Eur. J. Prosthodont. Rest. Dent., Vol.14, No. 1, pp 7-12
Printed in Great Britain

Surface Microhardness of a Resin Composite: a Comparison of a Tungsten Halogen and a LED Light Curing Unit, *in vitro*

Noel J. Ray*, Christopher D. Lynch†, Francis M. Burke‡ and Ailish Hannigan§

Abstract - *A comparison has been made between published surface microhardness numbers (VHN) of a commercial resin composite for different exposure times to a quartz tungsten halogen (QTH) and light-emitting diode (LED) light-curing unit (LCU). Both LCUs produced comparable hardness at both top and bottom surfaces, respectively, and similar bottom/top hardness ratios, for a specimen thickness of 1.5 mm, given sufficient exposure time (40 s) and an elapsed time of 24 h before measurement (for hardness numbers). However, some data are significantly different. There is no advantage in either LCU regarding optimal hardness and hardness ratios given an appropriate protocol. Immediate finishing (1 h) was more appropriate to the use of the LED LCU (with adequate exposure time). The effect of elapsed time after exposure on microhardness was more pronounced with the QTH LCU.*

KEY WORDS: Resin Composite; Surface Microhardness; Light-Activation; Tungsten Halogen; Light Emitting Diode; Exposure Times

INTRODUCTION

A number of aspects of visible light-activated resin composite tooth restorative materials are of particular importance in dentistry including the attainment of optimal mechanical properties on curing and early surface hardness to facilitate finishing. Optimised mechanical properties are achieved through adequate polymerisation[1] to ensure that the residual monomer content in the restoration is minimized. In light-activated restorative materials the degree of monomer conversion is governed, amongst other things, by the accessibility of light of adequate intensity and of suitable spectral distribution[2].

While a convenient and traditional curing source has been the quartz halogen (QTH) light curing unit (LCU), interest has recently been expressed in devices based on the solid-state light-emitting diode (LED)[3,4] as an alternative, and several units are commercially available. While the irradiance of such units is generally less than the QTH systems[4], the LED LCUs are more wavelength specific to the polymerisation of camphoroquinone, so that a simplistic comparison of curing efficiency, based on exposure intensity, may not be appropriate. A correlation has been reported between surface microhardness of resin composite and degree of conversion[5,6] and such determinations may be indicative of differences between different exposure protocols, eg for different LCUs.

Attenuation of light intensity with distance from the curing-tip into the polymerising material results in reduced polymerisation[7] and a limiting depth-of-cure[8] which is related to light scattering within the material[9]. The use of disks specimens for *in vitro* microhardness testing, where the activating light tip is in contact with the top surface only, allows the determination of bottom/top surface microhardness ratios, which are indicative of this depth-of-cure. The use of microhardness data suggests[10] that this ratio should be in excess of 0.8 for adequate polymerisation.

Data have recently been reported from this laboratory on the microhardness of a resin composite restorative material exposed using a commercially-available QTH LCU[11] and a LED LCU[12], respectively. It is now proposed to utilise these data sets to compare the efficacy of these LCUs with respect to the development of surface hardness and bottom/top hardness ratios.

The purpose of this investigation was to compare published data from this laboratory regarding the development of surface hardness in a resin composite cured with a QTH LCU and LED LCU, respectively, relating to:

1) The development of surface hardness with regard to exposure time and elapsed time after exposure

2) The variation of bottom/top hardness ratios with the above.

MATERIAL AND METHODS

The experimental procedure has been reported previously[11,12] the principal details of which were as follows: Groups of a resin composite material (Spectrum TPH, Shade C3, Dentsply DeTrey Gmbh, D-78467, Germany) were exposed in nylon washers using either a QTH LCU (*XL3000*, 3M, Dental Products Division, 225-1S-09 3M Center, St Paul MN 55144-1000, USA) at full intensity (nominally 630 mW/cm^2) or a LED LCU (Freelight, 3M/Espe, St Paul, MN, USA) at full intensity (nominally 310 mW/cm^2). A specimen depth of 1.5 mm was considered to be con-

