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Early Surface Microhardness of a Resin Composite Exposed to a Pulse-Delayed Curing Exposure: A Comparison of a Tungsten Halogen and a Plasma Arc Lamp, *In Vitro*

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Abstract - *Surface microhardness numbers of a commercial resin composite have been compared after exposure to a quartz tungsten halogen (QTH) and plasma arc (PAC) lamp respectively, using two exposure protocols. The effect of increased tip-to-composite distance has also been compared. Exposure with the PAC lamp tested is more appropriate to the early finishing of composite restorations after placement and curing than with the QTH. However both lamps were capable of producing comparable surface hardness, with appropriate exposure protocols. The effect of increased tip-to-composite distance was generally not significant between the lamps for continuous exposure.*

KEY WORDS: Resin composite; Surface microhardness; Light-activation; Tungsten halogen; Plasma arc; Exposure times; Tip-to-composite distance

INTRODUCTION

A number of aspects of visible light-activated resin composite tooth restorative materials are of particular importance in the practice of dentistry including:
a) minimum residual stresses on curing
b) appropriate early surface hardness to facilitate finishing
c) achievement of optimum mechanical properties

Stresses introduced through polymerization shrinkage in a setting resin composite material may result in leakage at the tooth-restorative interface[1] or, where the bonding is adequate, deformation of the tooth/restoration combination[2]. Minimization of polymerization stresses is associated with the time interval during which the polymerizing material exhibits flow properties. In this regard, a prolonged gel period is considered beneficial[3]. Modified exposure techniques have recently been described using stepped, pulse-delayed, or ramped protocols[4] in an attempt to extend this flow period. Alternatively, the lamp tip-to-composite distance may be increased.

Development of early surface hardness is considered desirable for the finishing of the restoration[5] (which is generally carried out immediately after placement and curing). In a study of 5 composite formulations[6], finished at various times after curing, it was found that an extended elapsed time was beneficial in determining surface smoothness in a number of materials. The smoothest finishes were considered to occur with the rapid attainment of optimum surface hardness.

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While an increased tip-to-composite distance is primarily of interest in relation to reduced curing stresses, it is reported[7] that light intensity does not decrease with the inverse square of the exposure distance in resin curing.

Data have recently been reported in this laboratory on the microhardness of a resin composite restorative exposed using a pulse-delayed protocol of constant intensity, using a commercially-available tungsten halogen lamp[8] and a plasma arc lamp[9], respectively. It is now proposed to utilise these data sets to compare the efficacy of these lamps with respect to the development of early surface hardness and the effect of tip-to-composite distance exposure variation.

The purpose of this investigation was to compare published data in this laboratory regarding the development of surface hardness in a resin composite cured with a PAC lamp and QTH lamp, respectively, relating to:
1) a pulse-delayed exposure protocol
2) an increased tip-to-composite distance

MATERIAL AND METHODS

The experimental procedure has been reported previously[8,9] the principal details of which were as follows: Groups of a resin composite material (Spectrum TPH, Shade C3, Dentsply DeTrey Gmbh, Konstanz, D-78467, Germany) were exposed in nylon washers (*Figure 1*) using either a quartz-halogen lamp (*XL3000*, 3M, Dental Products Division, 225-1S-09 3M Center, St Paul MN 55144-1000, USA) at full intensity (nominally 630mW/cm^2) or a plasma arc lamp (95E Apollo, Dental & Medical Diagnostic Systems Ltd, Xavier De Cocklaan 68/4, 9831 Deurle, Belgium) at full intensity (nominally 1300mW/cm^2). Groups #1 – #4 (n = 4 x15 specimens) were exposed using a 3-step pulse-delayed protocol with the light-

