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Light-emitting diode (LED) polymerisation of dental composites: flexural properties and polymerisation potential

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Abstract

The clinical performance of light polymerised dental composites is greatly influenced by the quality of the light-curing unit (LCU) used. Commonly used halogen LCUs have some specific drawbacks such as decreasing of the light output with time. This may result in low degree of monomer conversion of the composites with negative clinical implications. Previous studies have shown that blue-light-emitting diode (LED) LCUs have the potential to polymerise dental composites without having the drawbacks of halogen LCUs. Despite the relatively low irradiance of current LED LCUs, their efficiency is close to that of conventional halogen LCUs with more than twice the irradiance. This phenomenon has not been explained fully yet. Hence, more tests of the LED LCU's effectiveness and of the mechanical properties of oral biomaterials processed with LED LCUs need to be carried out. This study investigates the flexural properties of three different composites with three different shades, which were polymerised with either a commercial halogen LCU or an LED LCU, respectively. In most cases no significant differences in flexural strength and modulus between composites polymerised with a halogen LCU or an LED LCU, respectively, were found. A simple model for the curing effectiveness based on the convolution absorption spectrum of the camphorquinone photoinitiator present in composites and the emission spectra of the LCUs is presented. © 2000 Elsevier Science Ltd. All rights reserved.

Keywords: LED; Light-curing unit (LCU); Composites; Polymerisation; Spectrum; Flexural strength; Modulus

1. Introduction

Halogen technology light-curing units (LCU) used to polymerise dental materials have several drawbacks. For example, halogen bulbs have a limited effective lifetime of approximately 100 h [1]. The LCU's bulb, reflector, and filter can degrade over time due to the high operating temperatures and the large quantity of heat, which is produced during the operating cycles. This results in a reduction of the LCU's curing effectiveness over time [2]. The clinical implication is that with an ageing LCU, light-activated dental materials will be less well polymerised with poorer physical properties and an increased risk of premature failure of restorations, assuming no compensation for decreased LCU irradiance.

It has been shown that many halogen LCUs used by dental clinicians do not reach the minimum power output specified by the manufacturers [2–4]. This can be caused by a lack of maintenance, such as the absence of a critical check of the LCU's irradiance, or the fact that the filter and/or the halogen bulb are not replaced from time to time. The measured irradiance of LCUs also depends on the radiometers used and it appears that there is little consistency of irradiance measured with radiometers used in dental practice [4,5].

To overcome the problems inherent to halogen LCUs, solid-state light-emitting diode (LED) technology has been proposed for curing light-activated dental materials [6]. Rather than a hot filament as used in halogen bulbs, LEDs use junctions of doped semiconductors (p–n junctions) for the generation of light [7,8]. Under proper forward biased conditions, electrons and holes recombine at the LED's p–n junction leading, in the case of gallium nitride LEDs, to the emission of blue light. The spectral output of gallium nitride blue LEDs falls

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conveniently within the absorption spectrum of the camphorquinone photoinitiator (400–500 nm) present in light-activated dental materials, so no filters are required in LED LCUs. Furthermore, LEDs have an expected lifetime of several thousand hours without significant degradation of light emission over time [9].

Several studies have addressed the application of blue LED technology to cure dental materials [10–13]. Although these tests show the potential of LED LCUs, it is important to compare further the LED LCU's performance with that of commercial halogen LCUs with a typical irradiance as specified by the manufacturer. These specifications normally exceed the lower effective limit of irradiance for halogen LCUs of 300 mW cm^{-2} [14,15] by two to three times. Depth of cure and compressive strength measurements are clearly relevant to clinical aspects of composite curing. Additional information about the mechanical properties of composites polymerised with an LED LCU are, however, required to judge the LED LCU's clinical potential in more depth.

The aim of this study was to investigate the flexural properties of three different composites in three different shades, which were polymerised with either an LED LCU or a halogen LCU. Secondly, we aimed towards a simple model, which would help to explain the LED LCU's curing effectiveness compared to halogen LCUs.

2. Materials and methods

A Spectrum LCU model 201 R (Dentsply DeTrey GmbH, Konstanz, Germany) based on halogen technology and a custom-made LED LCU [13] were used in our study. The Spectrum LCU has a light guide tip diameter of 9 mm and uses a fan-cooled halogen bulb with a power consumption of 49 W. The irradiance of the halogen LCU is 755 mW cm^{-2} [13].