*MSc, PhD
†BDS, MFDRCSI
‡BDENTSc, MSc, PhD, FDSRCSEd, FFDRCSI
§BSc PhD

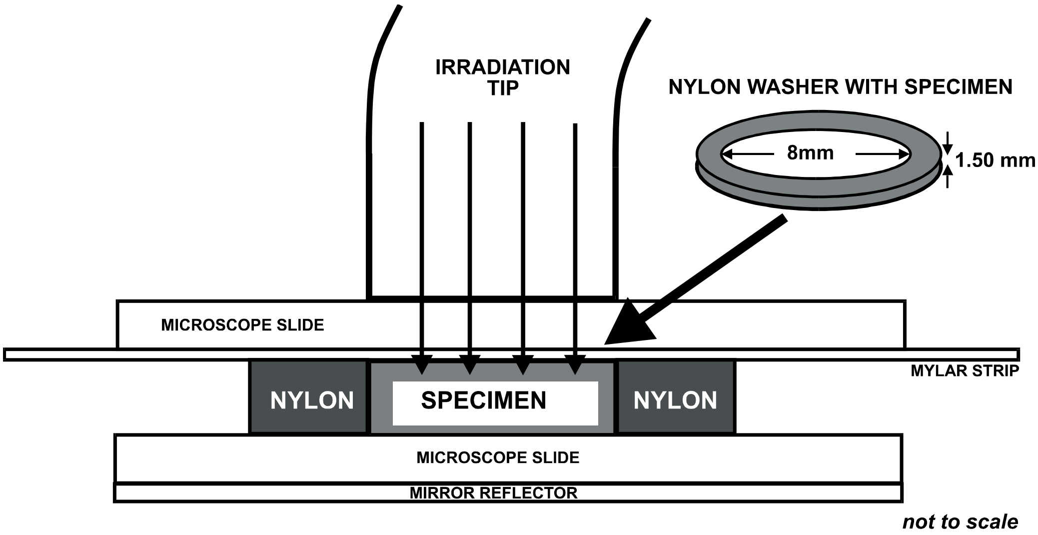

Figure 1. *Specimen preparation – a nylon washer of depth 1.5 mm is filled with resin composite and sandwiched between glass microscope slides before exposure*

Table 1. *Exposure protocols and surface microhardness numbers*

n = 15 *Exposure (secs)*	*Group #1* *5*	*Group #2* *10*	*Group #3* *15*	*Group #4* *20*	*Group #5* *30*	*Group #6* *40*
			Mean(SD) Vickers Microhardness Number (VHN) 1- hour (top surface)			
QTH	*43.4(3.8)*	*49.4(1.2)*	*50.5(1.2)*	*52.6(1.3)*	*53.7(1.3)*	*53.6(1.2)*
LED	*35.7 (4.7)*	*46.0(5.9)*	*51.0(3.0)*	*53.4(4.9)*	*57.6(3.9)*	*59.2(4.1)*
QTH - LED	*7.7*	*3.4*	*-0.5*	*-0.8*	*-3.9*	*-5.6*
			Mean(SD) Vickers Microhardness Number (VHN) 1- hour (bottom surface)			
QTH	*30.5(3.1)*	*43.1(1.6)*	*45.5(1.2)*	*48.6(1.2)*	*51.1(1.4)*	*50.8(2.5)*
LED	*13.4(1.8)*	*35.6(5.7)*	*38.5(4.3)*	*41.3(6.6)*	*50.7(2.8)*	*53.6(2.0)*
QTH - LED	*17.1*	*7.5*	*7*	*7.3*	*0.4*	*-2.8*
Hardness ratio QTH (bottom/top)	*0.70[0.68 0.73]**	*0.87[0.85 0.90]*	*0.90[0.89 0.92]*	*0.92[0.90 0.94]*	*0.95[0.94 0.97]*	*0.95[0.91 0.98]*
Hardness ratio LED (bottom/top)	*0.37[0.34 0.41]**	*0.77[0.72 0.83]*	*0.75[0.71 0.80]*	*0.77[0.71 0.82]*	*0.88[0.86 0.90]*	*0.91[0.87 0.94]*
			Mean(SD) Vickers Microhardness Number (VHN) 24- hour (top surface)			
QTH	*52.0(3.7)*	*59.2(1.7)*	*59.8(1.6)*	*60.2(1.3)*	*61.3(1.1)*	*61.0(1.3)*
LED	*39.7(6.2)*	*50.6(4.6)*	*53.2(2.7)*	*57.7(5.9)*	*61.0(3.7)*	*62.7(3.5)*
QTH - LED	*12.3*	*8.6*	*6.6*	*2.5*	*0.3*	*-1.7*
			Mean(SD) Vickers Microhardness Number (VHN) 24- hour (bottom surface)			
QTH	*35.1(3.8)*	*51.2(1.7)*	*53.7(1.2)*	*55.5(1.9)*	*58.9(1.4)*	*58.6(1.2)*
LED	*14.0(3.6)*	*37.9(6.9)*	*41.0(3.1)*	*45.8(8.0)*	*54.2(3.1)*	*57.8(2.4)*
QTH - LED	*21.1*	*13.3*	*12.7*	*9.7*	*4.7*	*0.8*
Hardness ratio QTH (bottom/top)	*0.67[0.65 0.70]**	*0.87[0.85 0.88]*	*0.90[0.88 0.91]*	*0.92[0.90 0.94]*	*0.96[0.95 0.97]*	*0.96[0.95 0.97]*
Hardness ratio LED (bottom/top)	*0.35[0.30 0.40]**	*0.75[0.68 0.80]*	*0.77[0.73 0.82]*	*0.79[0.73 0.84]*	*0.89[0.87 0.91]*	*0.92[0.89 0.96]*

