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Analysis of the Results of a Laboratory Test on Resin Composite Cements for Resin-bonded Bridges

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This letter addresses the apparent controversy between the results of two studies published previously in this journal. In a laboratory test—a so-called “rigid set-up” tensile strength test based on a method described by Feilzer *et al.* (1987)—it was found that in about 70% of the experiments, the tensile bond strength could not be measured, because the specimens fractured in an early stage of setting (Verzijden *et al.*, 1992). These so-called “early failures” were seldom seen in a parallel clinical study in which the same resin composite cements (RCCs) were used to bond posterior resin-bonded bridges (Verzijden *et al.*, 1994). Before an explanation could be found for the wide variation of shrinkage stresses generated and the difference in behavior of the RCCs in the laboratory and clinical study, some additional laboratory tests were done and used for re-assessment of the rigid set-up test.

One of the additional laboratory tests was a tensile bond strength test in which the RCCs were allowed to contract freely during setting. Details of materials and methods were described earlier (Verzijden, 1993) and are available upon request. The tensile bond strength values found in the free shrinkage experiment were: 10.4 ± 3.8 MPa for the combination Clearfil F2/etching, 22.4 ± 5.9 MPa for Panavia EX/sandblasting, and 39.2 ± 7.1 MPa for Microfill Pontic/silicated when stored in water at 37°C for one hour; and 11.3 ± 3.3 MPa, 37.5 ± 8.7 MPa, and 42.8 ± 7.2 MPa, respectively, when thermocycled before being tested (1000 cycles, from 5 to 60°C). All specimens cemented with Clearfil F2 and Panavia Ex failed at the cement-substrate interface. Such a failure occurred in only 25% of the specimens fixed with Microfill Pontic.

In other tests, the diametral tensile strength (at different storage conditions), the volumetric shrinkage (according to

De Gee *et al.*, 1991), and the modulus of elasticity were determined. The results of these tests are presented in the Table. The maximum theoretically expected shrinkage stress (S_{Tmax}), calculated according to the formula $S_{Tmax} = 0.85 \cdot e_s \cdot E$ (where $e_s \cdot E$ refers to the relationship between volumetric shrinkage [e_s] and modulus of elasticity [E]) (Feilzer, 1989), is added to the Table. A re-evaluation of the suitability of the rigid set-up test was done by comparison of the tensile bond strengths and modes of failure (Verzijden *et al.*, 1992, Table 2) with: (1) those found with the free shrinkage laboratory test, and/or (2) those found with the diametral tensile strengths of the RCCs after storage for one hour at 23°C. This comparison showed equal modes of failure for both laboratory tests and comparable values of tensile strength and diametral tensile strength in cases of cohesive failure.

The results of the rigid set-up test (Fig. 3 of Verzijden *et al.*, 1992) suggest that with increasing setting rate, shrinkage stress development increases progressively (the Fig. shows two curves representing the development of shrinkage stress of Clearfil F2 in combination with silicate-coated substrates, one in which the specimen failed at an early stage and one in which the specimen remained intact. Both curves, starting at 0 min, show a horizontal and an ascending part. The transition of the horizontal into the ascending part corresponds with the transition of the RCC from the liquid into the solid state (L-S transition). The horizontal part of the “early failure” curve is shorter than that of the “late failure” curve, while the slope of its ascending part is steeper, indicating that in the “early failure” experiments, the average rate of setting has been higher than in the experiments in which the specimens remained intact).

As a demonstration of the effect of setting rate on the rate of shrinkage stress development, these rates are plotted against each other in the Fig. for the RCCs Clearfil F2 and Panavia Ex. As a measure of the rate of shrinkage stress development, the slope of the recording of the shrinkage stress is taken just after the transition of the RCC from its liquid to its solid state (V_s initial). For the setting rate, the tensile strength of the RCC at the end of an experiment is

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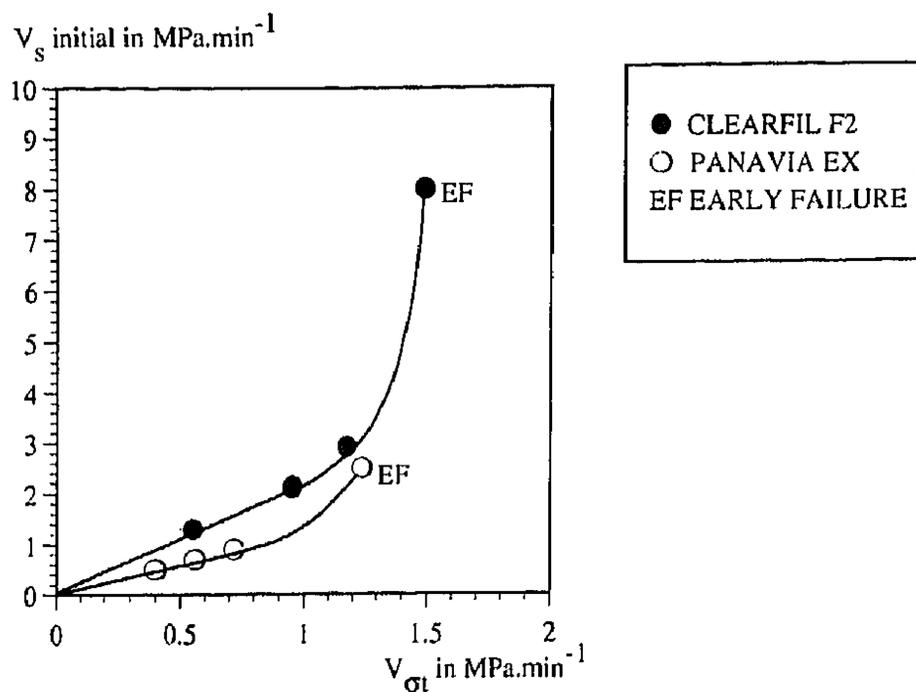