The LED LCU used in this study contains 27 blue LEDs (Nichia Chemical Industries Ltd., Anan, Japan). The light from this LCU is concentrated using a polymer optical taper with a 15 mm diameter input face and a 6 mm output face (light guide tip) directed towards the composite samples. This construction provides approximately a six-fold increase in output power density. The irradiance of the LED LCU is 350 mW cm^{-2} [13].

The dental composites used in this study were Spectrum TPH, shades A2 and A4 (Dentsply DeTrey GmbH, Konstanz, Germany), Z100, shades A2 and A4 (3M Dental Products, St. Paul, MN, USA) and Solitaire, shades A2, A3 (Heraeus Kulzer, Wehrheim, Germany). At the time of the tests there was no Solitaire shade A4 commercially available. Table 1 summarises the materials and the light exposure times used in this study.

The flexural properties of the composites were tested according to ISO 4049 [16]. In brief, the composites were filled into a stainless-steel mould of $25 \text{ mm} \times 2 \text{ mm} \times$

Table 1

Materials used in this study and light exposure times at each section of the specimen. The exposure times used in this study are recommended by the composite manufacturers

Material/shade	Type	Light exposure time per section (s)
Spectrum TPH/A2	Hybrid composite	20
Spectrum TPH/A4	Hybrid composite	40
Z100/A2	Hybrid composite	40
Z100/A4	Hybrid composite	40
Solitaire/A2	Polyglas	40
Solitaire/A3	Polyglas	40

2 mm in size, positioned on a glass slide. Subsequently, a second glass slide was positioned on top of the mould. The exit window of the LCU was then placed against the glass plate at the centre of the specimen and irradiation was applied according to the manufacturers recommended times as shown in Table 1. The exit window was moved to the section next to the centre overlapping the previous section. The light exposure proceeded in this way until the entire length of the specimen had been irradiated. This procedure was applied to both sides of the specimens. Due to the different diameters of the light guides of both LCUs, seven exposure steps per specimen side were required for the LED LCU whereas five steps were needed for the halogen LCU for polymerisation. The specimens were stored for 24 h in water at 310 K before the mechanical tests. Ten specimens for each shade were prepared and tested. The tests were performed with a Lloyd 2000 R mechanical tester in a water bath at 310 K with a crosshead speed of 0.75 mm/min to the point of specimen fracture. The maximum loads applied to the specimens were recorded.

The flexural strength σ in MPa was calculated from the following equation:

$$\sigma = \frac{3Fl}{2bh^2}, \quad (1)$$

where F is the maximum load, in Newtons exerted on the specimen, l is the distance between the specimen supports, b and h are the width and height, respectively of the specimen.

The flexural modulus E was calculated from the stress-strain curve by the mechanical tester's computer with the following equation:

$$E = \frac{F_1 l^3}{4bh^3 d}, \quad (2)$$

where F_1 is the load at a convenient point in the straight line portion of the curve, d is the deflection at F_1 . In our case F_1 was between 10 and 15 N.

A requirement that a material complies with ISO 4049 [16] in terms of flexural strength is that the mean flexural strength $\bar{\sigma}$ is larger than N where

$$N = (\bar{E} \times 0.0025) + 40 \quad (3)$$

and \bar{E} is the mean flexural modulus. A material fulfils the flexural strength requirements of ISO 4049 when its mean flexural strength is not lower than the value of N , and in any case not lower than 50 MPa. A more detailed and extended description of the sample preparation, especially the storage procedures of the specimen in water, and the test procedures can be found elsewhere [16].

The results of the flexural strength tests were analysed with a three-way ANOVA at a 95% confidence level, factors being the LCUs, the composites, and composites' shades. Multiple range tests (Fisher's LSD procedure) at a 95% confidence level were performed to identify homogenous groups.