** 95% confidence intervals*

sistent with the maximum allowable for the incremental curing of resin composites[13,14] (*Figure 1*). Groups #1 – #6 (15 specimens per group) were exposed according to the protocols indicated (*Table 1*) with the light-curing tip positioned at 0.9 mm above the specimen surface (which was the thickness of the separating microscope slide). A reflective background was used under each specimen. Top and bottom surface Vickers microhardness (VHN) numbers were recorded at 1 hour and 24 hours after the start of irradiation. During the intervals, the specimens were stored dry in an opaque box at 20°C. Group #6 acted as a control to yield optimised microhardness numbers (within the exposure parameters) – a 40 s exposure was considered to be the maximum continuous exposure time likely to be acceptable in the clinical environment. All microhardness measurements were performed using a calibrated 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 s.

Statistical analysis was carried out using SPSS for Windows (Version 11). A significance level of 5% was used for all statistical tests. A repeated measures analysis of variance was used to investigate the significance of the within-specimen factors – elapsed time (1 hour and 24 hours) and position (top, bottom). The between-specimen factor was the experimental group (groups #1 to #6) and LCU (QTH and LED). Post-hoc tests were used to test for differences between the levels of a factor. A multiple regression model, using all factors as predictors of surface microhardness numbers, was fitted to the data. 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

