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# Effects of strain rate and temperature on the mechanical properties of resin composites

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## KEYWORDS

Filled resin composite restorative materials;  
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Modulus of elasticity;  
Standardization

**Summary Objective.** To evaluate the effects of strain rate and temperature on the mechanical properties of resin composite restorative materials (RCs) and to investigate the construction of temperature-strain rate equivalence 'master curves'.

**Methods.** Four visible light-cured resin composite RCs, all of shade A3, were used: Heliomolar Radiopaque (HR) and Tetric Ceram (TC) (Ivoclar, Schaan, Liechtenstein), Filtek Z250 (FZ) (3M, St Paul, MN, USA) and Prodigy Condensable (PR) (Kerr, Orange, CA, USA). Bar specimens ( $1.0 \times 1.5 \times 16.0 \text{ mm}^3$ ) were cured for 50 s at an irradiance of  $500 \text{ mW cm}^{-2}$  and were randomly distributed into groups of six for each type of material. All specimens were stored in artificial saliva at pH 6, for 7 d. The specimens tested at 12, 24 and 37 °C were stored at the corresponding temperature but those tested at 0 °C were stored at 24 °C. Three-point bend tests for flexural strength ( $F$ ), flexural modulus of elasticity ( $E$ ) and total energy to failure ( $W$ ) were performed at cross-head speeds (XHS) of 0.1, 1.0, 10, 50 and 100  $\text{mm min}^{-1}$  for all materials as well as at 0.01, 0.03, 0.2 and 0.5  $\text{mm min}^{-1}$  for some materials.

**Results.** There was a common pattern of behavior across materials. At constant temperature,  $F$  showed a slight variation with cross-head speed, with a broad peak in the region of 1-10  $\text{mm min}^{-1}$ .  $E$ , on the other hand, showed a more marked and steady increase with XHS at all temperatures except at 0 °C, where it tended to level off above about 10  $\text{mm min}^{-1}$ . In contrast, the values of  $W$  showed a decline with increasing XHS, except at 37 °C where an initial rise followed by a decline was observed. At constant XHS, increase in temperature caused a small, but highly significant ( $P < 10^{-3}$ ) decline in  $F$  but a marked decline in  $E$ .  $W$ , again in contrast to  $F$  and  $E$ , showed a general increase with temperature. A master curve model for the temperature-strain-rate equivalence was fitted to the  $E$  and  $W$  data (all  $P < 10^{-5}$ ) and the fitted parameters interpreted in terms of strain rate and temperature sensitivity.

**Significance.** The mechanical properties of RCs are very sensitive to the test conditions of strain rate and temperature. This implies that properties determined at any temperature other than 37 °C, or at only one cross-head speed (or only one strain rate) are inadequate to describe their behavior in service. The master curve principle is applicable to RCs and can be used, inter alia, to determine property values under other than tested conditions. Conditions of testing in regard to XHS and temperature,

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as well as other factors, should clearly be stated to enable proper comparisons between studies, but more importantly the use of standardized test conditions is overdue.

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## Introduction

Resin composites (RCs) are being increasingly used in load-bearing areas of the posterior dentition and are therefore inevitably subject to masticatory forces of varying magnitude and rate of change as well as fluctuating temperatures. The polymer matrix is more susceptible to these changes than the filler and thus dominates the mechanical behavior of RCs. Polymers are characterized as viscoelastic materials,<sup>1,2</sup> that is, the mechanical properties are strain-rate dependent or, equivalently, sensitive to the rate of loading. Thus, given the circumstances of service in the mouth, the determination of the mechanical properties of RCs at one strain rate is, as a matter of principle, inadequate to explain or predict behavior in the oral environment.

More than just simple strain-rate sensitivity, cross-linked polymers, and therefore RCs alike, also exhibit dramatic changes in properties across the glass-transition temperature ( $T_g$ ), ranging from hard glass to rubber;<sup>1-3</sup> that is, they are also temperature-sensitive. It is understood that the setting reactions, depending as they do on diffusion, slow markedly when the cross-linking has proceeded far enough such that the  $T_g$  of the material has risen to the ambient temperature, essentially when the setting material has become glassy enough. Further elevation of the  $T_g$  does occur, but slowly. Obviously, this also depends on the thermal history of the restoration, especially when 'young'. This has implications for the storage conditions (time, temperature) of specimens for laboratory testing.

The typical maximum tooth surface temperature during the consumption of hot foodstuffs is  $\sim 47^\circ\text{C}$ ,<sup>4,5</sup> and although temperatures ranging from 0 to  $67^\circ\text{C}$  have been reported<sup>4</sup> such extremes are considered unrealistic.<sup>6,7</sup> Such fluctuations in oral temperature imply corresponding fluctuations in the observed mechanical properties of RCs.<sup>8,9</sup> However, mechanical testing of RCs is commonly done only at the prevailing room temperature ( $21-25^\circ\text{C}$ ), or at least is presumed to be in the absence of any explicit statement (see Table 1), or, more rarely, nominal body temperature ( $37^\circ\text{C}$ )—even if its relevance has been recognized,<sup>10</sup> despite the fact that storage before testing is often at this

temperature (Table 1). This has left the question of the mechanical behavior of RCs under service conditions largely unaddressed. Even published standards specifications have been ambivalent. For example, ISO 4049<sup>11</sup> had the material set at  $37^\circ\text{C}$  in distilled water, but the test was at  $23^\circ\text{C}$ , dry. These conditions are still required.<sup>12</sup>

The cross-head speed at which the determination of mechanical properties is done is a scaled proxy for strain rate. Unfortunately, there is no apparent consistency regarding the cross-head speed at which mechanical testing of resin composites: values in previous studies have ranged from 0.05 to  $12.7\text{ mm min}^{-1}$ , a 250-fold range (Table 1). Such variation complicates comparisons between studies. This is despite the existence of several published standards, although it must be said that their purpose is primarily for quality control rather than defining conditions under which scientific work should be done, a debate outside the present scope.

It is also apparent from Table 1 that while storage in distilled water is commonplace, presumably because the oral conditions were thought thereby to be better approached, it is notable that only rarely has the test medium been identified, and then only occasionally is it said to be distilled water, or the vaguer 'water', explicitly; media representing saliva in any way are rare. It can only be assumed that ambient air was the case otherwise. Given that it is well understood that absorbed water affects polymer properties, such oversight is unfortunate. Even ambient humidity has an effect.<sup>13</sup>

Thus, given the expectation of strain rate and temperature sensitivity in dental RCs, and the lack of published information on these matters, as well as the need to characterize products for clinical service, the primary objective of this study was to investigate the general behavior of this class of materials with a view, eventually, better to predict performance in the mouth.

There is a general principle in the mechanics of polymeric materials known as time-temperature equivalence, that is, that the outcome of a mechanical challenge at one timescale and temperature will be (essentially) the same as the outcome at a different timescale if the temperature is adjusted appropriately. Thus, response curves, say as a function of strain rate, at various

**Table 1** Conditions used in some strength tests for resin-based materials.

Reference	Material		Temp. (°C)	Medium	Test type	Cross-head speed (mm min <sup>-1</sup> )
27	Various	Store	37 ± 1	DW	Direct tension	0.0508
		Test	23 ± 2	NS		
28	Silicate, acrylic, Filled resin	Store	37 ± 2	" water"	Three-point bend	5.5
		Test	NS	NS(dry)		
29	Filled resin	Store	37	DW	Three-point bend	0.254
		Test	NS	NS		
30	Filled, unfilled resin	Store	37	Water	Diametral compression	0.5
		Test	RT	NS		
10	Composite	Store	RT	Air	Direct tension	(strain rate: 14 × 10 <sup>-3</sup> s <sup>-1</sup> )
		Test	RT	Air		
31	Filled/unfilled resin	Store	37	DW	Direct tension	1
		Test	NS	NS	Diametral compression	1
32	Light-cured composite	Store	37	DW	Compression	1 ± 0.1
		Test	RT (23 ± 2)	NS	Diametral compression	1 ± 0.05
33	Filled resin	Prep.	Various		Three-point bend Diametral compression	1 ± 0.05 0.1
		Store	NS	NS		
		Test	NS	NS		
34	Fiber-filled resin	Store	RT	Water	Three-point bend	(Load rate: 2 kg min <sup>-1</sup> )
		Test	NS	NS		
35	Filled resin	Store	37	Water	Compression	10
		Test	NS	NS	Stress-strain behaviour	0.05
36	Filled resin	Store	37	DW	Four-point bend	0.2
		Test	37	Wet		
37	Filled resin	Store	RT	Tap water	Three-point bend	0.5
		Test	NS	NS		
19	Denture acrylic	Store	37	DW	Three-point bend	5
		Test	37	Water	Four-point bend	5
38	Glass ionomer - resin mixture	Store	37	100% RH	Compression	0.127
		Test	NS	NS	Diametral compression	12.7
39	Filled, unfilled resin	Store	23	DW	Fracture toughness	0.127
		Test	NS	NS	Three-point bend	1
40	Filled resin	Store	37 ± 0.5	DW	Direct tension	10
		Test	NS	NS		
41	Heat-treated restorative resin	Store	37	Water	Diametral compression	10
		Test	NS	NS	Three-point bend	1
42	Filled resin	Store	37	DW	Four-point bend	0.2
		Test	37	Wet		
43-47	Filled resin	Store	37	Water	Diametral compression	10
		Test	NS	NS	Three-point bend	1
48	Filled resin	Store	37	DW	Diametral compression	0.508
		Test	NS	NS		
8	Filled resin	Store	37	100% RH	Three-point bend	0.05
		Test	various/RT	Water/air		
49	Filled resin	Store	37	DW	Fracture toughness	0.127