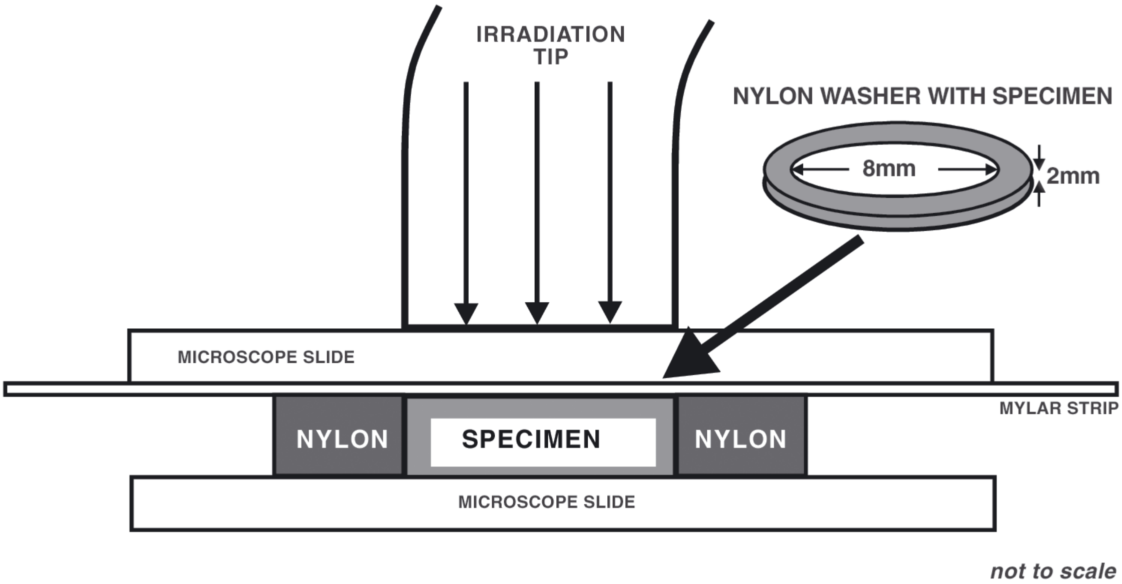

Figure 1. *Sample preparation – disk-shaped specimens are cured in nylon washers sandwiched between microscope slides*

Table 1. *Three-step pulse-delayed curing and mean surface hardness (VHN) for two light curing units*[8,9].

Incremental step	*Control #5*	*#1 Group #1*	*#2 Group #2*	*#3 Group #3*	*Group #4*
	constant intensity (30 secs, QTH; 3x2 secs PAC)	exposure (2 secs)	exposure (2 secs)	exposure (2 secs)	As for Group #3; microhardness measured 1 hour after start of initial exposure
	–	–	dark interval (60 secs)	dark interval (60 secs)	dark interval (60 secs)
			exposure (2 secs)	exposure (2 secs)	exposure (2 secs)
	–	–	–	dark interval (180 secs)	dark interval (180 secs)
	–	–	–	exposure (2 secs)	exposure (2 secs)
Time after start of initial exposure (secs)	3600	2	64	246	3600
Mean (SD) Vickers QTH	49.4(1.9)	18.1(1.7)	23.8(1.5)	29.4(2.5)	38.2(2.0)
Microhardness PAC	49.8(1.6)	29.7(1.5)	34.0(1.2)	36.4(1.2)	42.0(3.7)
Number (VHN) PAC-QTH	0.4	11.6	10.2	7	3.8

Table 2. *Curing distance and mean surface hardness for two light curing units*[8,9]

Distance of light tip from curing surface (mm)		0.9 Control #5	15 Group #6	30 Group #7	45 Group #8
Mean (SD) Vickers Microhardness Number (VHN)	QTH	49.4(1.9)	41.3(2.2)	32.2(2.4)	20.1(1.5)
	PAC	49.8(1.6)	41.5(1.5)	32.7(1.4)	23.6(1.2)

curing tip positioned at 0.9 mm above the specimen surface (which was the thickness of the separating microscope slide). The pulse-delayed technique involved exposure for 2 secs (Step #1, Group #1), followed by a dark interval of 60 secs and subsequent exposure for 2 secs (Step #2, Group #2), followed by a dark interval of 180secs and subsequent exposure for 2 secs (Step #3, Group #3). Upper surface microhardness readings were recorded (15 specimens per group, averaged) for each group and at 1 hour after commencement of initial exposure (Group #4). During the intervals between exposures, the specimens were stored in an opaque box at 20°C. A control group (n = 15 specimens, Group #5) was exposed for each of the curing lamps at maximum intensity (30 secs QTH: sequential 3 x 2sec exposure PAC) and microhardness numbers determined after 60 mins storage, as previously. All microhardness measurements were carried out using a calibrated (manufacturer) Vickers indenter (*MVK-H1,* Mitutoyo, 1-20-1 Sakato, Takatsu-Ku, Kawasaki-Shi 213, Japan) using a load of 300g and a dwell time of 10 secs. SpectrumTPH is a light curing hybrid resin composite restorative material. The inorganic filler is a barium boroaluminosilicate glass (mean particle size < 1μ), together with colloidal silica (particle size = 0.04μ). The resin phase comprises a BisGMA adduct with hexamethylene diisocyanate, an ethoxylated bisphenol-A-dimethacrylate, and triethylene glycol dimethacrylate. Total inorganic filler content is 57% (vol), 77% (mass).

