample were calculated, resulting in a total of 25 data for the irradiated surface and 25 data for the opposite surface.

2.3 Percentage of maximum hardness

To assess the quality of polymerization related to the maximum hardness of the resin for each group, the percentages corresponding to this value, called PMH, were calculated; they relate the opposite surface from the irradiated by light (lower) with the irradiated surface (upper).

2.4 Flexural strength and flexural modulus

The test was performed according to ISO 404912 [15], and bar shaped samples of reduced dimensions ($1 \times 2 \times 10$ mm–height × width × length) were adopted [16]. They were prepared with a stainless steel mold covered in an appropriate layer of black ink in which the composite resin was inserted and sandwiched between strips of polyester and pressed glass slides. Following the photoactivation, the samples were stored dry in a black receptacle at 37 °C for 24 h. Five samples were made for each of the 5 experimental conditions. Three-point bending test was carried out in a universal machine (Kratos Ltda, model K 2000MP, Cotia, SP, Brazil) with 6 mm span between the supports, at a crosshead speed of 0.5 mm/min. The FS of the samples was calculated according to the following equation: $R_f = 3LF/2bh^2$, where R_f is the FS (MPa), L is the distance between the supports (mm), F is the load at fracture (N), b is the sample width (mm), and h is the height of the sample that was individually measured with a 0.01 mm precision digital caliper (Absolute Digimatic, Mitutoyo, Tokyo, Japan). The averages of the FM values were obtained during the test of FS, through software TRACOMP-W95 (TRCV606).

The data from the five groups were analyzed by one-way ANOVA, separately for each variable (SS, VM, PMH, FS, and FM). Tukey's test was used for multiple comparisons, with global significance level of 5%.

3 Results

There was a statistically significant difference at the 5% level related to SS, evident also in the mean values (Fig. 1). The SS increased sharply at the initial stage, and the slope became more gradual near 50 seconds until reaching a plateau after 200 seconds. The L3 group (1.27 ± 0.59) had the least amount of SS, followed by the group L1 (2.66 ± 0.44) which was statistically similar to the L4 group (3.45 ± 0.75), which itself was similar to the groups L2 (3.67 ± 0.23) and H (4.52 ± 0.71).

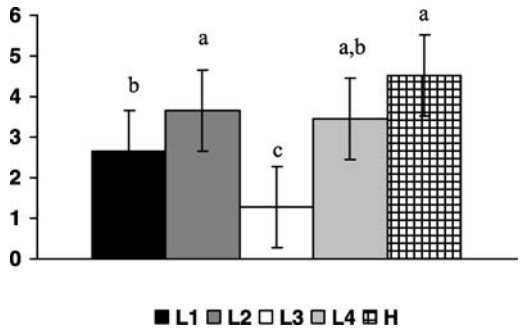

Fig. 1 Average SS (MPa). Same letter represents statistic similarity ($p < 0.05$)

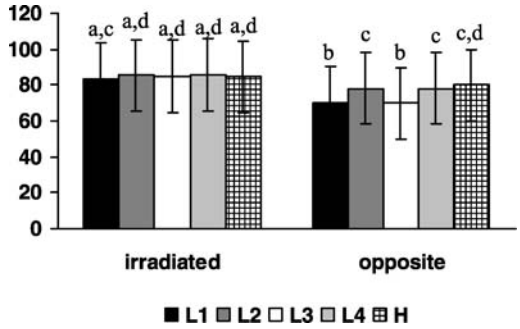

Fig. 2 Average VM in the irradiated surface and in the opposite surface to the irradiation. Same letter represents statistic similarity ($p < 0.05$)

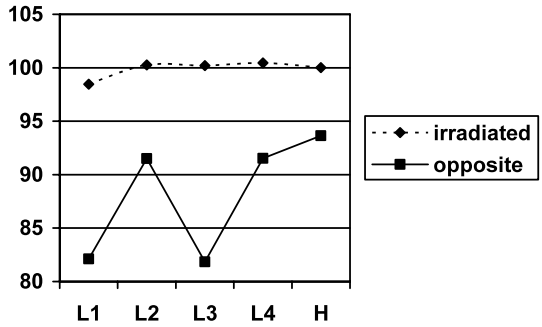

Fig. 3 PMH (%) in the irradiated surface and in the opposite surface to the irradiation (2 mm thickness)