*(continued on next page)*

Table 1 (continued)

Reference	Material		Temp. (°C)	Medium	Test type	Cross-head speed (mm min <sup>-1</sup> )
50	Unfilled resin	Test	NS	NS	Three-point bend	0.254
		Store	NS	NS	Three-point bend	5
		Test				
51	Filled resin	Store	37	0.9% Saline	Three-point bend	0.5
		Test	NS	NS	Compression	0.5
52	Filled resin	Prep	23 ± 1	50 ± 5% RH	Three-point bend	0.5
		Store	37	DW	Fracture toughness	0.5
		Test	NS	NS		
53	Filled resin	Store	37	DW	Tension	0.5
		Test	23 ± 1	" Blotted dry"	Compression	0.05
54	Compomer	Store	37	Buffers, DW	Shear punch	2
		Test	NS	NS		
55	Filled resin	Store	37	DW	Fracture toughness	0.13
		Test	NS	NS	Three-point bend	0.254
56	Compomer, filled resin	Store	37	Various	Compression	0.5
		Test	NS	NS	Diametral compression	0.5
57	Filled resin	Store	NS	NS	Three-point bend	0.1
		Test	NS	NS	Compression	0.5
					Diametral compression	0.5
58	Filled resin	Store	NS	NS	Shear	0.5
		Test	NS	NS	Three-point bend	5
59	Filled resin	Store	37 ± 1	NS	Compression	0.5
		Test	NS	NS	Diametral compression	1
60	Filled resin	Store	37	DW	Three-point bend	0.5
		Test	NS	NS	Tensile	0.1
					Three-point bend	0.1
61	Fiber-filled resin	Store	37, 5 ~ 55	Water	Tensile bond strength	0.5
		Test	NS	NS	Three-point bend	1
62	Filled resin	Store	37	DW	Three-point bend	0.75
		Test	NS	NS		
63	Filled resin	Store	37	Water	Compression	1
		Test	NS	NS		
64	Filled resin	Store	37	DW	Diametral compression	10
		Test	NS	NS	Three-point bend	0.75
					Fracture toughness	0.5
65	Filled resin	Store	37	Saline	Three-point bend	0.5
		Test	37/23	Saline/NS	Fracture toughness	0.5
66	Filled resin	Store	37	Water	Three-point bend	0.75
		Test	37	Water		
67	Unfilled resin	Store	NS	NS	Biaxial flexure	1
		Test	NS	NS		
68	Fiber-filled resin	Store	37	DW	Three-point bend	0.5
		Test	NS	NS		
69	Fiber-filled resin	Store	37	DW	Three-point bend	1
		Test	NS	NS	Compression	1
					Fracture toughness	1
70	Fiber-filled resin	Store	37	DW	Three-point bend	1
		Test	NS	NS		
71	HA filled resin	Store	37	DW	Three-point bend	0.75
		Test	NS	NS		
9	Filled resin	Store	37	~ 100% RH	Three-point bend	0.5

(continued on next page)

Table 1 (continued)

Reference	Material		Temp. (°C)	Medium	Test type	Cross-head speed (mm min <sup>-1</sup> )
		Test	various/RT	Water/air	Diametral compression	5
72	Filled resin	Store	37, 4 ~ 60	DW	Three-point bend	5
		Test	NS	NS		
73	Filled resin	Store	37	DW	Three-point bend	0.1
		Test	NS	NS	Fracture toughness	0.1
74	Filled resin	Store	RT	DW	Three-point bend	0.75
		Test	NS	NS		
75	Filled resin	Store	37	DW → DX	Three-point bend	0.75
		Test	NS	NS		
76	Fiber-filled resin	Store	37	Water	Three-point bend	1
		Test	NS	NS		
77	Filled, unfilled resin	Store	NS	NS	Direct tension	1
		Test	NS	NS	Three-point bend	0.75
					Compression	1
					Fracture toughness	10
78	Filled resin	Store	37	Water	Diametral compression	1
		Test	NS	NS	Fracture toughness	0.25
79	Filled resin	Store	37	Water	Three-point bend	0.75
		Test	37	Water		
80	Fiber-filled resin	Store	NS	NS	Three-point bend	0.5
		Test	NS	Air	Biaxial flexure	0.5
81	Filled resin	Store	37	DW	Three-point bend	0.75
		Test	37	Water		
82	Fiber-filled resin	Store	7 ~ 63	Water	Three-point bend	2
		Test	NS	Air, water	Fatigue (3-point bend)	2

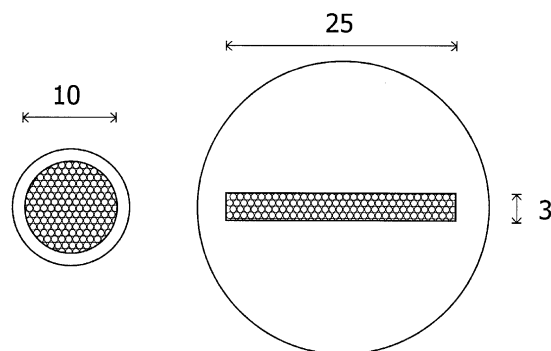
DW, distilled or deionized water; DX, desiccated; NS, not stated; RT, room temperature; RH, relative humidity; HA, hydroxyapatite; ~ for temperature, cycled between limits.

temperatures may be superposed by applying a systematic offset to generate a master curve.<sup>2</sup> As such a procedure allows data from a variety of experimental regimes to be combined, and predictions made about other regimes, it was a further objective of this work to determine whether such a master curve could be constructed for this class of material.

## Materials and methods

Three types of material were studied, one microfill: Heliomolar Radiopaque (HR) (Batch A20633, Ivoclar, Schaan, Liechtenstein), two hybrid: Tetric Ceram (TC) (Batch A17994, Ivoclar) and Filtek Z250 Universal Restorative (FZ) (Batch OFY, 3M Dental Products, St. Paul, MN, USA), and one packable, Prodigy Condensable (PR) (Batch 007686, Kerr, Orange, CA, USA), all of shade A3. Bar specimens of gage length 16 mm, section  $1.0 \times 1.5 \text{ mm}^2$ , were prepared using the split aluminum mold previously described<sup>14</sup> at  $24 \pm 1 \text{ }^\circ\text{C}$ ,  $50 \pm 5\%$  RH. They were cured using a modified plasma-arc lamp (Arc Light

IIM, Air Techniques, Hicksville, NY, USA) for 50 s at an irradiance of  $500 \text{ mW cm}^{-2}$  (Demetron 100, Demetron Research, Danbury, CT, USA) referenced through a silicon detector (DET110, ThorLabs, Newton, NJ, USA) and conventional curing lamp (Luxor-4000, ICI, Macclesfield, UK); the liquid-filled light guide was replaced by a special fiber-optic



**Figure 1** Schematic diagram of the adit and exit configurations of the special fiber-optic for irradiation of the beam specimens in one shot (dimensions in millimeters). Fibers were randomized before the exit better to ensure even irradiation.

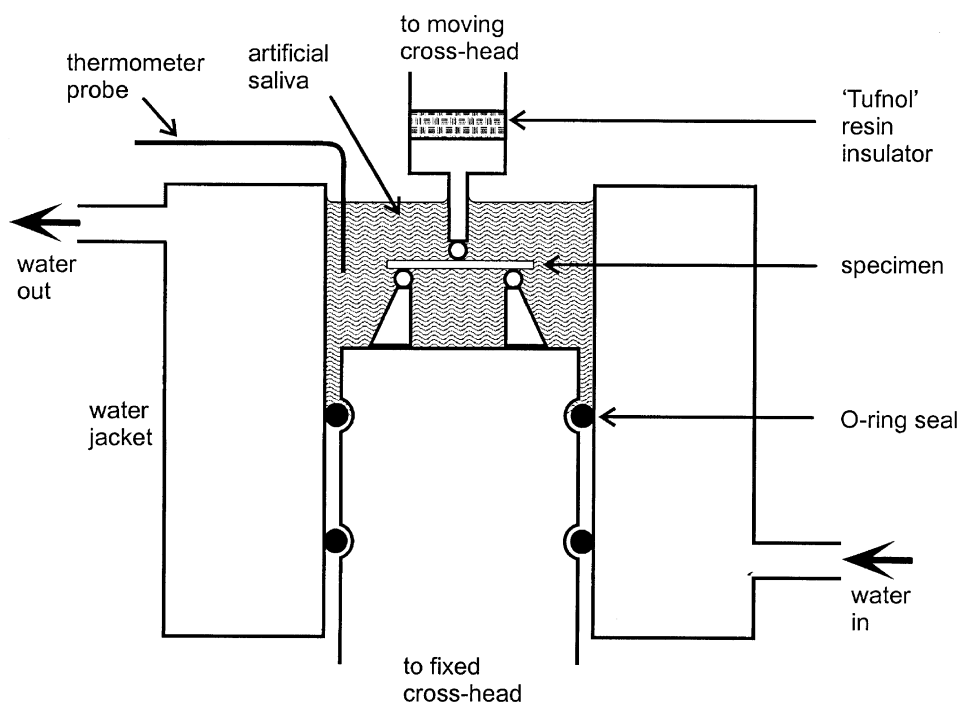


Figure 2 Schematic diagram of three-point bend test set-up. Exterior insulation and draft-shielding not shown.

light guide (C Technologies, Cedar Knolls, NJ, USA) for single-shot exposure of the whole specimen (Fig. 1). Specimens were randomly distributed into groups of six for each type of material (total: 156 TC, 144 HR, 120 each FZ and PR). All specimens were stored in artificial saliva (AS)<sup>15</sup> at pH 6 for 7 d before testing. The pH was chosen to represent that of resting saliva, being the time-majority condition of exposure in service;<sup>15</sup> 7 d was chosen to allow post-irradiation reactions to proceed to completion and allow equilibration with respect to water uptake.<sup>13</sup> Those to be tested at 12, 24 and 37 °C were stored at the corresponding temperature but those tested at 0 °C were stored at 24 °C as it was felt that the post-irradiation reactions might be slowed too much, and any interference would be detected by inconsistency in response.