Figure. The initial rate at which the shrinkage stress (V_s) developed as a function of the rate setting (V_{σ}) for Clearfil F2 (●) and Panavia Ex (○) (EF: specimens fractured at an early stage of setting).

divided by the time counted from the L-S transition. For those experiments in which the specimens failed in an early stage, the tensile strength at the end of the experiment was taken as equal to the diametral tensile strength of the completely set RCCs (see Table, conditions C and D). Only the results of those experiments were used in which a cohesive mode of fracture was observed. From the Fig., it appears for setting rates of setting lower than 1 MPa per min, the rate of shrinkage stress development is directly proportional to the rate of setting. For setting rates over 1 MPa per min, the rate of shrinkage stress development increased progressively.

A possible reason for the variation in setting rates as seen in the Fig. may be the variation in the actual temperature within the RCC layers. The most plausible cause for this variation in temperature is a variation in the amount of heat generated in the RCC layer. The sooner the mixed RCC is inserted between the substrates, the larger the portion of the total heat liberated during setting added to the RCC layer will be. Although the time between mixing and insertion of the RCC was not registered, it appeared that in all "early failure" experiments, the time needed to prepare the

specimen after the mix was completed amounted to about 30 sec (all specimens were mounted by one operator highly experienced with this type of experiment). In the experiments in which the specimens remained intact, this time interval varied between one and 1½ min (these experiments were done by another operator, who was much less experienced with the performance of these experiments). Accordingly, we concluded that the shorter the time between complete mix and insertion of the RCC, the higher the actual temperature of the RCC layer and the higher the setting rate will be (resulting in an increase in the rate of shrinkage stress development).

Another point of interest is that, until the L-S transition, part of the total amount of the volumetric shrinkage of a RCC will be relieved by flow. The remaining part generates a shrinkage stress which (at some time during setting) might exceed the cohesive strength of the RCC. Since flow is a time-dependent phenomenon, the relief of the shrinkage stress decreases with an increase of the setting rate. In addition, at a certain degree of setting, the cohesive strength of the RCC will be lower when the actual temperature is higher. Moreover, according to Van Krevelen and Hoftijzer (1976), the modulus of elasticity of an elastomer at a certain degree of setting increases with an increase in temperature. Together, these conditions—a decrease of the relief of the shrinkage stress, an increase in the modulus of elasticity, and a temperature-related decrease of the cohesive strength of the RCC—most probably explain the occurrence of early failures and the wide spread in the shrinkage stresses measured.

Apparently, shrinkage stress is directly related to the time interval between completion of the mix and insertion of the RCC. The longer this time interval, the lower the final shrinkage stress and the higher the bond strength after complete setting. In the clinical study, insertion of the RCC usually needed from one to 1½ minutes after completion of the mix. This relatively long time might explain why early fractures were seldom observed in the clinical study.

Since the working time of an RCC is an indication of its intrinsic setting rate, it should be realized that its value depends on the ambient temperature at which it has been measured. In most of the manufacturers' instructions, temperature is not mentioned. It is recommended, therefore, that the setting rate of an RCC be measured at a

Table. The diametral tensile strength after different storage conditions, volumetric shrinkage, modulus of elasticity, and maximal theoretically expected shrinkage stress of the RCCs

RCC	Diametral Tensile Strength at				Volumetric Shrinkage (e_v) Vol%	Modulus of Elasticity (E) GPa	$S_{T_{max}}^c$ MPa
	1 hr, 23°C ^a	1 hr, 37°C ^a	100 hr 5-60°C ^b	100 hr, 37°C ^a			
Clearfil F2	30.8 (1.9) ^d	37.5 (6.4)	43.5 (1.7)	45.2 (1.2)	2.54 (0.16)	6.25 (0.73)	134.5
Panavia Ex	14.8 (2.6)	20.8 (4.0)	29.9 (6.0)	33.6 (2.1)	2.89 (0.29)	5.88 (0.49)	142.5
Microfill P.C.	26.3 (3.0)	29.4 (1.7)	41.6 (1.2)	41.0 (1.1)	4.16 (0.20)	5.67 (1.13)	199.8

^a Immersed in water.

^b Immersed in water thermocycled between 5 and 60°C (1000 cycles).

^c $S_{T_{max}}$: the calculated maximal theoretically expected shrinkage stress.

^d Mean in MPa (standard deviation) (n = 5).

standardized ambient temperature.

Comparison of all laboratory results brought us to the conclusion that the rigid set-up test is suitable to investigate the adhesive and cohesive properties of an RCC. However, to be of predictive value for the clinical situation, the test demands a precise imitation of the clinical circumstances. When, in the execution of the rigid set-up test, the time of insertion is adjusted and standardized accordingly, it is expected that the reliability of this test will be improved considerably. Moreover, the measured values of the shrinkage stress will better match the clinical situation.

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