# 利用雷射熱解音洩技術來評估牙用複合材內部的界面接合性質

# Lase Acoustic Emmision Thermal Technique for Investigating Filler/Matrix Interaction in Dental Composite


## 中文摘要

由於感測器及高速資料擷取系統的發達，一種用來研究複合材料中各部份接合狀態的技術－雷射熱解音洩技術(Laser Acoustic Emission Thermal Technique，簡稱 LAETT)已逐漸發展成熟。以雷射當熱源聚焦於牙用複合材會產生音洩訊號(Acoustic Emission, 簡稱 AE)，這與材料的斷裂或鍵結解離有關。若 AE 是檢測複合材斷裂的有效工具，則 75%酒精老化複合材的結果(兩者具相近的溶解參數)值得以此觀察。本研究中，測量經 75%酒精老化後牙用複合材之機械性質與 LAETT 測試後所釋放的音洩訊號，來研究複合材內部之界面接合性質。將模型樹脂(75/25 Bis-GMA/TEGDMA)與模型複合材(填料含量分別為 12.6、30.0 和 56.5 vol%，平均直徑為 8μm 之鋇玻璃粒子，分為矽化與未矽化兩種)製成 3.47mmH 直徑 X7.3mm 高度、4.65mm 直徑 X 2.2mm 厚度和 4.65mm 直徑 X0.5mm 厚度之試樣，分別進行壓縮試驗、直徑式壓縮試驗與 LAETT 測試。機械力學測試之試樣先浸泡於 37℃ 75%酒精中達 0、1、7、14 和 30 天，LAETT 試樣則浸泡 0、1、3、7 和 14 小時。採用具連續波的 $CO_2$ 雷射當熱源以產生 1-5watt 數種不同功率(可經由分光鏡及功率測量器得知並校正)，產生的音洩訊號則以高感度感測器接收，先經前置放大器(具 40dB 放大率與 100-300 kHz 帶通濾波範圍)再送至主系統(具 20dB 放大率與 10-1200 kHz 帶通濾波範圍)，將類比電壓訊號轉換成數位訊號，如 hit、count、energy 和 amplitude。並以掃瞄式電子顯微鏡觀察機械測試後的試樣斷面和 LAETT 測試後之試樣表面。結果顯示，以矽化填料所組成的複合材其機械性質有明顯增加，而 75%酒精會降低複合材的機械性質。而 LAETT 所產生的音洩訊號則與填料體積分率、填料矽化處理、雷射功率以及酒精浸泡時間皆有關；除了高樹脂含量(0 vol%組和 12.6 vol%組)之試樣外，音洩訊號隨雷射功率增加而增加，而且在相同瓦數時之音洩訊號量大致符合以下趨勢：模型樹脂組>12.6vol%組>30.0vol%組>56.5vol%組，同時未矽化組之訊號量普遍大於矽化組。模型樹脂在 1-2watt 時會出現低振幅集中式的音洩訊號，可能與樹脂的微斷裂現象有關。而高填料試樣之音洩釋出現象可依脆性材料之耐熱震性質解釋；經酒精浸泡後未矽化填料複合材的熱膨脹係數之增加可能與其基質／填料鍵結力之降低有關。雷射熱解音洩技術似乎可成為傳統機械測試有價值之輔助工具。

## 英文摘要

Due to development of data acqusition system, a new technique called Laser Acoustic Emission Thermal Technique has been successfully used to study the interfacial bonding status in epoxybased composites. Laser heating of dental composite will produce acoustic emission (AE) which may be related to fracture in dental composites. Storage in a food simulating liquid, i.e., 75% ethanol, having a solubility parameter approximating that of Bis-GMA resin has been shown to dramatically affect its mechanical properties. If AE is an effective tool for monitoring fracture process in dental composite then the composites' response to storage in 75% EtOH should be of interest. In this study, mechanical properties and laser-induced acoustic emission of ethanol-aged experimental dental composites were studied to evaluate filler/matrix interaction inside the materials. Model resin(75/25 Bis-GMA/TEGDMA)and model composites (filler content 12.6, 30.0 and 56.5 vol% silanated/unsilanated 8μm BaSiC6) were fabricated into 3.47mm diam. x 7.3mm length, 4.65mm diam. x 2.2mm thick and 4.65mm diam. x 0.5mm thick specimens, respectively, for compressive, diametral tensile and LAETT tests. Before test, the specimens for mechanical tests were soaked in 37℃ 75% EtOH for 0, 1, 7, 14 and 30 days and those for LAETT test for 0, 1, 3, 7 and 14 hours. A continuous wave CO2 laser (Ultra Lasertech RFCL-28)was used at several power levels in the range of 1 to 5 watts, which can be read and adjusted by a beam splitter and a power meter. Acoustic emission was detected by a high-sensitivity transducer (Model R-30), then filtered and processed by MISTRAS 2001(Physical Acoustic Corp., with built-in 20dB gain and 10-1200 kHz bandpass filter) with a 1220A preamplifier (40dB gain and 100- 300 kHz bandpass filter). Detected AE signals were converted to digital parameters such as hit, count, energy and amplitude. The fractured surfaces of mechanical test and laser-heated specimens were examined by a scanning electron microscope (Hitachi S-2400, Japan). The results showed that mechanical properties are enhanced by increasing of silanated filler content but overall reduced after ethanol storage. Acoustic emission generated by laser heating is dependent on the filler volume fraction, filler silane treatment, amount of laser power and storage time. Generally Acoustic emission increased with laser power up to 5 watts except for high resin-rich (0 vol% and 12.6 vol%) groups. The amount of AE signals generated by the same laser power followed the trend: model resin group > 12.6 vor% group>30.0 vol% group > 56.5 vol% group, as well as unsilanated group>silanated group. The acoustic signal released from the model resin produced a low amplitude clusterring pattern at 1-2 watt laser power, indicating the resin microcracking. The thermal stress-induced acoustic emission of high filler content composite (56.5 vol%) can be explained by thermal shock resistance property of

brittle material. The increased thermal expansion coefficient of unsilanated composites by ethaol storage might be correlated with the decreased filler/matrix interfacial bond stregth. Laser-induced AE appears to be a valuable adjunct to conventional mechanical testing.