Specimens were tested immersed in AS.<sup>15</sup> Temperature control was obtained via a thermostatted water-jacket, the temperature of the circulating water being adjusted to give the intended test temperature at the specimen site (Julabo VC, Julabo Labortechnik, Seelbach, Schwarzwald, Germany) (Fig. 2), monitored by a type-K thermometer probe (model 2013, Kane-May, Burrowfield, UK). The temperatures and the cross-head speeds used for each material are summarized in Table 2.

Mechanical testing of the specimens to determine flexural strength, flexural elastic modulus and total energy to failure was done in three-point bend (16 mm span) using a universal testing machine

(model 1185, Instron, High Wycombe, UK). Data acquisition was done in software (LabVIEW version 5.1, National Instruments, Austin TX, USA) on a personal computer, as was the programmed calculation of ultimate flexural strength ( $F$ ), using the corrected formula, and flexural elastic modulus ( $E$ ) as:

$$F = \frac{3PL}{2bd^2} \left[ 1 + \frac{6D^2}{L^2} - \frac{4dD}{L^2} \right] \quad (1)$$

$$E = \left( \frac{\Delta P}{\Delta y} \right) \frac{L^3}{4bd^3} \quad (2)$$

where  $P$  is load to fracture,  $L$  the support span,  $D$  the maximum deflection of the beam,  $d$  and  $b$

Table 2 Summary of test conditions of cross-head speed and temperature used for the four materials.

Cross-head speed (mm min <sup>-1</sup> )	Temperature (°C)			
	0	12	24	37
0.01	TC, HR			
0.03	TC			
0.1	All	All	All	All
0.2	TC			
0.5	TC, HR		TC, HR	TC, HR
1	All	All	All	All
10	All	All	All	All
50	All	All	All	All
100	All	All	All	All

TC, Tetric Ceram; HR, Heliomolar Radiopaque; FZ, Filtek Z250; PR, Prodigy Condensable.

specimen depth and breadth, and  $\Delta P/\Delta y$  the gradient of the (steepest) linear portion of the deflection curve.<sup>16</sup> Total energy to failure ( $W$ ) was calculated as the area under the load-displacement curve.

The elementary equations for beam-bending strictly apply only to materials for which stress is linearly proportional to the strain, that is to say, materials that obey Hooke's law.<sup>17</sup> Polymers as well as RCs are non-Hookean and therefore expressions of flexural strength in the three-point (and four-point bend) situations are strictly valid only for very small deflections, where stress remains nearly proportional to strain. In the region near the point of application of the load there are localized stresses, but those may be considered to be negligible for large span/depth ( $L/d$ ) ratios.<sup>17</sup> For beams with  $L/d$  of about 10 or more the shearing deflection is sufficiently small compared with the bending deflection that it can usually be neglected in practical testing.<sup>17</sup> The American Society for Testing and Materials<sup>18</sup> recommends a value for  $L/d$  of 16 (tolerance +4, -2). But thin beams (large  $L/d$ ) encourage large deflections and departure from simple beam theory, in which case a correction factor is required.<sup>16,18</sup> A significant difference has been reported between values of elastic modulus calculated using simple and corrected formulae for both the three- and four-point tests.<sup>19</sup> The ratios of flexural strength values for the two tests were more consistent using the corrected formulae<sup>19</sup> which are, therefore, preferred.

The two portions of the beam were retrieved after the failure of the specimen;  $d$  and  $b$  were measured at the fracture using a micrometer screw gauge (No. 193-111, Mitutoyo, Tokyo, Japan). Load string stiffness was sufficiently high that cross-head displacement was an accurate enough proxy for beam deflection, simplifying the set-up; the maximum displacement error was calculated to be about +1% of  $D$ , but the effect on the calculated value of  $E$  was much less because of the definition of  $\Delta P/\Delta y$ . Data were processed and plotted in software (SigmaPlot v.7, SPSS, Chicago, IL, USA).

Testing at cross-head speeds of  $10 \text{ mm min}^{-1}$  and above was done without the signal filters in the load cell amplifier in order to avoid time-constant damping. The signal filters are normally switched into the circuit to reduce noise (jitter) in the plot, that is, high-speed recorder pen movements are suppressed. However, at high XHS, this biases the recorded load downwards appreciably.

Based on the general pattern of the results, their strain-rate temperature equivalence was tested by the construction of master curves. Inspection showed the presence in general of a slight curvature

(concave to the abscissa) that was effectively removed by taking the logarithm of the dependent variable. Nominally, the temperature effect can be represented by an offset of the following form:

$$X' = X \times 10^{a T/10} \quad (3)$$

where  $X$  is the cross-head speed of the test,  $X'$  the adjusted (or offset) value,  $T$  the test temperature in °C, and  $a$  the offset rate in decades of XHS per 10 K (to work to a convenient scale). The interpretation of the offset value  $X'$  as defined is the cross-head speed at 0 °C that would give the same result as the higher temperature test actually conducted. The value of the shift parameter was estimated by least squares from the following equation:

$$\log_{10}(Z) = b_0 + b_1\{\log_{10}(X) + a(37 - T)/10\} \quad (4)$$

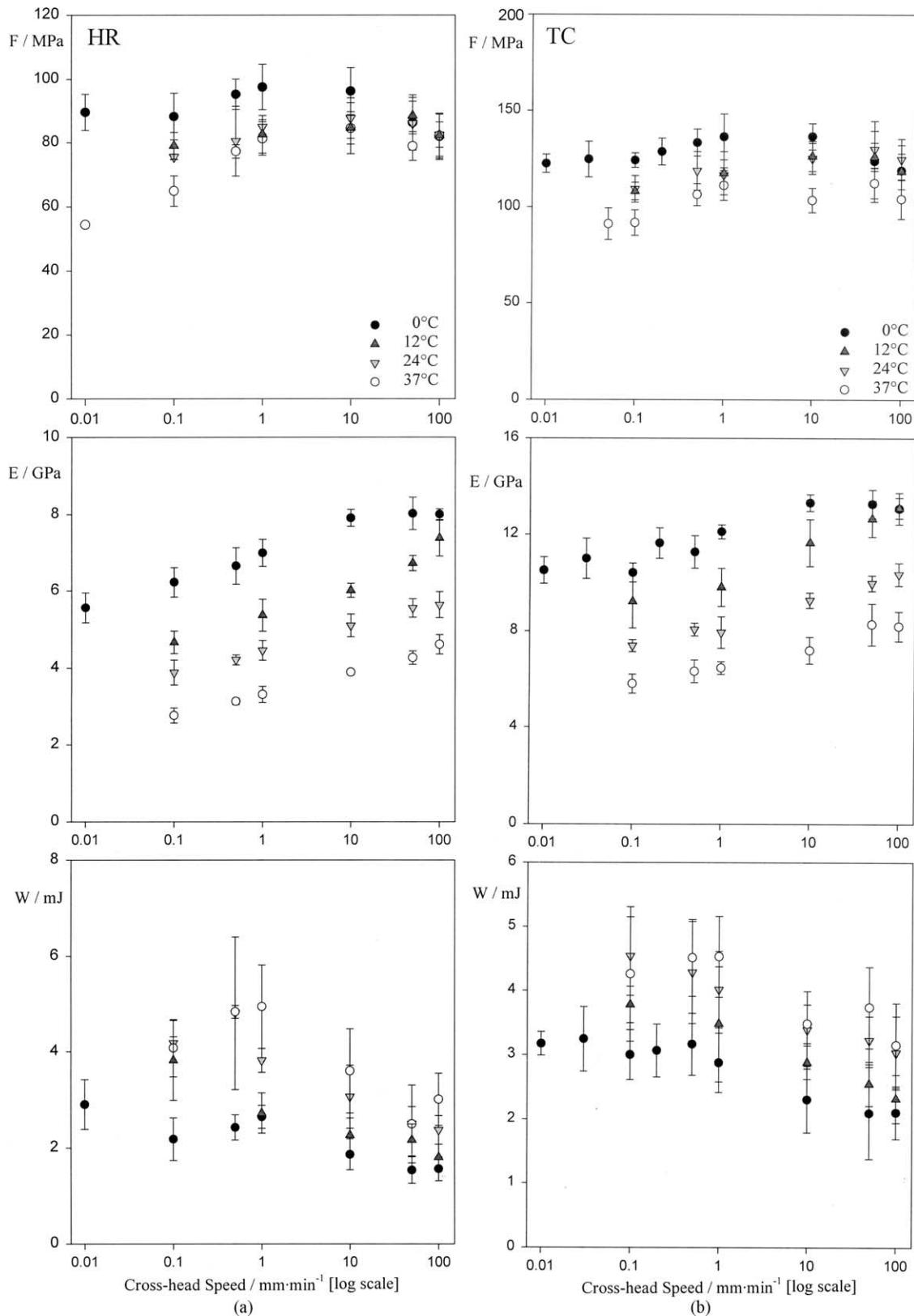
where  $Z$  is the relevant measured property ( $E$ ,  $W$ ), and  $b_0$ ,  $b_1$  are the constant and general slope estimates (TableCurve 3D v.4, Systat, Richmond, CA, USA), now choosing 37 °C as the reference temperature, and  $a$  is the offset rate, as above.