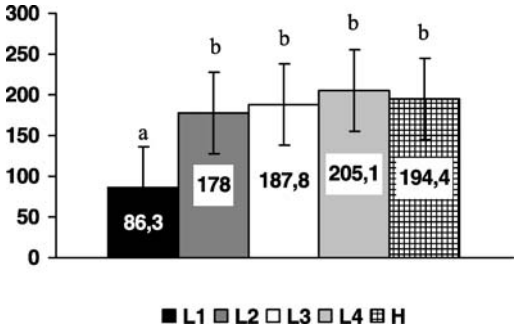

Fig. 4 Average FS (MPa). Same letter represents statistic similarity ($p < 0.05$)

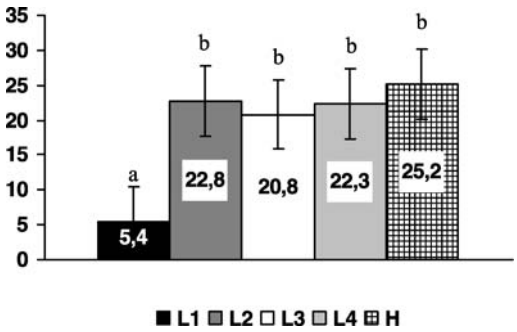

Fig. 5 Average FM (GPa). Same letter represents statistic similarity ($p < 0.05$)

No significant differences were found in the values of VM in the irradiated surface. For the surface opposite from the irradiation, there was no statistically significant difference in VM when the same time was used; the time being 20 seconds longer (Fig. 2). When comparing the irradiated and opposite surfaces, there was always a decrease in the values of VM of the opposite surface to the corresponding irradiated surface, except for the halogen light.

Regarding PMH, as the argon ion laser (at 200 mW for 20 s) promoted greater hardness on the irradiated surface, the average (85.4) was used as the reference value of greater hardness obtained (100%). All groups reached a PMH of at least 80% (Fig. 3).

Regarding FS and FM, only the group L1 showed a statistically significant difference from other groups, the respective values being lower (Figs. 4 and 5).

4 Discussion

According to Ferracane and Mitchem [3], the low contraction of a composite resin promotes the lowest stress on the interfacial bond, and this resulted in a smaller marginal gap formation and a lower leakage. The least value of SS for photoactivated composite resin with argon ion laser at 250 mW for 10 seconds was probably due to the lower value of VM, with the depth of 2 mm in that group, although there has been no significant difference in the VM of this group with the photoactivated group with argon ion laser at 200 mW for 10 seconds.

Several studies have demonstrated that a composite's degree of conversion and, consequently, its physical properties are directly related to the total amount of light delivered to the polymer, referred to as the radiant exposure [17–19]. In fact, nonlinear relationships between radiant exposure, FM and microhardness have been demonstrated [20, 21]. In this study, although there has been no significant difference in the values of VM, within 2 mm of depth, between sources of light when the same time was used, the time of 20 seconds being superior, no significant difference was observed in the values of FR and FM between the group light-cured with argon ion laser at 250 mW for 10 seconds and photoactivated groups for 20 seconds (lasers at 200 and 250 mW and halogen light). A possible explanation for such a finding is that the material's properties noticeably improve up to a certain threshold of polymer network formation. Above that point, increases in conversion will not significantly affect its mechanical behavior [22] but can affect the SS.

To measure the VM, the samples of 2 mm of in thickness were made, and the photoactivation was performed at a distance of 0 mm from the tip of the equipment to the surface

of the sample. To Rueggeberg et al. [7] there was no difference in VM values up to a depth of 2 mm, and, according to Rode et al. [13], an increase in distance of the light source from the resin composite surface promoted a decrease in the microhardness values and in the degree of conversion for the three types of light sources studied (halogen light, argon ion laser and LED). The quality of cure or polymerization is determined by the degree of conversion of monomer into polymer, indicating the amount of reactive metacrylate groups that reacted with each other. Consequently, there is a high degree of association between the success of the restoration and the ability of polymerization of the visible light radiated within a certain period of time.