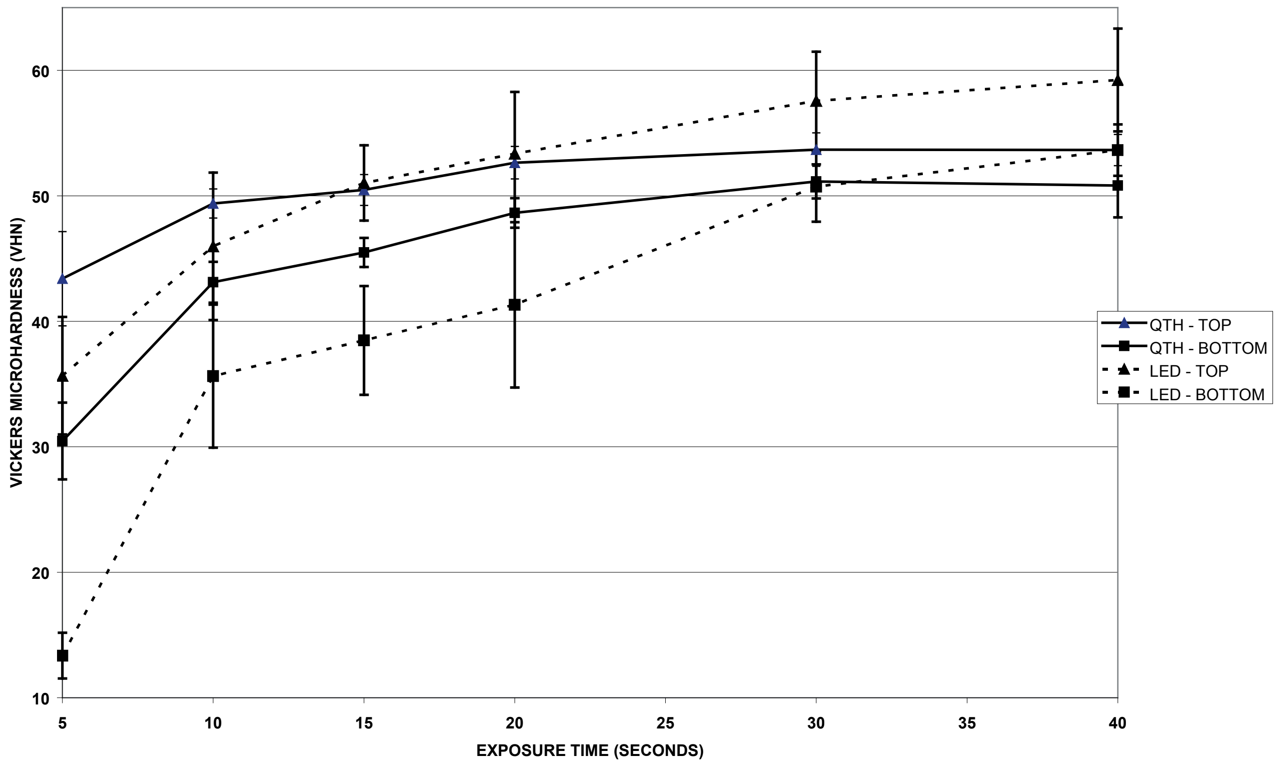

Figure 2. *Mean surface microhardness (VHN) for a resin composite exposed to a quartz halogen and a LED light curing unit (1-hour)*

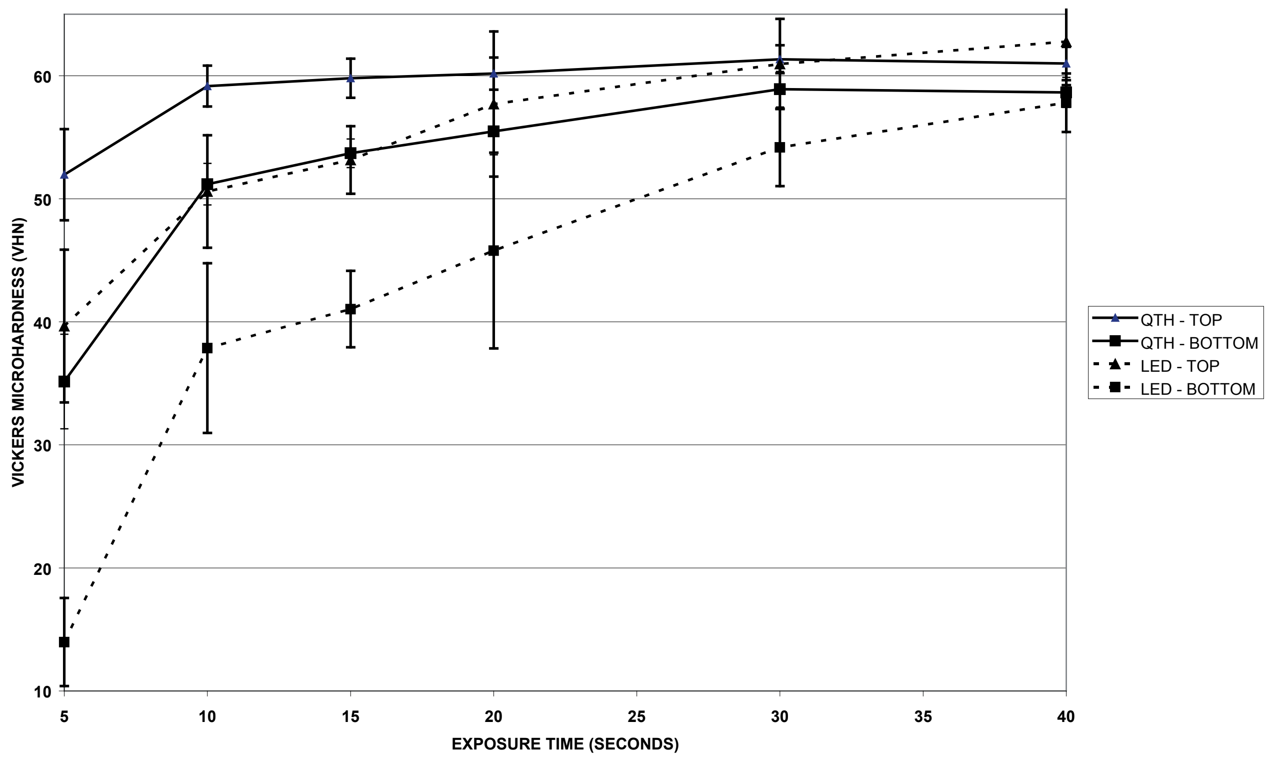

Figure 3. *Mean surface microhardness (VHN) for a resin composite exposed to a quartz halogen and a LED light curing unit (24-hour)*