## Results and analysis

All materials under all conditions failed abruptly in an apparently brittle manner; there was no clearly distinguishable yield point. The results for the effects of strain rate and temperature on the mechanical properties of flexural strength ( $F$ ), flexural elastic modulus ( $E$ ) and total energy to failure ( $W$ ) of the four products tested are illustrated in Fig. 3a-d. The findings may be summarized as follows.

There is a general good agreement in the pattern of mechanical behavior for the three types of material tested (microfill, hybrid and packable) in response to the change in cross-head speed. At constant temperature,  $F$  showed a generally slight curvature with respect to cross-head speed (XHS), there being a maximum broadly in the region of  $1\text{--}10 \text{ mm min}^{-1}$ .  $E$ , on the other hand, showed a more marked and steady increase with XHS at all temperatures except at 0 °C where it tended to level-off at a XHS of above about  $10 \text{ mm min}^{-1}$ . In contrast, the values of  $W$  showed a decline with increasing XHS, except at 37 °C, where an initial rise followed by a decline was observed. At constant XHS, increase in temperature caused a general decline in  $F$  but a marked decline in  $E$ .  $W$ , again in contrast to  $F$  and  $E$ , showed a general increase with temperature.

For the construction of the master curves, noticing that at 0 °C and at XHS of 50 and  $100 \text{ mm min}^{-1}$ , the values for  $E$  seemed to level off, and similarly, at 37 °C and low XHS the values



**Figure 3** Variation of flexural strength ( $F$ ), modulus of elasticity ( $E$ ) and total energy to failure ( $W$ ) with cross-head speed at test temperatures of 0, 12, 24 and 37 °C. (a) Heliomolar Radiopaque, (b) Tetric Ceram, (c) Filtek Z250 and (d) Prodigy Condensable.

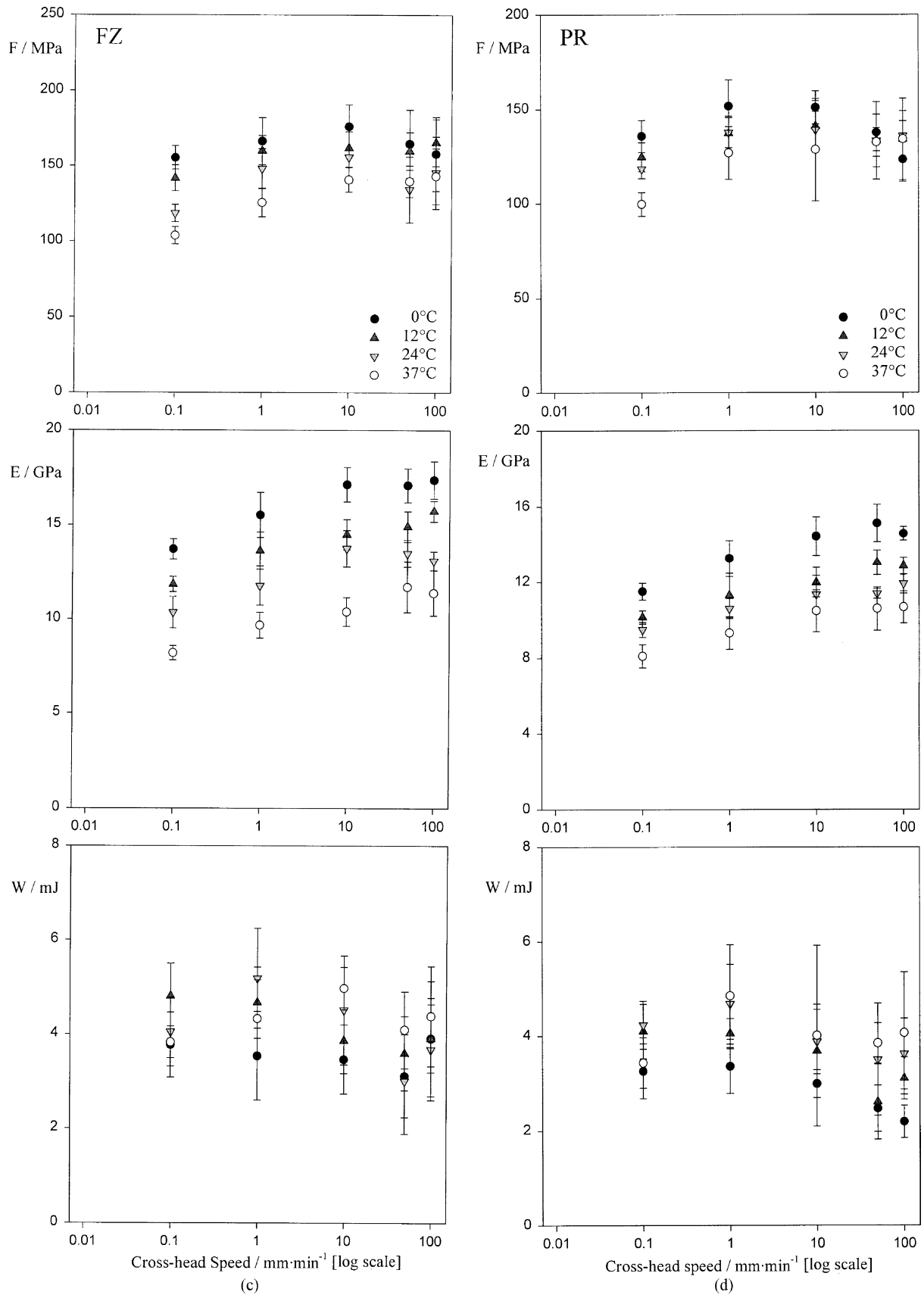


Figure 3 (continued)

**Table 3** Values and standard errors of fitted parameters of master curves for elastic modulus ( $E$ ) and work to failure ( $W$ ).

	HR	TC	FZ	PR
<b><math>E</math></b>				
$b_0$ ( $E_{ref}$ , error)	0.5234 ± 0.0042 <sup>a</sup> (3.34, 1.0%)	0.8160 ± 0.0046 <sup>b</sup> (6.55, 1.1%)	0.9805 ± 0.0054 <sup>c</sup> (9.56, 1.3%)	0.9600 ± 0.0053 <sup>d</sup> (9.12, 1.2%)
$b_1$ (SRS/decade, error)	0.0604 ± 0.0021 <sup>a</sup> (14.9%, 0.5%)	0.0504 ± 0.0022 <sup>b</sup> (12.3%, 0.5%)	0.0417 ± 0.0027 <sup>c</sup> (10.1%, 0.6%)	0.0354 ± 0.0027 <sup>c</sup> (8.5%, 0.6%)
$a$	1.438 ± 0.050 <sup>a</sup>	1.502 ± 0.063 <sup>a</sup>	1.340 ± 0.096 <sup>a,b</sup>	1.081 ± 0.097 <sup>b</sup>
$r^2$ , $n$	0.953, 122	0.917, 136	0.864, 110	0.784, 107
<b><math>W</math></b>				
$b_0$	0.6611 ± 0.0144 <sup>a</sup> < 1 × 10 <sup>-13</sup>	0.6582 ± 0.0139 <sup>a</sup> < 1 × 10 <sup>-13</sup>	0.7037 ± 0.0265 <sup>a</sup> < 1 × 10 <sup>-13</sup>	0.7127 ± 0.0238 <sup>a</sup> < 1 × 10 <sup>-13</sup>
$P$	< 1 × 10 <sup>-13</sup>	< 1 × 10 <sup>-13</sup>	9.6 × 10 <sup>-4</sup>	1.9 × 10 <sup>-8</sup>
$b_1$	-0.0869 ± 0.0060 <sup>a</sup> < 1 × 10 <sup>-13</sup>	-0.0617 ± 0.0056 <sup>b</sup> < 1 × 10 <sup>-13</sup>	-0.0433 ± 0.0115 <sup>b</sup> 9.6 × 10 <sup>-4</sup>	-0.0600 ± 0.0100 <sup>b</sup> 1.9 × 10 <sup>-8</sup>
$P$	< 1 × 10 <sup>-13</sup>	< 1 × 10 <sup>-13</sup>	1.3 × 10 <sup>-4</sup>	1.3 × 10 <sup>-7</sup>
$A$	0.989 ± 0.081 <sup>a</sup>	0.985 ± 0.102 <sup>a</sup>	0.765 ± 0.240 <sup>a</sup>	0.966 ± 0.175 <sup>a</sup>
$P$	< 1 × 10 <sup>-13</sup>	< 1 × 10 <sup>-13</sup>	1.3 × 10 <sup>-4</sup>	1.3 × 10 <sup>-7</sup>
$r^2$ , $n$	0.759, 129	0.594, 144	0.187, 101	0.424, 100

By row, superscript letters indicate distinguishable values at  $P < 0.05$  (Tukey test). All  $P < 1 \times 10^{-13}$ .

for  $W$  tended to decrease again, these values were excluded from the fitted data. The results of this analysis are shown in Table 3, and the master curves so generated in Fig. 4a–d. There was no indication that this could be done for  $F$ ; indeed, this was not expected. Instead, these data were treated by two-way analysis of variance (Table 4). The main effects, i.e. of temperature and cross-head speed, were both highly significant in each case (all  $P < 5 \times 10^{-4}$ ), and only the interaction for FZ failed to reach significance.