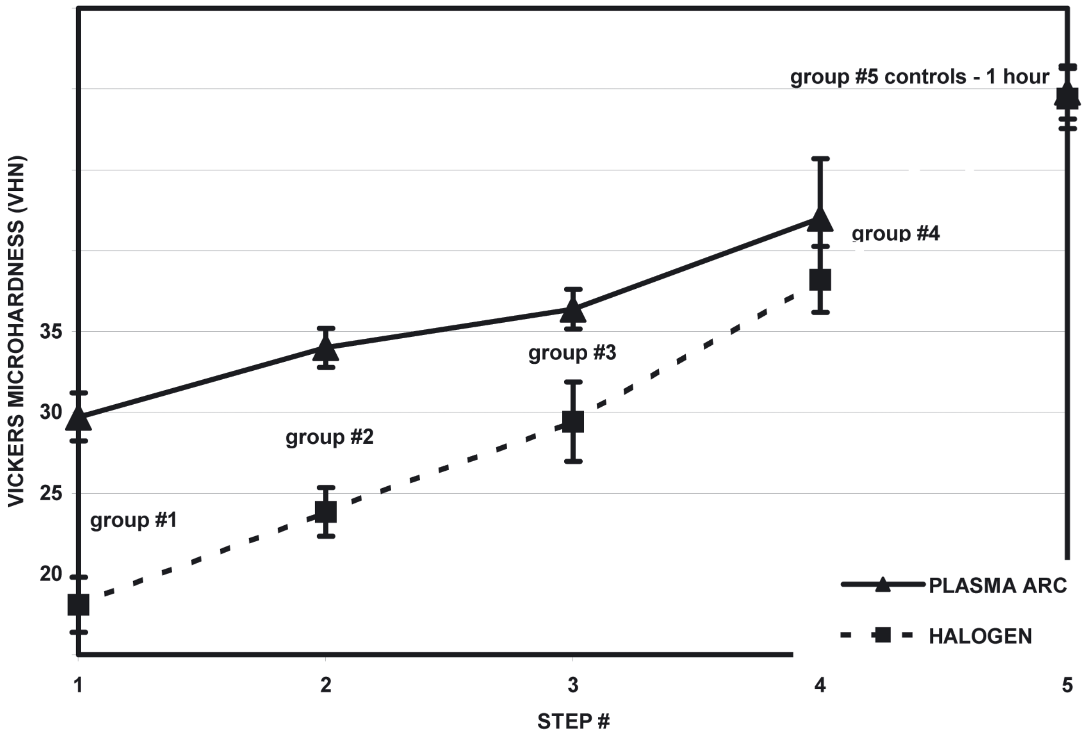

Figure 2. *Mean surface microhardness (VHN) and pulse-delayed protocol for a plasma arc and tungsten halogen curing unit, respectively*