3. Results

The results of the flexural tests are summarised in Table 2. From Table 2 it can be seen that all the materials but one fulfil the ISO 4049 [16] requirement in terms of flexural strength, i.e. they have a mean flexural strength value of greater than 50 MPa. In most cases, however, the flexural strength value required by the standard was exceeded by far for both LCUs. However, Solitaire, shade A3, polymerised with the LED LCU achieved a mean flexural strength of 49 MPa (standard deviation 2.9), which is 1 MPa below the value required by the standard. A three-way ANOVA at the 95% confidence level shows that the factors *LCU* ($P = 0.027$) and *material*

($P = 0.000$) have a statistically significant effect on the flexural strength whereas the factor *shade* does not ($P = 0.954$). In other words the mean flexural strength of all specimens polymerised with the halogen LCU is statistically significantly greater than the mean flexural strength of all specimens polymerised with the LED LCU.

A multiple range test (Fisher's LSD procedure) at the 95% confidence level shows that there are no statistically significant differences in the mean flexural strengths in the Spectrum group and the Solitaire group (homogenous groups), respectively. In other words, there are no statistically significant differences in flexural strengths in these groups when polymerised with either LED LCU or a conventional halogen LCU, regardless of the shade. This applies also for Z100 with the exception of the shade A4, polymerised with a halogen LCU.

A three-way ANOVA at the 95% confidence level shows the factors *LCU* ($P = 0.000$), *material* ($P = 0.000$) and *shade* ($P = 0.005$) have a statistically significant effect on the flexural moduli. In other words the mean flexural modulus of all specimens polymerised with the halogen LCU is statistically significantly greater than the mean flexural modulus of all specimens polymerised with the LED LCU.

For the mean flexural moduli the first three samples of Spectrum shown in Table 2 form a homogenous group, i.e. there are no statistically significant differences in mean flexural modulus for these samples when they are polymerised with either an LED LCU or a conventional halogen LCU. For the other materials only certain pairs with particular shades do not differ significantly statistically in their mean flexural moduli, an example being Z100, shade A2, polymerised with a halogen LCU and Z100, shade A4, polymerised with the LED LCU.

Table 2
Mean flexural strength and modulus values for the materials tested^a

Material/shade	LCU	Mean flexural strength (MPa)	Homogenous groups flexural strength	Mean flexural modulus (GPa)	Homogenous groups flexural modulus	N value (MPa)	ISO requirements fulfilled
Spectrum TPH/A2	Halogen	122.7 (SD 9.1)	× ×	9.6 (SD 0.6)	× ×	64.0	✓
Spectrum TPH/A2	LED	120.9 (SD 14.0)	×	9.2 (SD 0.6)	×	62.9	✓
Spectrum TPH/A4	Halogen	117.2 (SD 11.3)	×	9.1 (SD 0.8)	×	62.8	✓
Spectrum TPH/A4	LED	111.7 (SD 10.4)	×	9.8 (SD 0.6)	×	64.4	✓
Z100/A2	Halogen	135.2 (SD 18.8)	×	13.0 (SD 0.9)	× ×	72.4	✓
Z100/A2	LED	133.1 (SD 23.6)	× ×	12.3 (SD 0.6)	×	70.7	✓
Z100/A4	Halogen	147.8 (SD 18.1)	×	13.3 (SD 0.7)	×	73.1	✓
Z100/A4	LED	138.6 (SD 14.1)	× ×	12.7 (SD 0.8)	× ×	71.8	✓
Solitaire/A2	Halogen	56.3 (SD 9.3)	×	4.8 (SD 0.5)	×	51.9	✓
Solitaire/A2	LED	50.1 (SD 6.6)	×	3.6 (SD 0.1)	×	49.1	✓
Solitaire/A3	Halogen	56.8 (SD 5.9)	×	3.9 (SD 0.4)	×	49.8	✓
Solitaire/A3	LED	49.0 (SD 2.8)	×	3.3 (SD 0.1)	×	48.1	×

^aSD: standard deviation. Homogenous groups, i.e. groups without significant difference at a 95% confidence level are indicated by a × (Fisher's LSD procedure). The N values are also given. A material fulfils the flexural strength requirements of ISO 4049 when its mean flexural strength is not lower than the value of N , and in any case not lower than 50 MPa. Note that all materials fulfil ISO 4049 requirements except Solitaire shade A3.

For all materials, the mean flexural strength is larger than the N value. All materials except Solitaire, shade A3 fulfil the ISO 4049 [16] in terms of flexural strength when polymerised with either an LED LCU or a conventional halogen LCU.