## Discussion

The finding that the mechanical properties of RCs depend on the conditions of the tests, i.e. cross-head speed and temperature, was not unexpected and is relevant to their performance and survival in the oral environment. The low values of  $E$  at low XHS and vice versa indicates that a relaxation mechanism exists that allows stress relief to occur. When viscoelastic materials are subjected to a constant strain, at a constant temperature, decay in the value of the stress originally obtained occurs.<sup>2,20</sup> This decay is due to internal flow, the process being called stress relaxation, and is clearly a time-dependent phenomenon.<sup>20</sup> Even if gross viscous flow does not occur because of cross-linking, chain segment relaxation will also decrease the observed value of  $E$ .<sup>2,21</sup> Conversely, at very short time-scales (high XHS), this does not occur, resulting in a high  $E$ . A similar explanation applies to  $W$ . Stress relief due to viscous flow or other relaxation processes causes the material to

absorb more energy to failure at a low XHS than at high.

The effect of temperature is due to the fact that the relaxation in polymers is by activated processes, being diffusive in nature. This accounts for the observed decline in  $E$  with increasing temperature at a constant XHS, as has been reported before,<sup>8,9</sup> despite the difference in immersion liquid. As indicated, stress relaxation enables the absorption of more energy before failure occurs, resulting in higher  $W$  values at elevated temperatures. The tendency to a peak  $W$  value corresponds to an expected peak in toughness for polymers in general.<sup>22</sup> Likewise, the tendency for  $E$  to level-off at  $\sim 50 \text{ mm min}^{-1}$  at 0 °C, in TC, HR and PR is taken as an indication of the glass transition ( $T_g$ ) at the corresponding strain rate where the material is then glassy and therefore brittle, but after which no great variation in  $E$  is expected. It can be noted that while the value of  $T_g$  is expected to be a little above the final setting (storage) temperature, when water absorption has occurred, as here, its plasticizing effect must lower the value.

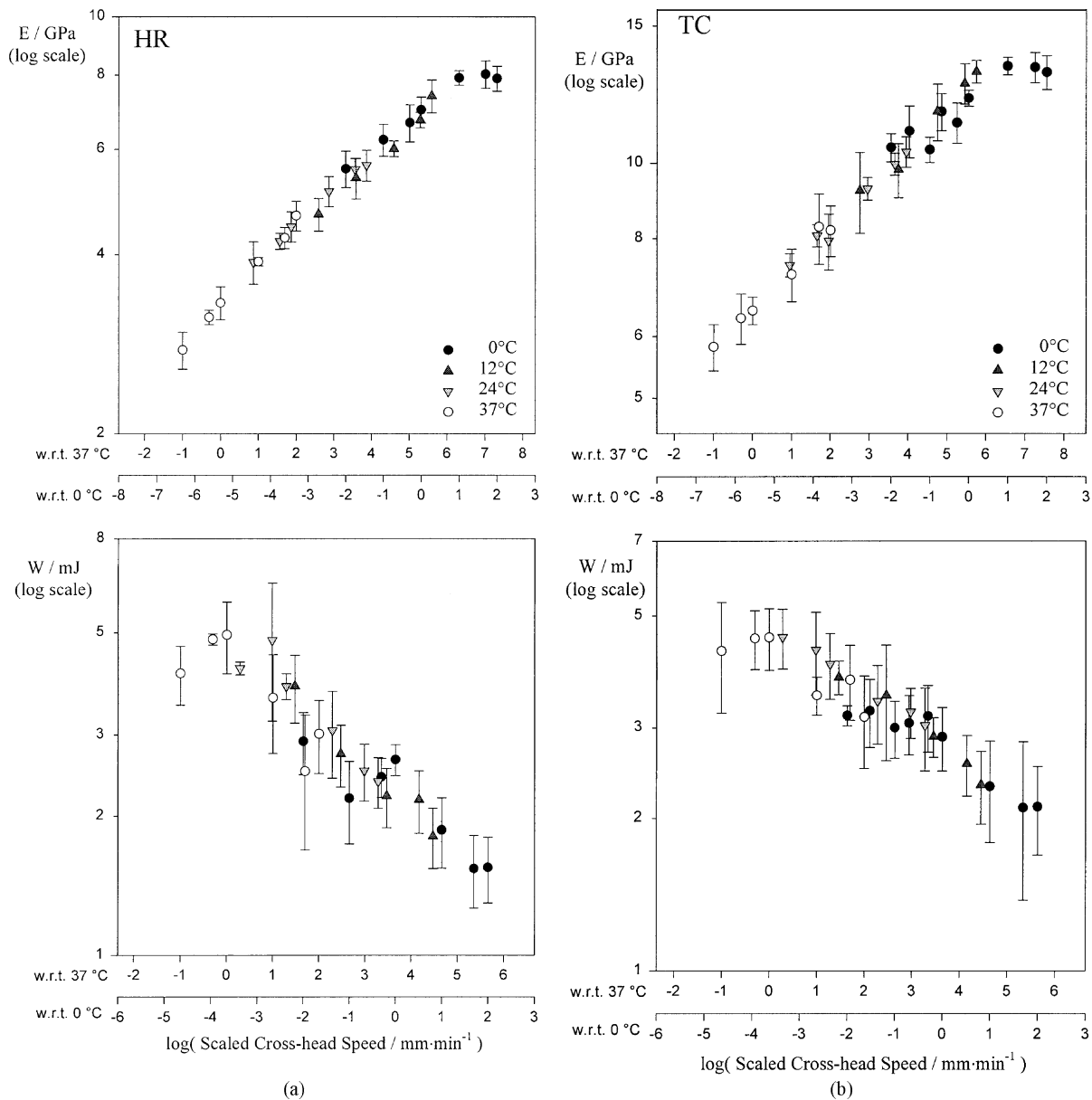
The general slight variation of  $F$  with XHS, with a tendency to show a slight peak, observed in all four materials, is also consistent with expected polymer behavior. Reduction in the time available for viscous flow as XHS is raised lowers the extent to which stress relief can occur and therefore results in increased  $F$  values. Such an effect has also been reported previously.<sup>8,9</sup> Nevertheless, the effects of both XHS and T on  $F$  are highly significant (Table 4), and there is a very clear need to control and standardize both closely if spurious differences in

observed values are to be avoided. Even then, problems may remain that require careful analysis,<sup>23</sup> for example environmental conditions at preparation, nature of platen contact<sup>24</sup> and surface finish, to say nothing of the detail of an individual's manner of preparation and handling of specimens.

In contrast, the variation of both  $E$  and  $W$  with temperature was both more marked and more regular, both consistent with the concept of time-temperature equivalence.<sup>2,21</sup> Based on this, a master curve was constructed for  $E$  and  $W$  for each material (Fig. 4). Superficially, this bears a resemblance to the 'universal function' of Williams,

Landel and Ferry<sup>25</sup> but, as they themselves were at pains to point out, their particular approach is inapplicable near to and below the glass transition. Even so, in view of the general strain-rate sensitivity of polymers, including at temperatures below the glass transition, the attempt was made, and can be seen to have been very effective. The approach taken here permits direct physical interpretation of the fitted parameters without arbitrary factors as the reference temperature was chosen to match the service condition.

It can be seen that, taken as a whole, the four materials are distinguishable by their patterns of



**Figure 4** Master curves for modulus of elasticity ( $E$ ) and total energy to failure ( $W$ ) against equivalent cross-head speed referred to  $1 \text{ mm min}^{-1}$  with respect to (w.r.t.) a reference temperature of, upper abscissa,  $37^\circ\text{C}$ ; lower abscissa,  $0^\circ\text{C}$ , derived from fitting Eq. (4). (a) Heliomolar Radiopaque, (b) Tetric Ceram, (c) Filtek Z250 and (d) Prodigy Condensable.