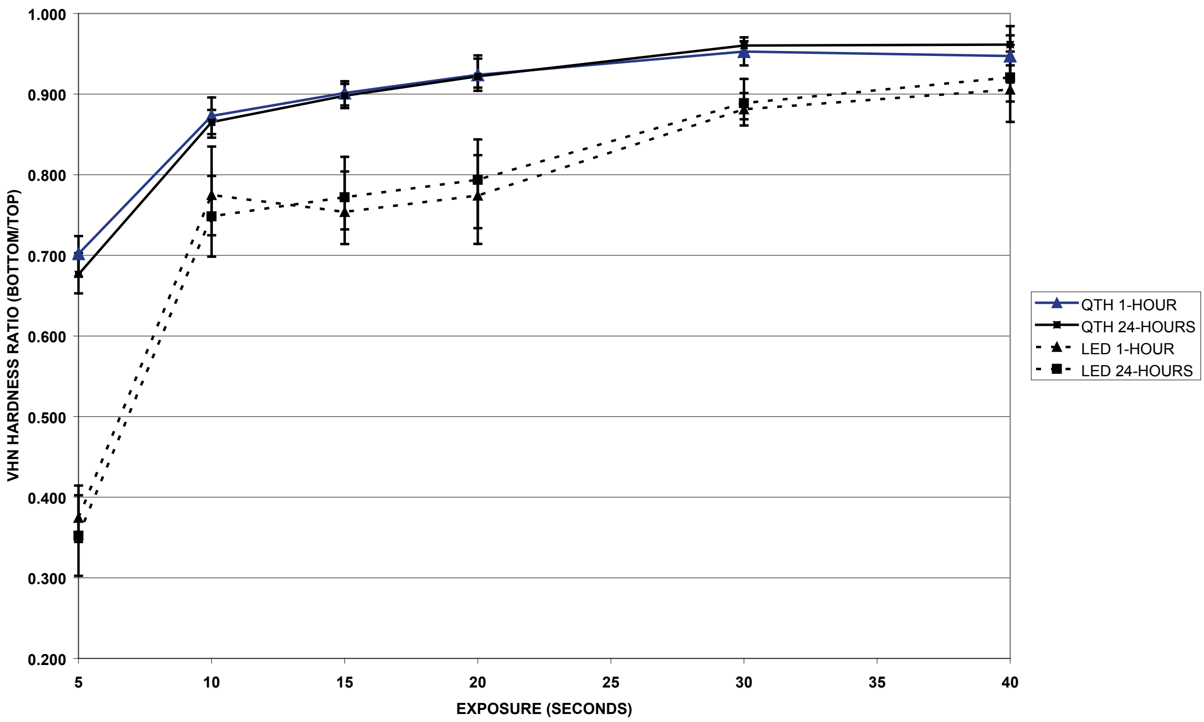

Figure 4. *Microhardness ratios (VHN bottom/top) for a resin composite exposed to a quartz halogen and a LED light curing unit*

silica (particle size = 0.04µ). The resin phase comprises a BisGMA adduct with hexamethy-lene diisocyanate, an ethoxylated bisphenol-A-dimethacry-late, and triethylene glycol dimethacrylate. Total inorganic filler content is 57% (vol), 77% (mass).

RESULTS

The results are presented in *Table 1* and in *Figures 2–6*. Overall, surface location (top, bottom), elapsed time after activation (1 h and 24 h), exposure time (group) and LCU (QTH, LED) were all statistically significant factors in determining hardness numbers ($p < 0.001$). The difference between measurements taken for each LCU at the top at 1h is statistically significant for groups #1, #2, #5 and #6 ($p < 0.05$). The difference between measurements taken for each LCU at the top at 24 h is statistically significant for groups #1, #2 and #3 ($p < 0.001$). The difference between measurements taken for each LCU at the bottom at 1 h is statistically significant for groups #1, #2, #3, #4 and #6 ($p < 0.01$). The difference between measurements taken for each LCU at the bottom at 24 h is statistically significant for groups #1, #2, #3, #4 and #5 ($p < 0.001$). Overall, there is a statistically significant difference ($p < 0.001$) between the measurements taken at the top at 1 h and 24 h for the QTH LCU and the LED LCU. The same is true for measurements taken at the bottom at 1 h and 24 h. For the LED LCU these differences are statistically significant for the top (1 h vs 24 h) for all groups ($p < 0.05$). These differences are statistically significant for the bottom (1 h vs 24 h) for groups #4, #5 and #6 ($p < 0.01$). For the QTH LCU, these differences are statistically significant for the top (1 h vs 24 h) for all groups ($p < 0.001$). These differences are statistically significant for the bottom (1 h vs 24 h) for all groups ($p < 0.001$).