4. Discussion

A number of studies have addressed the application of blue LED technology to cure dental materials [10–13]. For example, an LED source containing 61 individual blue LEDs with a typical peak wavelength of 470 nm was used in a previous study [11]. This LED source produced a depth of cure and a degree of monomer conversion that was significantly greater than that obtained with a halogen source, even though all the sources were adjusted to give the same irradiance of 100 mW cm^{-2} . Mills and Jandt used an LED LCU containing 25 blue LEDs with an irradiance of 290 mW cm^{-2} and a conventional halogen LCU that was adjusted to an irradiance of 300 mW cm^{-2} to compare the depths of cure of dental composites [12]. Under these conditions, the LED LCU cured (polymerised) fine filled, microfilled, midfilled, and hybrid composites of medium shades significantly deeper than did the halogen LCU.

A recent study [13] compared the performances of a commercial halogen LCU with an irradiance of 755 mW cm^{-2} and LED LCU with an irradiance of 350 mW cm^{-2} . It was shown that there is no statistically significant difference in the compressive strength of composites polymerised with either LCU. Both LCUs exceeded by far the minimum of composite depth of cure according to ISO 4049 [16]. The LED LCU did not polymerise the composites as deep as the halogen LCU. The differences of depth of cure between the LCUs were small but statistically significant. It is surprising that there are only minor differences in the composite's mechanical performance when polymerised with one or the other LCU although the LED LCU had less than half of the halogen LCU's irradiance.

To further judge the ability of the LED LCU to polymerise composites a suitable test had to be first chosen. Clinically it is the physical properties of a dental material that are important. Hence, indirect tests such as fourier transform infrared (FTIR) spectroscopy to determine the degree of cure of the resin may be considered as second best. Mechanical properties including compressive strength, flexural strength and flexural modulus are important factors to be studied. This applies especially for restorative materials that are used where high biting forces and stresses can exacerbate inherent materials defects, resulting in inadequate fracture resistance of the materials. Some mechanical tests such as depth of cure and compressive strengths tests have been carried out previously with the LCUs described in this study to

compare the LCUs' performances with different materials [13]. Flexural strength tests have been used previously to study the effects on conventional halogen LCUs light exposure on composites [17]. The method described in ISO 4049 to test flexural properties of composites is suitable as a simple qualitative pass/fail procedure for composite materials. A 'weak' LCU would show up by the specimen being insufficiently polymerised, and exhibiting low flexural strength and modulus. It was therefore decided to carry out flexural strength and flexural modulus tests.

With one exception, all materials polymerised with either the halogen unit or the LED unit in the present study fulfil the flexural strength required by ISO 4049. In most cases, however, the flexural strength value required by the standard was exceeded by far for both LCUs. The fact that there are no statistically significant differences in the mean flexural strengths for the three groups of materials (with the exception of Z100, shade A4, polymerised with a halogen LCU) is consistent with earlier results [13] where compressive strength was studied and no statistically significant differences between composites polymerised with either LCU were found. In addition both LCUs used in the present study previously exceeded minimum requirements in terms of composites' depth of cure according to ISO 4049 and the depth of cure and the composites' compressive strength stated by the manufacturer [13]. Although additional mechanical tests may be carried out in future, it appears that under laboratory conditions LED LCUs are a satisfactory alternative in composite curing. It, however, remains surprising that there are few significant differences in the composite's mechanical performance polymerised with either LCU despite the LED LCU having less than half the halogen LCU's irradiance.

To understand this phenomenon, one has to look at the spectral distribution of the emitted light of both LCUs. In an earlier study, the irradiance over the whole of the emitted light spectrum of the Spectrum LCU and the LED LCU was measured to be 755 and 350 mW cm^{-2} , respectively [13], i.e. the total irradiance of the halogen LCU is 2.2 times higher than the irradiance of the LED LCU. One might therefore expect the physical properties of composites cured with the halogen LCU to be superior to those cured with the LED LCU, but the situation is in fact not so simple. The rate of light-induced chain polymerisation is proportional to the square root of the light intensity [18]. In other words, the rate of polymerisation increases only 1.44 times when the intensity is doubled. A prerequisite to the application of this relationship to two LCUs with different light intensities is that the curing times and all other parameters (material, spectrum of emitted light, tip diameter, etc.) are kept constant. In the present case, the first prerequisite is fulfilled but not the second, since the spectra of emitted light and the tip diameters of the LCUs differ.

