Conversion-dependent stress relaxation in dental resins and composites

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Statement of Purpose: Shrinkage stress development during photopolymerization is one of the major drawbacks of using polymeric dental composites as it can be very detrimental to the interfacial bonding between the tooth surface and the composite. Stress development is a function of factors such as shrinkage, modulus and double bond conversion. At any given conversion, the stress value is a balance between stress development and relaxation. Understanding the stress relaxation at various conversion levels is critical to understanding the overall stress evolution process. In this work, stress relaxation is measured via two different approaches and the equivalence of both is explored.

Methods: Both unfilled and filled resin systems were used. The resin was composed of 2,2-bis[4-(2-hydroxy-3-methacryloxyprop-1-oxy)phenyl]propane (BisGMA) with triethyleneglycol dimethacrylate (TEGDMA) in the weight ratio 70:30. For the filled system, 70 wt% of 0.7 micron silanized Ba glass was used. The iniferter isopropylxanthic disulfide (IPD) at 0.1 wt% was used to stabilize the conversion at a particular value. The materials were irradiated with light at 70 mW/cm^2 for varying amounts of time to achieve a wide range of conversions. Specimens were made into 1x2x25 mm bars. Stress relaxation was measured using a universal testing machine using two different methods. Stress relaxation tests were performed in the 3-point bending mode in which samples were subjected to a constant strain of 2% for 1 hour and the decrease in stress monitored. In an alternate approach, samples were subjected to different strain rates (0.00075, 0.005, 0.015 and 0.075 min^{-1}) and the stress-strain slopes were calculated. The slopes for each sample were plotted against the strain rate and extrapolated to get a strain rate where the stress-strain slope would be zero.

Results: Figure 1 shows the plot of the stress-strain slopes at different conversion values for the filled system versus the strain rate. When the curve is extrapolated to zero stress-strain slope, the stress is zero but there is still a finite strain rate. At this strain rate there is a balance between the stress development due to the imposed strain and the internal stress relaxation. Figure 2 shows the plot of the inverse of the strain rate at zero stress-strain slope, which can be representative of the time for stress relaxation (τ_c), versus the conversion for both the systems. It can be seen that as the conversion reduces, τ_c also reduces till the materials reach about 50 % conversion. It then increases slowly as the materials get lower in conversion. It can also be seen that at lower conversions there is a crossover in the τ_c values for the filled and unfilled systems and the unfilled values exhibit higher τ_c than the filled systems for a particular conversion. Looking at the actual stress relaxation values obtained from the conventional stress relaxation tests it can be seen that they follow a similar trend (Figure 3).

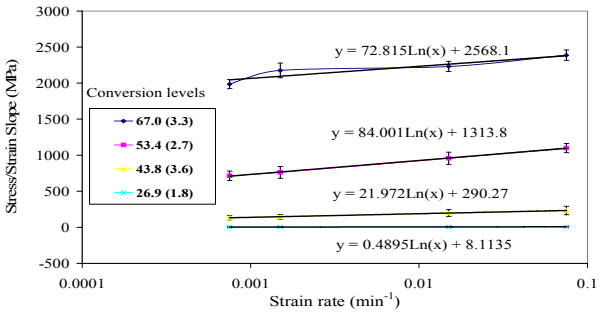

Figure 1. Variation of stress/strain slope with strain rate for the filled system.

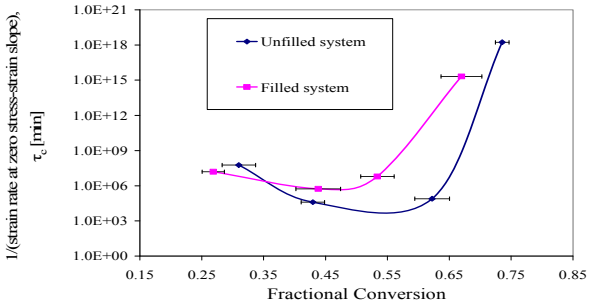

Figure2. Variation of inverse of strain rate at zero stress/strain slope with fractional conversion.

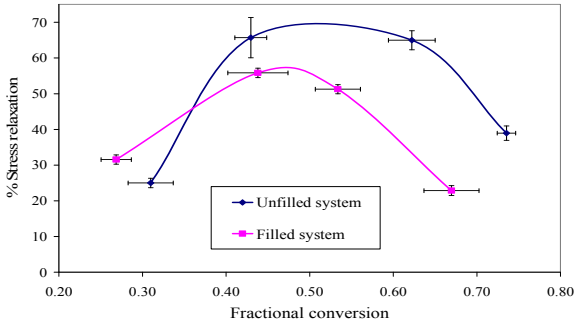

Figure 3. Change in stress relaxation with fractional conversion for both systems.

Conclusions: Both the methods described can be used to study the stress relaxation of dental materials. The advantage of using different strain rates is that it is much faster and yields similar information as compared with the conventional stress relaxation experiment. The knowledge gained from studying stress relaxation as a function of conversion can be extended to studying the effect of reaction rates on stress evolution. These can then be combined to form models that can predict the development of stress at various stages for a photopolymerizable dental composite.

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