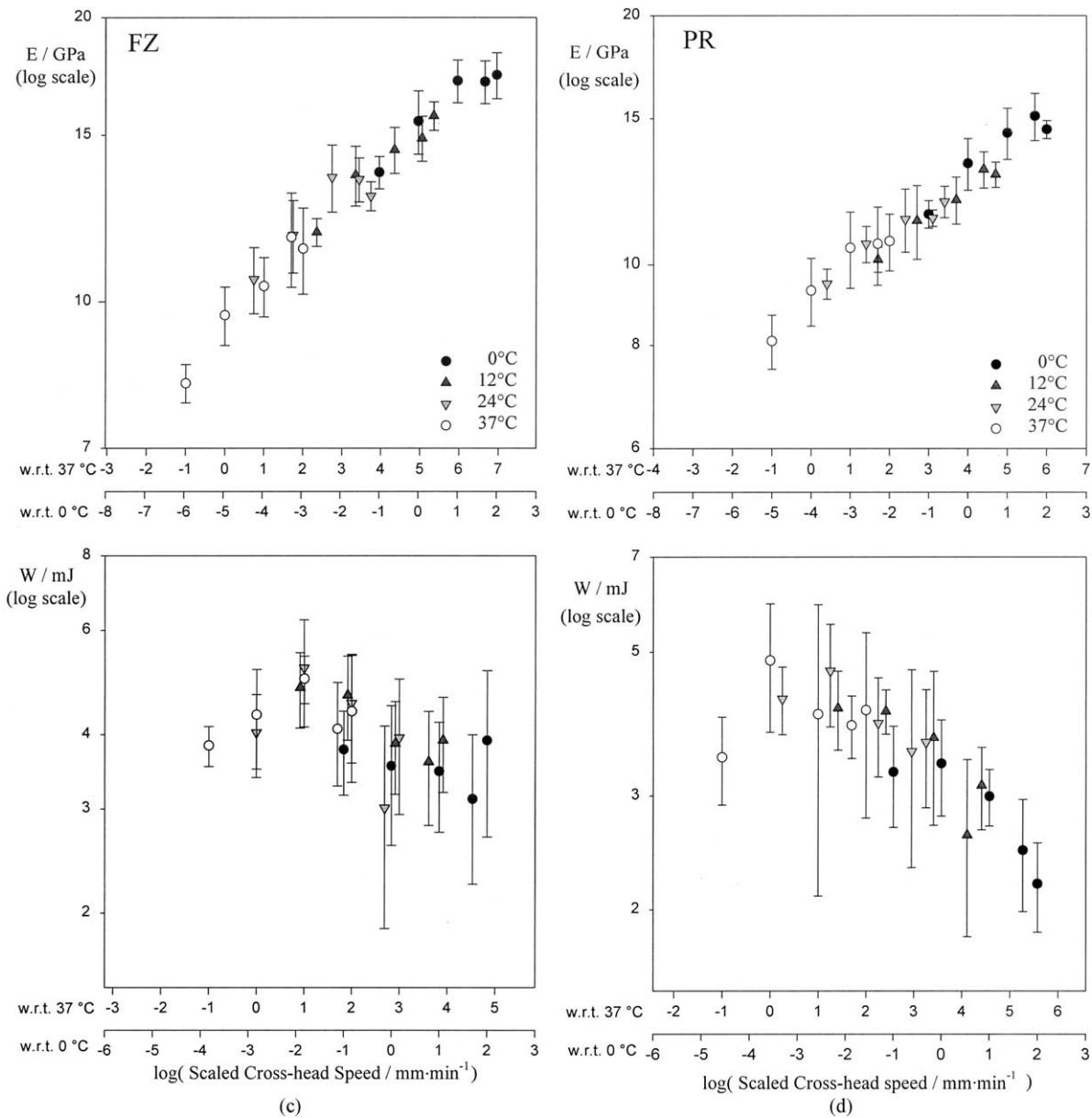


Figure 4 (continued)

values for the fitted parameters, although  $a$  and  $b_0$  values for  $W$  do not differ significantly, with  $P = 0.69$  and  $P = 0.09$ , respectively. The low value for  $a(E)$  for PR is suggestive of a substantial difference in matrix properties, a point re-emphasized by the value of  $b_1(E)$  also being the lowest, this parameter representing the strain-rate sensitivity on the material.

It is worth pointing out that our purpose in selecting the products used was by way of testing the generality of the effects over a range of types and manufacturer rather than a product comparison per se. Thus, we make no comment of the merits or otherwise on these materials.

Since  $W$  depends on both the strength and ductility of the material, i.e. degree of cross-linking

and resistance to segmental motions as well as drawing capacity, a more complicated pattern was not unexpected compared with that for  $E$ . This is borne out by the marked reversal in the curves at low XHS and high  $T$ . This suggests that under normal oral conditions, especially with the anticipated low cyclic fatigue loading, damage will accumulate and conceivably this would relate to both surface and marginal (interfacial) degradation. Fracture toughness studies may well be revealing in this light. Even so, the significance of strain rate to the interpretation of material properties in attempts to relate them to service behavior is in need of detailed examination. It may be that strain-rate sensitivities need to be documented on a routine basis, in addition to the usual set of values, i.e.  $F$  and  $E$ .

**Table 4** Two-way analysis of variance of flexural strength by temperature ( $T$ ) and cross-head speed (XHS).

	Source	df	SS	MS	F	P
HR	$T$	3	2902.866	967.622	19.766	$9.1 \times 10^{-11}$
	XHS	6	2224.010	370.668	7.572	$4.6 \times 10^{-7}$
	Residual	142	6951.492	48.954		
	Total	151	11908.127	78.862		
	(-)	$T \times XHS$	12	1533.465	127.789	3.209
	Residual	109	4340.449	39.821		
TC	$T$	3	10217.950	3405.983	34.124	$< 1 \times 10^{-13}$
	XHS	8	3888.888	486.111	4.870	$2.5 \times 10^{-5}$
	Residual	144	14372.866	99.812		
	Total	155	29345.239	189.324		
	(-)	$T \times XHS$	12	2700.283	225.024	2.349
	Residual	104	9961.293	95.782		
FZ	$T$	3	21241.041	7080.347	32.800	$< 1 \times 10^{-13}$
	XHS	4	11810.904	2952.726	13.679	$6.2 \times 10^{-9}$
	$T \times XHS$	12	3720.859	310.072	1.436	0.162
	Residual	100	21586.139	215.861		
	Total	119	60103.970	505.075		
PR	$T$	3	3622.173	1207.391	6.916	$2.8 \times 10^{-4}$
	XHS	4	6183.313	1545.828	8.854	$3.7 \times 10^{-6}$
	$T \times XHS$	12	4438.146	369.845	2.118	0.022
	Residual	99	17284.409	174.590		
	Total	118	31303.028	265.280		

Because of the unbalanced design (not all values of XHS were tested at each temperature, Table 2), interactions are not available for the full data set for HR and TC; for these materials the data for the extra XHSs were deleted and the test rerun (-). The main effects remained highly significant.

The interpretation of the parameters is of some interest. For  $E$ , the value of  $b_0$  represents the logarithm of the modulus of elasticity at  $T = 37^\circ\text{C}$  and  $XHS = 1 \text{ mm min}^{-1}$ , which may be taken as reference conditions, hence the value of  $E_{\text{ref}}$  is shown as  $10^{b_0}$  (i.e.  $\text{anti-log}_{10}$ ) (Table 3). (The standard error of a logarithmic value is a multiplier or divisor for the argument value and the upper and lower values are therefore not symmetrical. However, to indicate the magnitude of the error band, the value of  $(\text{anti-log}_{10}(\text{s.e.}) - 1) \times 100$  is shown as the percentage standard error upper limit for the  $\text{anti-log}_{10}$  of the fitted parameter.) The value of  $b_1$  is, as indicated, a measure of the strain rate sensitivity (SRS) of the material, that is, the rate of change of the value of  $\log(E)$  with XHS, at least over the range tested here. SRS is shown as calculated in similar fashion:  $(\text{anti-log}_{10}(b_1) - 1) \times 100$ , and its error correspondingly. At around 8.5-15% per decade for these products, the effects on

reported values are not negligible, especially given that the values for XHS for such tests have ranged over  $0.05\text{-}5 \text{ mm min}^{-1}$  (Table 1), that is, potentially with a value effect of up to 30%, but varying by product. It cannot be said that such a figure represents a maximum for this type of material and there is the possibility of stronger effects being encountered. Likewise, the value of  $a$  represents the temperature-sensitivity of the material in decades of cross-head speed per 10 K. The shift from a nominal 'room temperature' of  $23^\circ\text{C}$  to the nominal body temperature of  $37^\circ\text{C}$  therefore represents the equivalent of changing from  $XHS = 1\text{-}0.008 \text{ mm min}^{-1}$  for TC, for example to divide by  $\text{anti-log}_{10}(1.502 \times 14/10)$ . Equivalently, an error of 1 K in  $T$  is equivalent to an error in XHS of about  $40\% - (\text{anti-log}_{10}(1.502/10) - 1) \times 100$ . The import of this is shown by the second abscissa in the graphs of Fig. 4: depending on the reference temperature, the equivalent XHS may be very different. Similar remarks can be made in respect of  $W$ .

It is abundantly clear that temperature and cross-head speed have a profound influence on the values of mechanical properties of filled-resin restorative materials (RCs) (and by implication all materials with related matrix chemistry). Judging by the literature, this is not widely appreciated. Given also that the age and exposure of the specimen is of substantial effect,<sup>13,14</sup> to say nothing of the irradiation conditions,<sup>26</sup> the urgent need for standardization in laboratory studies<sup>13</sup> is re-emphasized; it is certainly long overdue. A further point is that properties determined at temperatures other than  $37^\circ\text{C}$  are inadequate to describe expected behavior in service, but equally, values at any one XHS are also insufficient, given that strain rates in the mouth must vary widely.

It is regrettable both that the documentation in the literature is less than complete and that test conditions have varied so much and with little reference to service conditions. Certainly, there is hardly any evidence that such matters have been considered with any seriousness. What this means is that very little comparability exists between studies, such that only trends or contrasts within a study can be used to understand material structure-property relationships, and that few definitive values can be said to exist. It is evident that similar comments must apply to the other common test modes: (axial) compression, diametral compression ('indirect tensile strength') (but see Ref. 24 for other problems with these tests), and four-point bend, as well as all biaxial flexure tests. Essentially, the basic tenet of science for work to be capable of

being reproduced is violated when conditions are inadequately specified.