There is a significant difference between the bottom/top ratio for each LCU for groups #1, #2, #3, #4 and #5 at both 1 hr and 24 hrs ($p < 0.05$). The ratios for group #6 for each LCU are not significantly different from one another at either 1 h or 24 h.

DISCUSSION

The results regarding to each LCU separately have already been discussed in the literature[11,12]. On comparison of the data, it is clear that both LCUs will produce similar hardness numbers at both top and bottom surfaces, respectively, and similar hardness ratios, given sufficient exposure time and elapsed time before measurement (in the case of hardness numbers, only). In the present case, these data are 40 s exposure and an elapsed time of 24 h before measurement (*Figures 3* and *4*). Thus, both LCUs are equally efficient at curing the composite material, both at the surface and with increasing distance from the curing tip (depth-of-cure), given appropriate parameters.

These observations are consistent with a comparison of depth of cure and surface microhardness between three commercial LED LCUs and two QTH LCUs which concluded that the former were suitable for the routine curing of resin composite[3]. However, it is of interest to consider these data as they relate to the internal exposure controls for each LCU, as reported[11,12]. For micro-hardness measurements at 1 h for the QTH LCU[11], a minimum of 30 s exposure was adequate (within the range of intervals investigated) to optimise both top and bottom values with respect to the controls at the same time. However, the microhardness ratio (bottom/top) had optimised at 20 s exposure. For measurements at 24 h, a minimum of 15 s exposure was satisfactory (within the range of intervals in-