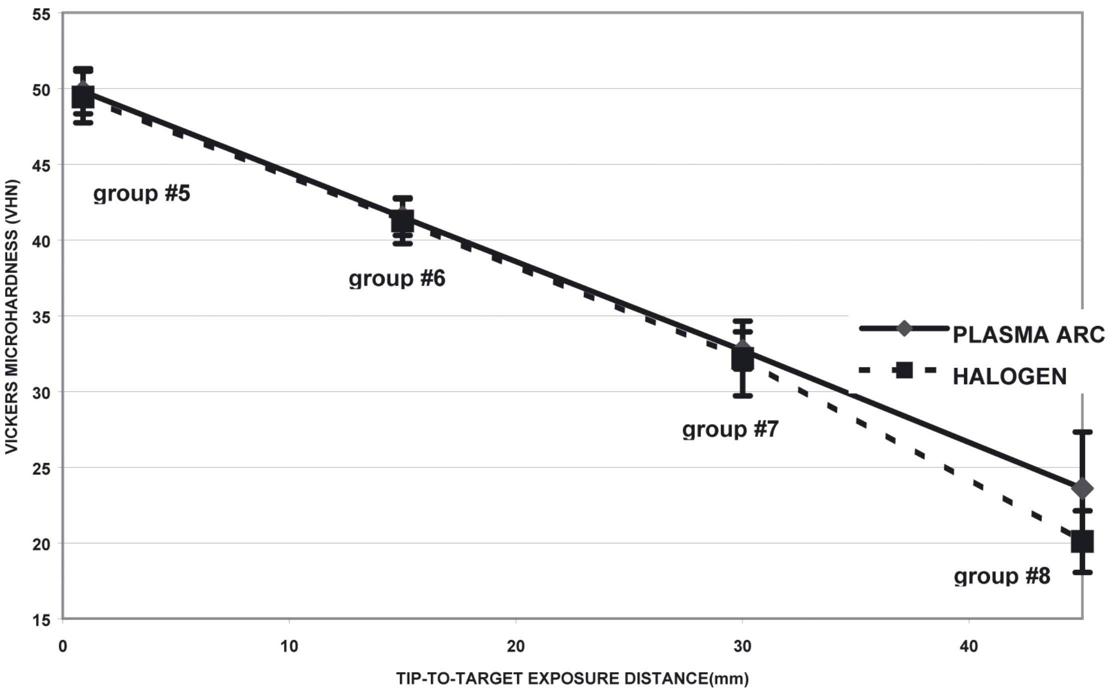

Figure 3. *Mean surface microhardness (VHN) versus exposure distance for a plasma arc and tungsten halogen curing unit, respectively*

The statistical software package SPSS was used for the additional data comparisons and a significance level of 5% was used for all tests.

The data were analysed using a one-way ANOVA with repeated measures:

a) with lamp as a between-subjects factor with respect to exposure
b) with lamp as a between-subjects factor with respect to tip-to-composite distance

The posthoc test used to compare the two lamps was Tukey's test.

RESULTS

The results are presented in *Tables 1* and *2* and in *Figures 2* and *3*.

The differences between the lamps with respect to exposure was statistically significant ($p < 0.01$) for the respective groups #1 to #4 but there was no statistically significant difference for group #5 (control). As previously reported[8,9] the effect of increasing the tip-to-composite exposure distance from the control distance of 0.9 mm yielded a progressive and significant decrease in mean microhardness number with increasing distance for both lamps. The additional analyses indicated that there was no statistically significant difference between the measurements taken at 0.9 mm, 15 mm and 30 mm for the two lamps but there was a statistically significant difference ($p < 0.001$) between the measurements taken at 45mm for the two lamps with the PAC exhibiting the greater hardness.

Regression analysis was used to model the relationships between microhardness and time. The relationship between microhardness and distance was also modelled. Diagnostic checks were carried out to check the validity of the assumptions underlying the linear models. Linear Regression analysis for Groups #1-#4 indicated a positive linear relationship between hardness and the logarithm of time after initial exposure (QTH; VHN = 14.885 + 6.209log(t)[8], PAC: VHN = 27.939 + 3.767log(t), both models giving an R^2 value of 87%. Linear Regression analysis for Groups #5-#8 indicated a negative linear relationship between microhardness number and distance as previously reported[8,9].

DISCUSSION

The polymerization kinetics of visible-light activated dental resins have been discussed in the literature[10]. In general, it is expected that the rate of amine radical formation in the camphoroquinone (CQ)/amine(A) activation system will be proportional to the activation light intensity I_o, at the absorption wavelengths of CQ. In addition, a correlation has been reported[11] between surface microhardness and percentage conversion in resin composite materials, so that hardness numbers may be an indication of differences in mean molecular mass, $\overline{m}_w$. However, it is clear that not all radical reactivity in the setting material was related to chain initiation and/or extension. Thus, any relationship between surface hardness and exposure intensity may not be as clearcut. Nevertheless, it is of interest that exposure to the PAC lamp produced significantly different (greater) mean hardness numbers at each incremental step #1-#3 of the protocol compared with the QTH. In particular, completion of step #3, which corresponds with an elapsed time of 246 secs (4 mins 6 secs) after exposure initiation, was temporally more reasonable in terms of restoration finishing than at 1-hour (group #4, at which time the hardness numbers were still significantly different). Thus, curing with the PAC lamp is more consistent with early surface finishing. However, since no significant difference was observed between the hardness numbers in the control exposure, it is clear that comparable values are obtainable for these lamps given suitable exposure protocols. These latter data are not inconsistent with observations in the literature that QTH lamps are capable of producing ultimate hardness numbers either greater than, or similar to, a PAC exposure[12–14].