Part of this discussion has been in terms of XHS, but of course the correct variable of concern is strain rate. It should be noted that the strain  $\varepsilon$  on the convex surface of a beam in flexure is (roughly):<sup>17</sup>

$$\varepsilon \approx \frac{d}{2r} \quad (5)$$

where  $d$  is the beam thickness and  $r$  the radius of curvature. Then, assuming as a first approximation that the deflected beam is close to the arc of a circle (which it is known not to be exactly),

$$r = \frac{4D^2 + L^2}{8D} \quad (6)$$

where  $D$  is the deflection and  $L$  the span, as before, and therefore

$$\varepsilon = \frac{4dD}{4D^2 + L^2} \approx \frac{4dD}{L^2} \quad (7)$$

when  $D$  is small in comparison with  $L$ . The maximum deflection observed here (HR, 37 °C, 0.01 mm min<sup>-1</sup>) was about 1.9 mm, when the non-linearity error of Eq. (7) is  $\sim +6.3\%$ . Since XHS is the rate of change of  $D$  (again, an approximation because of machine compliance issues, although it should be checked that the error is slight, as indeed it was here), strain rate is found directly. Hence, standardization of XHS is insufficient on its own: specimen thickness and span must also be standardized to achieve strain-rate comparability, or at least the factor  $4d/L^2$  used to establish the actual maximum strain rate in the specimen. That factor here is  $\sim 2 \times 10^{-3} \text{ mm}^{-1}$ , to put the present results in perspective. Given that it is a commonplace that the measured value of  $E$  depends on the test mode, it is evident that attention must be paid first to the true strain rate, and then to the strain-rate sensitivity of the material, if results of any value for cross-study comparison are to be obtained. Perhaps editors and referees need to be more alert to such deficiencies.

Although we have employed here a flexural test to determine behavior, it is to be noted that, as far as we are aware, nobody has stated or unequivocally shown that such tests have any relevance to the clinical performance of these materials. We make no such claim now, although it is conceivable that it could be argued for certain cases. The challenges in service are multifactorial: aging, wear, external chemical effects, fatigue, and so on. Clearly, in this light, the use of any test which does not allow for such factors leads to problems of interpretation and invites challenges to validity. The best that can be

said, therefore, is that some information of a comparative nature may be obtained, but equally clearly this is predicated on testing the same thing the same way. Hence, the author's suggestion that some better uniformity of approach be used.

Even so, it is argued that relevance is at least improved if temperature and immersion medium are chosen appropriately. Thus, the three-point bend test was selected to make these points because there are many reports of its use (without suggesting that this is in itself a justification in general), which illustrates the author's general cause for concern, but also because such tests as axial and diametral compression have even bigger problems.<sup>24</sup>

## Conclusions

Resin composites are very sensitive to the test conditions of cross-head speed and temperature and this needs to be taken into account both in the design of a test protocol and the interpretation of the results in the context of service conditions. In fact, the strain-rate and the temperature sensitivity could usefully be determined more routinely for many materials. These matters are in addition to the previously reported factors of preparation and storage conditions as well as irradiation for light-cured materials, which factors have marked effects on property values.

Fuller understanding of the ranges of property values can be obtained more conveniently by taking advantage of the master curve principle. That is, extremes of XHS can be avoided by an appropriate choice of temperature, and vice versa. The regularity of the behavior across the four products suggests a fundamental commonality in the structure-property relationship, indeed confirms elementary polymer science expectations.

The actual strain rate imposed by a test needs to be reported as a matter of course for materials known to be strain rate-sensitive (at least) if measured properties are to be of value. Indeed, the standard of documentation in published work is in need of considerable improvement (which falls at the door of authors and supervisors), but which in turn suggests that the standard of refereeing of papers has on occasions left something to be desired, although constructive comments are often made.

Standardization of test protocols for filled resins and similar materials is long overdue. It is supposedly self-evident that this should be the case, yet has rarely been acknowledged. A representation of oral conditions (37 °C, AS) is critical to the validity and success of this.

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## References

- Moss JB. Properties of engineering materials. London: Butterworths; 1971. p. 183 et seq.
- Ward IM. Mechanical properties of solid polymers. Chichester: Wiley; 1983. p. 18 et seq.
- Jayatilaka ADS. Fracture of engineering brittle materials. London: Applied Science Publishers; 1979.
- Palmer DS, Barco MT, Billy EJ. Temperature extremes produced orally by hot and cold liquids. *J Prosthet Dent* 1992;**67**:325–7.
- Roberts MW, Folio J, Moffa JP, Guckes AD. Clinical evaluation of composite resin systems with a dentine bonding agent for restoration of permanent posterior teeth—a three-year study. *J Prosthet Dent* 1992;**67**(3):301–6.
- Plant CG, Jones DW, Darvell BW. The heat evolved and temperature attained during setting of restorative materials. *Br Dent J* 1974;**137**(6):233–8.
- Gale MS, Darvell BW. Thermal cycling procedures for laboratory testing of dental restorations. *J Dent* 1999;**27**:89–99.
- Vijayaraghavan TV, Hsiao J. Flexural behavior of visible light-cured composites as a function of temperature under water immersion test conditions. *Dent Mater* 1994;**10**:347–52.
- Ho CT, Vijayaraghavan TV. Flexural behavior of post-cured composites at oral simulating temperatures. *J Oral Rehabil* 2001;**28**:658–67.
- Lloyd CH, Baigrei DA, Jeffrey IW. The tensile strength of composite repairs. *J Dent* 1980;**8**(2):171–7.
- ISO, International Standard 4049: Resin-based dental filling materials. Geneva: International Organization for Standardization; 1978.
- ISO, International Standard 4049: Dentistry-polymer-based filling, Restorative and Luting materials. Geneva: International Organization for Standardization; 1999.
- Musanje L, Darvell BW. Aspects of water sorption from the air, water and artificial saliva in resin composite restorative materials. *Dent Mater* 2003;**19**:414–22.
- Musanje L, Man S, Darvell BW. Water sorption and mechanical behavior of cosmetic direct restorative materials in artificial saliva. *Dent Mater* 2001;**17**:394–401.
- Darvell BW. The development of an artificial saliva for *in vitro* amalgam corrosion studies. *J Oral Rehabil* 1978;**5**:41–9.
- Heap RD, Norman RH. Flexural testing of plastics. London: The Plastics Institute; 1969.
- Timoshenko S. Strength of materials: part 1, elementary theory and problems. New York: Krieger; 1976.
- ASTM, Standard test methods for flexural properties of unreinforced and reinforced plastics and electrical insulating materials. ASTM Standard D 790-99. West Conshohocken: American Society for Testing Materials; 1999.
- Chitchumnong P, Brooks S, Stafford G. Comparison of three- and four-point flexural strength testing of denture-base polymers. *Dent Mater* 1989;**5**(1):2–5.
- Felbeck DK, Atkins AG. Strength and fracture of engineering solids, 2nd ed. New Jersey: Englewood Cliffs; 1996.
- Aklonis JJ, MacKnight WJ. *Introduction to polymeric viscoelasticity*, 2nd ed. New York: Wiley; 1983.
- Darvell BW. Materials science for dentistry. Hong Kong: B.W. Darvell; 2002.
- Darvell BW, Chan APC. Diametral tensile strength and strain rate. *Dent Mater* 1988;**4**(4):223.
- Darvell BW. Uniaxial compression tests and the validity of indirect tensile strength. *J Mater Sci* 1990;**25**:757–80.
- Williams ML, Landel RE, Ferry JD. The temperature dependence of relaxation mechanisms in amorphous polymers and other glass-forming liquids. *J Am Chem Soc* 1955;**77**:3701–7.
- Musanje L, Darvell BW. Polymerization of resin composite restorative materials: exposure reciprocity. *Dent Mater* 2003;**19**:531–41.
- Bowen RL, Rodriguez MS. Tensile strength and modulus of elasticity of tooth structure and several restorative materials. *J Am Dent Assoc* 1962;**64**:378–87.
- Forsten L, Valiaho M-L. Transverse and bond strength of restorative resins. *Acta Odont Scand* 1971;**29**:527–37.
- Reisbick M, Brodsky J. Strength parameter of composite resins. *J Prosthet Dent* 1971;**26**(2):178–85.
- Harrison A, Draughn RA. Abrasive wear, tensile strength, and hardness of dental composite resins—is there a relationship? *J Prosthet Dent* 1976;**36**(4):395–8.
- Zidan O, Asmussen E, Jorgensen KD. Tensile strength of restorative resins. *Scand J Dent Res* 1980;**88**:285–9.
- Bassiouny MA, Grant AA. Physical properties of a visible-light-cured composite resin. *J Prosthet Dent* 1980;**43**(5):536–41.
- Bausch JA, de Lange C, Davidson CL. The influence of temperature on some physical properties of dental composites. *J Oral Rehabil* 1981;**8**:309–17.
- Yazdanie N, Mahood M. Carbon fiber acrylic resin composite: an investigation of transverse strength. *J Prosthet Dent* 1985;**54**(4):543–7.
- Li Y, Swartz ML, Phillips RW, Moore BK, Roberts TA. Effect of filler content and size on properties of composites. *J Dent Res* 1985;**64**(12):1396–401.
- Söderholm K-JM. Flexure strength of repaired dental composite. *Scand J Dent Res* 1986;**94**:364–9.
- De Groot R, Peters MC, De Haan YM, Dop GJ, Plasschaert A. Failure stress criteria for composite resin. *J Dent Res* 1987;**66**(12):1748–52.
- Mathis RS, Ferracane JL. Properties of glass-ionomer/resin-composite hybrid material. *Dent Mater* 1989;**5**:355–8.
- Braem M, Finger W, Van Doren VE, Lambrechts P, Vanherle G. Mechanical properties and filler fraction of dental composites. *Dent Mater* 1989;**5**:346–8.
- von Fraunhofer JA, Curtis P. The physical and mechanical properties of anterior and posterior composite restorative materials. *Dent Mater* 1989;**5**:365–8.
- Asmussen E, Peutzfeldt A. Mechanical properties of heat treated restorative resins for use in the inlay/onlay technique. *Scand J Dent Res* 1990;**98**:564–7.
- Söderholm K-JM, Roberts MJ. Variables influencing the repair strength of dental composites. *Scand J Dent Res* 1991;**99**:173–80.
- Peutzfeldt A, Asmussen E. Influence of carboxylic anhydride on selected mechanical properties of heat-cured resin composite. *J Dent Res* 1991;**70**(12):1537–41.
- Peutzfeldt A, Asmussen E. Mechanical properties of three composite resins for the inlay/onlay technique. *J Prosthet Dent* 1991;**66**:322–4.
- Peutzfeldt A, Asmussen E. Influence of aldehydes on selected mechanical properties of resin composites. *J Dent Res* 1992;**71**(8):1522–4.