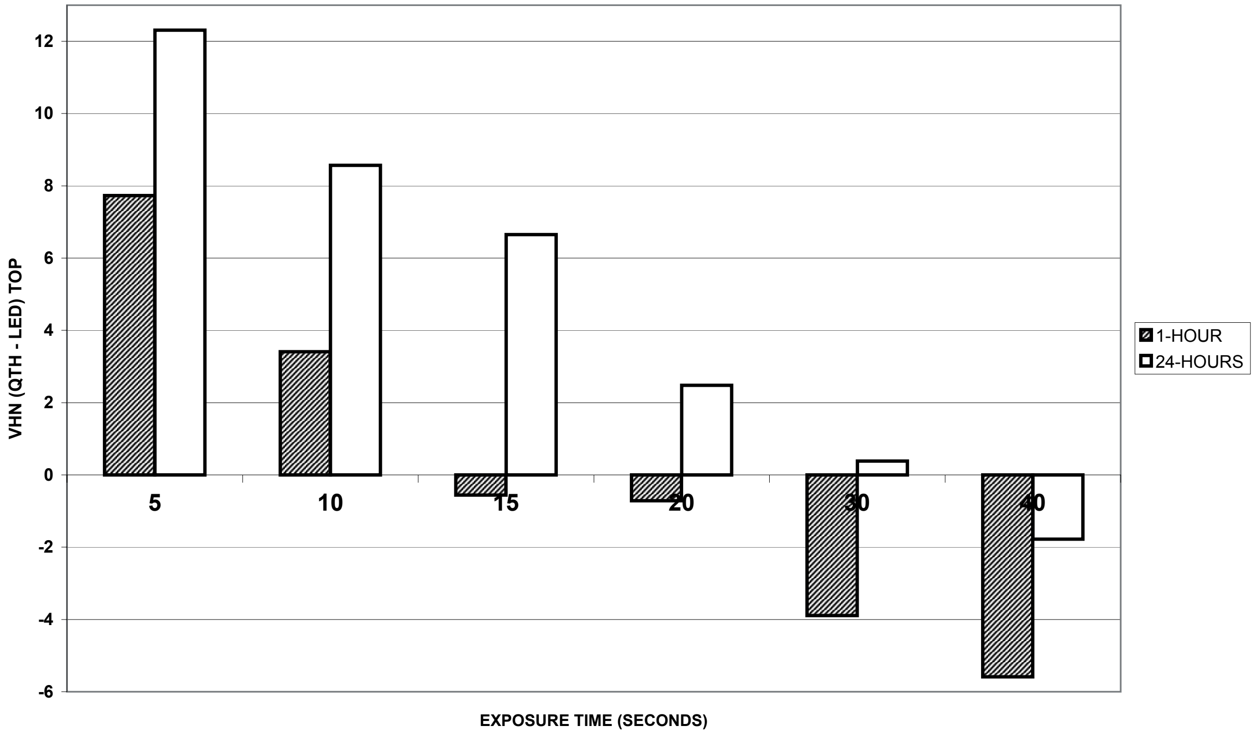

Figure 5. *Mean difference in microhardness for a resin composite exposed to two light-curing units (QTH – LED), top surface*

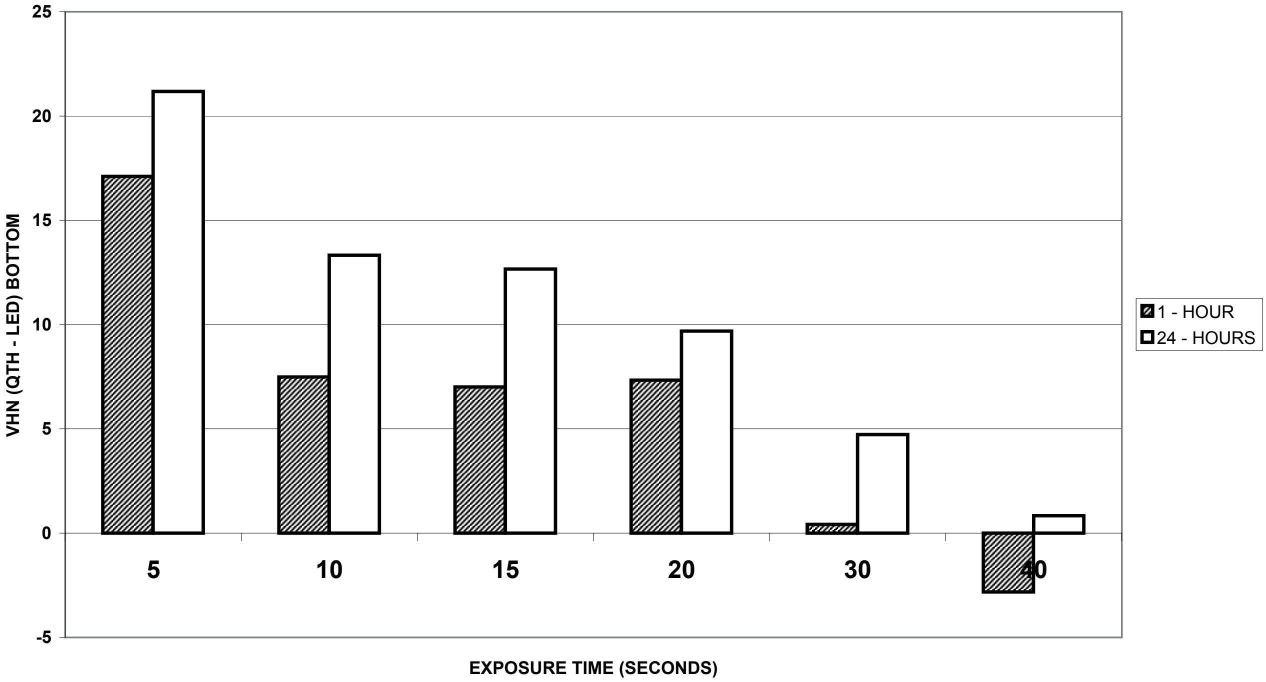

Figure 6. *Mean difference in microhardness for a resin composite exposed to two light-curing units (QTH – LED), bottom surface*

vestigated) to optimise the top surface data and a minimum of 20 s for the bottom surfaces with respect to the controls at the same time. The microhardness ratio (bottom/top) had again optimised at 20 s exposure.

For microhardness measurements at both 1 h and 24 h for the LED LCU[12], a minimum of 30 s exposure was adequate (within the range of intervals investigated) to optimise the top surface values with respect to the internal controls at the same time. The corresponding data for the bottom surfaces was 40 s exposure. The microhardness ratio (bottom/top) had optimised at 30 s exposure for both 1 hour and 24 hour measurements. Clearly, the 40 s exposure is the more appropriate to maximise these parameters with respect to the controls.