An assessment of the LED and the halogen LCUs performances simply based on a comparison of irradiances is therefore not straightforward.

Fig. 1a shows the spectral flux for both the Spectrum LCU and the LED LCU (after [13]). The spectral flux represents the optical power output from the LCUs in milliwatts at each given wavelength. The y-axis of the graph in Fig. 1a was calibrated from the power meter measurement for each LCU. The area under each curve represents the total power output of the respective device. Fig. 1b shows the spectral irradiance as a function of emitted wavelength for both units (after [13]). The spectral irradiance is the spectral flux divided by the area of the light guide tip, i.e. it depends on the tip diameter.

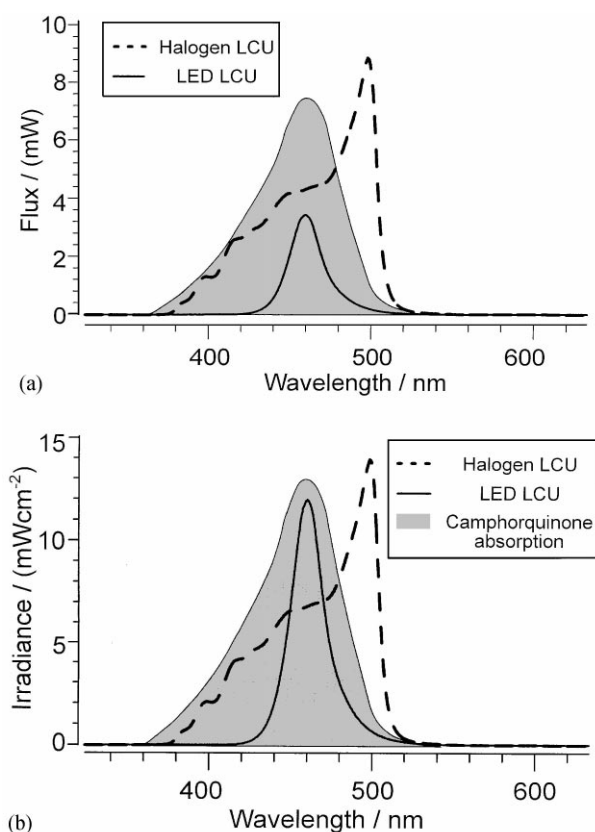


Fig. 1. (a) Spectral flux of both, the halogen (dotted curve) and the LED (bold curve) LCU used in this study (after [13]). Note the relatively sharp peak of the LED LCU at 465 nm. The flux of the halogen LCU exhibits a broader distribution than the LED LCU. (b) Spectral irradiance of both the halogen (dotted curve) and the LED (bold curve) LCU used in this study (after [13]). The curves take the different diameters of the LCU's light guides (9 mm for the halogen LCU and 6 mm for the LED LCU) into consideration. Note that both units produce their maximum irradiance in different regions of the spectrum. The fine line (grey area) shows the absorption spectrum of the camphorquinone used in Spectrum TPH (Manufacturer's information, Rahn AG, Zürich, Switzerland, shown for comparison only). The light absorption of the camphorquinone photoinitiator (fine line) envelops the LED curve.

The Spectrum LCU has a broad spectral distribution of emission with a peak at long wavelengths and a long tail to shorter wavelengths. Ninety-five per cent of the light output falls between 398 and 507 nm with a maximum at 497 nm. The full-width at half-maximum (FWHM) is 40 nm. The LED LCU, on the other hand, has a narrower peak in its spectrum with 95% of the emission being concentrated between 438 and 501 nm, and an FWHM of 23 nm. The LED LCU has a maximum spectral irradiance at 465 nm. In the region between 450 and 470 nm, the emission is almost twice that of the Spectrum LCU, but the Spectrum LCU exhibits a larger output in all other spectral regions, as shown in Fig. 1b.