46. Peutzfeldt A, Asmussen E. Influence of ketones on selected mechanical-properties of resin composites. *J Dent Res* 1992; **71**(11):1847–50.
47. Peutzfeldt A, Asmussen E. Effect of temperature and duration of post-cure on selected mechanical properties of resin composites containing carboxylic anhydrides. *Scand J Dent Res* 1992; **100**:296–8.
48. Covey DA, Tahaney SR, Davenport JM. Mechanical properties of heat-treated composite resin restorative materials. *J Prosthet Dent* 1992; **68**:458–61.
49. Ferracane JL, Hopkin JK, Condon JR. Properties of heat-treated composites after aging in water. *Dent Mater* 1995; **11**:354–8.
50. Labella R, Braden M, Clarke RL, Davy KW. THFMA in dental monomer systems. *Biomaterials* 1996; **17**(4):431–6.
51. Huysmans MC, van der Varst PG, Lautenschlager EP, Monaghan P. The influence of simulated clinical handling on the flexural and compressive strength of posterior composite restorative materials. *Dent Mater* 1996; **12**:116–20.
52. Miyazaki M, Oshida Y, Moore BK, Onose H. Effect of light exposure on fracture toughness and flexural strength of light-cured composites. *Dent Mater* 1996; **12**:328–32.
53. Beatty MW, Swartz ML, Moore BK, Phillips RW, Roberts TA. Effect of microfiller fraction and silane treatment on resin composite properties. *J Biomed Mater Res* 1998; **40**:12–23.
54. Lavis JF, Peters TC, Makinson OF, Mount GJ. Changes to Dyract restorative resin immersed in various media. *Am J Dent* 1997; **10**(3):133–6.
55. Ferracane JL, Berge HX, Condon JR. In vitro aging of dental composites in water—effect of degree of conversion, filler volume, and filler/matrix coupling. *J Biomed Mater Res* 1998; **42**(3):465–72.
56. Cattani-Lorente MA, Dupuis V, Moya F, Payan J, Meyer J-M. Comparative study of the physical properties of a polyacid-modified composite resin and a resin-modified glass ionomer cement. *Dent Mater* 1999; **15**:21–32.
57. Brosh T, Ganor Y, Belov I, Pilo R. Analysis of strength properties of light-cured resin composites. *Dent Mater* 1999; **15**:174–9.
58. Burgess JO, DeGoes M, Walker R, Ripp AH. An evaluation of four light-curing units comparing soft and hard curing. *Pract Periodont Aesthet Dent* 1999; **11**:125–32.
59. Combe EC, Shaglouf A-MS, Watts DC, Wilson NHF. Mechanical properties of direct core build-up materials. *Dent Mater* 1999; **15**(3):158–65.
60. Hasegawa T, Itoh K, Koike T, Yukitani W, Hisamitsu H, Wakumoto S, Fujishima A. Effect of mechanical properties of resin composites on the efficacy of the dentin bonding system. *Oper Dent* 1999; **24**(6):323–30.
61. Behr M, Rosentritt M, Lang R, Handle G. Flexural properties of fiber reinforced composite using a vacuum/pressure or manual adaptation manufacturing process. *J Dent* 2000; **28**:509–14.
62. Hofmann N, Hugo B, Schubert K, Klaiber B. Comparison between a plasma arc light source and conventional halogen curing units regarding flexural strength, modulus, and hardness of photoactivated resin composites. *Clin Oral Investig* 2000; **4**:140–7.
63. Jandt KD, Mills RW, Blackwell GB, Ashworth SH. Depth of cure and compressive strength of dental composites cured with blue light emitting diodes (LEDs). *Dent Mater* 2000; **16**(1):41–7.
64. Kelsey WP, Latta MA, Shaddy RS, Stanislav CM. Physical properties of three packable resin-composite restorative materials. *Oper Dent* 2000; **25**:331–5.
65. Manhart J, Kunzelmann K-H, Chen HY, Hickel R. Mechanical properties and wear behaviour of light-cured packable composite resins. *Dent Mater* 2000; **16**:33–40.
66. Stahl F, Ashworth SH, Jandt KD, Mills RW. Light-emitting diode (LED) polymerisation of dental composites: flexural properties and polymerisation potential. *Biomaterials* 2000; **21**(13):1379–85.
67. Tilbrook DA, Clarke RL, Howle NE, Braden M. Photocurable epoxy-polyol matrices for use in dental composites I. *Biomaterials* 2000; **21**(17):1743–53.
68. Xu HHK, Eichmiller FC, Antonucci JM, Schumacher GE, Ives LK. Dental resin composites containing ceramic whiskers and precured glass ionomer particles. *Dent Mater* 2000; **16**(5):356–63.
69. Xu HHK, Smith DT, Schumacher GE, Eichmiller FC. Whisker-reinforced dental core buildup composites: effect of filler level on mechanical properties. *J Biomed Mater Res* 2000; **52**(4):812–8.
70. Bae JM, Kim KN, Hattori M, Hasegawa K, Yoshinari M, Kawada E, Yutaka O. The flexural properties of fiber-reinforced composite with light-polymerized polymer matrix. *Int J Prosthodont* 2001; **14**:33–9.
71. Domingo C, Arcis RW, Lopez-Macipe A, Osorio R, Rodriguez-Clemente R, Murtra J, Fanovich MA, Toledano M. Dental composite reinforced with hydroxyapatite: mechanical behavior and absorption/elution characteristics. *J Biomed Mater Res* 2001; **56**:297–305.
72. Kawano F, Ohguri T, Ichikawa T, Matsumoto N. Influence of thermal cycles in water on flexural strength of laboratory-processed composite resin. *J Oral Rehabil* 2001; **28**:703–7.
73. Kim KH, Ong JL, Okuno O. The effect of filler loading and morphology on the mechanical properties of contemporary composites. *J Prosthet Dent* 2002; **87**(6):642–9.
74. Sabbagh J, Vreven J, Leloup G. Dynamic and static moduli of elasticity of resin-based materials. *Dent Mater* 2002; **18**:64–71.
75. Arcis RW, Lopez-Macipe A, Toledano M, Osorio E, Rodriguez-Clemente R, Murtra J, Fanovich MA, Pascual CD. Mechanical properties of visible light-cured resins reinforced with hydroxyapatite for dental restoration. *Dent Mater* 2002; **18**(1):49–57.
76. Ellakwa AE, Shortall AC, Marquis PM. Influence of fiber type and wetting agent on the flexural properties of an indirect fiber reinforced composite. *J Prosthet Dent* 2002; **88**(5):485–90.
77. Gegauff AG, Garcia JL, Koelling KW, Seghi RR. Thermoplastic composites for veneering posterior teeth—a feasibility study. *Dent Mater* 2002; **18**(6):479–85.
78. Jandt KD, Al-Jasser AM, Al-Ateeq K, Vowles RW, Allen GC. Mechanical properties and radiopacity of experimental glass-silica-metal hybrid composites. *Dent Mater* 2002; **18**(6):429–35.
79. Munksgaard EC. Changes in expansion and mechanical strength during water storage of a traditional and three modified resin composites. *Acta Odont Scand* 2002; **60**(4):203–7.
80. Ruddell DE, Maloney MM, Thompson JY. Effect of novel filler particles on the mechanical and wear properties of dental composites. *Dent Mater* 2002; **18**(1):72–80.
81. Yap AU, Chandra SP, Chung SM, Lim CT. Changes in flexural properties of composite restoratives after aging in water. *Oper Dent* 2002; **27**(5):468–74.
82. Drummond JL, Bapna MS. Static and cyclic loading of fiber-reinforced dental resin. *Dent Mater* 2003; **19**:226–31.