For the PAC exposures, the contribution to VHN in the linear regression analysis with respect to elapsed time after initial exposure (t = 1 sec) was 27.9, while the corresponding value for the QTH lamp was 14.9. These values may be a reflection of the respective exposure intensities and initial chain development – the greater intensity of the PAC lamp initially nucleating more contributions to initial rapid chain extension and increased $\overline{m}_w$. However, it is of interest that the slope relating to time (log(t)) was greater for the QTH lamp than for the PAC, suggesting that the rate of hardness development was greater for the former within the experimental protocol. Thus, it would appear that the pulsing technique was more effective with the QTH lamp at increasing the rate of such events, resulting in the ultimate coalition of the hardness numbers in the control groups.

Notwithstanding the greater nominal exposure intensity of the PAC lamp, it is of interest that no significant difference was observed between the lamps with respect to increased tip-to-composite distance using the continuous exposure protocol except for distance = 45mm.

A degree of caution should, however, be used in relating surface VHN data to percentage conversion (degree of cure) in these systems, in the absence of more specific investigations, such as near-infrared Raman spectroscopy[15].

CONCLUSIONS

1) Exposure using the PAC lamp may be more appropriate to the early finishing of composite restorations than the QTH lamp due to the significantly greater mean surface hardness at each stage of the protocol used.
2) Both the PAC and QTH lamps are capable of producing similar surface hardness with an appropriate exposure protocol.
3) The effect of increasing the tip-to-composite distance generally produced similar surface hardness for both the PAC and QTH lamps for the continuous exposure used.

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REFERENCES

1. Krishnan, V.K., Bindhu, D.B. and Manjusha, K. Studies on microleakage associated with visible light cured dental composites. *J. Biomater. Appl.,* 1996; **10:**348–359.
2. Suliman, A.A., Boyer, D.B. and Lakes, R.S. Interferometric measurements of cusp deformation of teeth restored with composites. *J. Dent. Res.,* 1993; **72:**1532–1536.
3. Rueggeberg, F.A. and Craig, R.G. Correlation of parameters used to estimate monomer conversion in a light-cured composite. *J. Dent. Res.,* 1988; **67:**932–937.
4. DeWald, J.P. and Ferracane, J.L. A comparison of four modes of evaluating depth of cure of light activated composites *J.*

Dent. Res., 1987; **66:**727–730.

5. Yap, A.U., Sau, C.W. and Lye, K.W. Effects of finishing/polishing time on surface characteristics of tooth-coloured restoratives. *J. Oral Rehabil.,* 1998; **25:**456–461.
6. Heath, J.R., Jordan, J.H. and Watts, D.C. The effect of time of trimming on the surface finish of anterior composite resins. *J. Oral Rehabil.,* 1993; **20:**45–52.
7. Rueggenberg, F.A. and Jordan, D.M. Effect of light-tip distance on polymerization of resin composite. *Int. J. Prosthodont.,* 1993; **6:**364–370.
8. Lynch, C., Ray, N., Hagan, L. and Burke, F. The effect of different exposure routines on the surface microhardness of a light-activated resin composite. *Quint. Int.,* 2003; **34:**468–472.
9. Hickey, A., Lynch, C.D., Ray, N.J., Burke, F.M. and Hannigan, A. Surface microhardness of a resin composite exposed to pulse-delayed plasma arc lamp irradiation, *in vitro. Eur. J. Prosthodont. Rest. Dent.,* 2002, **10:**107–111.
10. Cook, W.D. Photopolymerization kinetics of dimethacrylates using the camphoroquinone/amine initiator system. *Polymer,* 1992; **33:**600–609.
11. Rueggeberg, F.A. and Craig, R.G. Correlation of parameters used to estimate monomer conversion in a light-cured composite. *J. Dent. Res.,* 1988; **67:**932–937.
12. Price, R.B., Ehrnford, L., Andreou, P. and Felix, C.A. Comparison of quartz-tungsten-halogen, light-emitting diode, and plasma arc curing lights. *J. Adhes. Dent.,* 2003; **5:**193–207.
13. Price, R.B., Derand, T., Lonewy, R.W. and Andreou, P. Effect of light source and specimen thickness on the surface hardness of resin composite. *Am. J. Dent.,* 2002; **15:**47–53.
14. Park, S.H., Krejci, I. and Lutz, F. Microhardness of resin composites polymerised by plasma arc or conventional visible light curing. *Oper. Dent.,* 2002; **27:**30–37.
15. Soares, L.E.S., Martin, A.A., Pinheiro, A.L.B. and Pancheco, M.T.T. Vicker's hardness and Raman spectroscopy evaluation of a dental composite cured by an argon laser and a halogen lamp. *J. Biomedical Optics,* 2004; **9:**601–608.