The absorption curve of camphorquinone used in the Spectrum TPH composite (similar camphorquinone is used in the other two materials) has been superimposed on the curves of the spectral irradiance from both LCUs in Fig. 1b. The absorption peak of the camphorquinone in methacrylate resin is 467 nm (Rahn AG, Zürich, Switzerland) which approximately coincides with the emission peak of the LED LCU at 465 nm. The most efficient wavelength for the degree of polymerisation conversion of the resin when camphorquinone is used as initiator has been reported to be 470 nm [19]. It was further shown by Nomoto [19] that in the 450–490 nm wavelength range the degree of conversion is only weakly sensitive to wavelength and the light intensity within this range is more important than the peak wavelength. Outside this range, however, the wavelength dependence is much stronger and the conversion rate drops rapidly [19]. Consequently, the optimum wavelength for polymerisation and the output of an ideal LCU should lie within the 450–490 nm range for a composite using camphorquinone as the initiator. Further examination of Fig. 1b shows that the FWHM is 23 nm (measured between 449 and 472 nm) for the LED LCU and well within the range specified by Nomoto [19] for an ideal LCU. The FWHM of the halogen LCU is 40.6 nm (measured between 463 and 504 nm).

To estimate the curing potential of the LCUs, their emission spectra and the absorption spectrum of the camphorquinone photoinitiator can be convoluted. We assume that the curing potential is proportional to the number of photons that are available for absorption and the probability that they will be absorbed. Assuming the emission spectrum $E(\lambda)$ gives the relative number of photons of a light source and that the absorption spectrum $A(\lambda)$ gives the relative probability that a photon of the wavelength λ will be adsorbed, the relative curing potential is

$$CP_{\text{Rel}} = E(\lambda)A(\lambda). \quad (4)$$

To compare the two LCUs, however, it is important to look at the integrated relative curing potential. Care must be taken that the integral is over a sufficient

bandwidth that covers all regions where $E(\lambda)A(\lambda) > 0$. This leads to an integrated relative curing potential

$$\text{ICP}_{\text{Rel}} = \int_{\lambda_1}^{\lambda_2} E(\lambda)A(\lambda) d\lambda \quad (5)$$

The values for the integrated relative curing potential are shown in Table 3. Despite the higher irradiance of the halogen LCU, the LED LCU possesses a slightly higher integrated relative curing potential than the halogen LCU as can be seen from Table 3.

Our measure of the curing potential is normalised to equal irradiances of both LCUs. If we assume that the polymerisation potential is proportional to the rate of light-induced polymerisation then it must be also considered that the polymerisation rate increases with the square root of the irradiance [18]. We, therefore, have to calculate the ratio of the irradiances of both LCU

$$R = \frac{\text{irradiance halogen LCU}}{\text{irradiance LED LCU}} = \frac{755 \text{ mW cm}^{-2}}{350 \text{ mW cm}^{-2}} = 2.16, \quad (6)$$

i.e. the halogen LCU should be $\sqrt{2.16} = 1.47$ times as efficient as the LED LCU. If one considers the wavelengths emitted by the LCUs, i.e., the results obtained with Eq. (5) in the calculations, the halogen LCUs efficiency is $1.47 \times 0.62 = 0.91$ compared to a value of 0.84 for LED LCU. That means the LED LCU has only 92% of the halogen LCUs performance (polymerisation rate). Hence, slightly higher results in flexural strength of materials polymerised with the halogen LCU compared with those polymerised with the LED LCU can be expected, which is what is observed.

Rather than only comparing irradiances of LCUs, it appears more useful to compare mechanical properties of composites cured with LED or halogen technology. Thus, although the differences in irradiances of the two types of LCUs are large, no statistically significant differences in the composites flexural strength have been found for most materials. This effect, which is in agreement with the results from other mechanical tests, may possibly be explained by the correlation between the absorption spectrum of the camphorquinone and the emission spec-

trum of the LED LCU. As blue LED technology continues to improve, LED curing should become a useful adjunct to existing curing methods, and the prospect of constructing a small compact torch-like LED LCU appears feasible.

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Table 3

Relative curing potential and integrated relative curing potential for both LCUs^a

LCU	Irradiance (mW cm ⁻²)	Relative curing potential	Integrated relative curing potential
LED	350	1.00	0.84
Halogen	755	0.74	0.62

^aNote that the LED LCU's curing potential is higher than that of the halogen LCU although the halogen LCU has a much higher irradiance.

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