Thus, it is clear that adequate exposure is more critical to the LED LCU in achieving optimal hardness, and that elapsed time after exposure is more critical to the QTH LCU, such that both produce comparable results. These effects are best seen in the differences between the VHN numbers (*Figures 5* and *6)* and almost certainly relate to an interplay between exposure intensity and wavelength specificity to the activation of camphoroquinone. However, maturation (elapsed time of 24 h) removes any significant differences between the LCUs in the case of 40 s exposure. Maturation is more effective with the QTH LCU in that all groups feature significantly increased hardness between 1 h and 24 h post exposure. In the case of hardness ratios, maturation has no effect for either LCU and the sole determinant, within the experimental protocol, is the exposure time.

Development of early surface hardness is considered desirable to the finishing of the restoration[15] which is generally carried out immediately after placement and curing. In a study of 5 composite formulations[16], finished at various times after curing, it was found that the smoothest finishes were considered to relate to the rapid attainment of optimum surface hardness. Thus, immediate finishing (1 h) is more appropriate with the LED LCU, with adequate exposure time.

CONCLUSIONS

1. Both LCUs produced a similar hardness at both top and bottom surfaces, respectively, and a similar hardness ratio, given sufficient exposure time and elapsed time before measurement.
2. Immediate finishing (1 h) was more appropriate with the LED LCU, with adequate exposure time.
3. The effect of elapsed time after exposure on microhardness was more pronounced with the QTH LCU.

ACKNOWLEDGEMENTS

DENTSPLY DeTrey GmbH for the donation of SpectrumTPH

ADDRESS FOR CORRESPONDENCE

Noel J. Ray, Department of Restorative Dentistry, National University of Ireland, Cork, Ireland. E-mail: n.ray@ucc.ie

REFERENCES

1. Ferracane, J.L. and Condon, J.R. Postcure heat-treatments for composites: properties and fractography. *Dent. Mater.*, 1992;**8**:290–295.
2. Stansbury, J.W. Curing dental resins and composites by photo polymerization. *J. Esthet. Dent.*, 2000; **12**:300–308.
3. Tsai, P.C.L., Meyers, I.A. and Walsh, L.J. Depth of cure and surface microhardness of composite resin cured with blue LED curing lights. *Dent. Mater.*, 2004; **20**:364–369.
4. Uhl, A., Sigush, B.W. and Jandt, K.D. Second generation LEDs for the polymerisation of oral biomaterials. *Dent. Mater.*, 2004; **20**:80–87.
5. 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.
6. 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.
7. Caldas, D.B., de Almeida, J.B., Correr-Sobrinho, L., Sinhoreti, M.A. and Consani, S. Influence of curing tip distance on resin composite knoop hardness number, using three different light curing units. *Oper. Dent.*, 2003; **28**:315–320.
8. Fan, P.L., Schumacher, R.M., Azzolin, K., Geary, R. and Eichmiller, F.C. Curing-light intensity and depth of cure of resin-based composites tested according to international standards. *J. Am. Dent. Assoc.*, 2002; **133**:429–434.
9. Taira, M., Okazaki, M. and Takahashi, J. Studies on optical properties of two commercial visible-light-cured composite resins by diffuse reflectance measurements. J. Oral Rehabil.,1999; 26:329–337.
10. Pilo, R. and Cardash, H.S. Post-irradiation polymerization of different anterior and posterior visible-light–activated resin composites. *Dent. Mater.*, 1992; **8**:299–304.
11. Coffey, O., Ray, N.J., Lynch, C.D., Burke, F.M. and Hannigan, A. Surface microhardness of a resin composite exposed to a quartz halogen lamp, in vitro. *Quint. Int.*, 2004; **35**:795–800.
12. Keogh, P., Ray, N.J., Lynch, C.D., Burke, F.M. and Hannigan, A. Surface Microhardness of a Resin Composite Exposed to a “First-Generation” LED Curing Lamp, in Vitro. *Eur. J. Prosthodont. Rest. Dent.*, 2004; **12**:177–180.
13. Ruggenberg, F.A., Caughman, W.F. and Curtis, J.W. Jr. Effect of light intensity and exposure duration on cure of resin composite. *Oper. Dent.*, 1994; **19**:26–32.
14. Maris, J.T., Dannheimer, M.F., Germishuys, P.J. and Borman, J.W. Depth of cure of light-cured composite resin with lightcuring units of different intensity. *J. Dent. Assoc*. S. Afr., 1997; **52**:403-407.
15. 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.
16. 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.