Regarding FS, although ISO 4049 recommends dimensions of 25 × 2 × 2 mm for samples used, Muench et al. [16] observed that samples with reduced dimensions can lead to FS values similar to the ones obtained with standardized sample (ISSO 4049), with the advantage of demanding less material and being less time consuming. FS and FM were linearly related to the degree of conversion in composite resins, and with radiant exposure above 4 J/cm^2 the degree of conversion did not vary significantly [22, 23]. It is possible that the degree of conversion of photoactivated group with argon ion laser at 250 mW for 10 seconds is similar to the groups exposed for 20 seconds.

In order to evaluate the quality of polymerization under the microhardness test, the percentage of hardness in relation to the irradiated surface can be considered. According to Watts et al. [24], the level of 80% of the superficial hardness can be considered as a minimum level of polymerization to accept as satisfactory. Based on the results of the present study, all groups were able to promote satisfactory polymerization because a PMH of at least 80% was reached.

Since all groups in this study reached a PMH of at least 80%, with 2 mm of thickness, and that there was no significant difference in the VM of the irradiated surface between the groups, it would be more appropriate to use a source of light that promotes lower SS, as occurred with the group light-cured with argon ion laser at 250 mW for 10 seconds. According to Ferracane [25], a higher degree of hardness in the surface can be translated into better degree of conversion or polymerization of the resin.

Though the degree of conversion and mechanical properties do not increase linearly with the radiant exposure, as it was observed for the values of FS and FM obtained in this study, recent studies have raised a concern that significant increases in SS can still occur at high radiant exposure values [26, 27]. When composite shrinkage is restricted by bonding to cavity walls, polymerization leads to stress development, which may reduce the clinical longevity of a restoration [28]. During the reaction, a composite's viscoelastic behavior changes from viscous (pre-gel phase) to predominantly elastic (post-gel phase), and its capacity to accommodate the reduction in volume through flow decreases accordingly [26].

Several authors have reported the effectiveness of low intensity or pulse-delay methods in the reduction of SS of composite resins [27–30]. In this study, a reduction of approximately 72% in the SS was obtained with the photo activation with argon ion laser at 250 mW for 20 seconds, beyond reducing the activation time and diminishing the working time with the tested resin by 50%, when compared with the control group (halogen light for 20 seconds). Some studies reported a decrease of approximately 28% in SS obtained with pulse-delay methods, using similar test equipment [28, 30, 31]. Witzel et al. [32] and Pfeifer et al. [33] obtained these results with a clinical time of at least 220 seconds, and Lim et al. [30] with 185 seconds. Resin samples photoactivated by different pulse-delay methods showed the degree of conversion and hardness values similar to samples cured by continuous high intensity irradiation [32].

The lowest time of photoactivation with the use of the argon ion laser at 250 mW can be related to the characteristics of their light (monochromatic, coherent and collimated) allowing a large concentration of energy in a small area, with better use and enhanced power of penetration to depths [6].

Associated with these findings, other characteristics of the argon ion laser also promote its use, such as the use as catalyst of agents for cleansing and the increase in the acid resistance of the enamel or of the root surface towards decay lesions [34–36]. After irradiation with argon ion laser, the surface of these substrates is rich in calcium, phosphate, and fluoride [35]. According Powell et al. [37] the demineralization was reduced when human enamel was exposed to argon ion laser. It would be a great progress if during the photoactivation of composite resin an increase in the acid resistance of surrounding substrate was promoted.

5 Conclusions

In conclusion, within the limitations of this in vitro study, the presented results suggest that photoactivation with argon ion laser at 250 mW for 10 seconds would suffice to ensure adequate mechanical properties and minimum SS, reducing the activation time by 50% and diminishing the working time with the tested resin.

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