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Determination of Young's modulus of dental composites: a phenomenological model

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The Young's moduli of isotropic dental restorative composites are determined with a non-destructive dynamic method, which is based on the measurement of the duration of the fundamental period for the first harmonic of a freely oscillating sample. Statistical analysis of these results yields a phenomenological model in which Young's modulus is given by an exponential rule of mixtures of the matrix phase and the filler phase of the composites. It is found that this phenomenological rule is substantiated empirically.

1. Introduction

Dental composites consist of three phases: a matrix-phase, a filler phase, and a coupling phase. A blend of organic resins constitutes the matrix phase. Bis–GMA (2,2-Bis[4-(2′-hydroxy-3′-methacryloxy-propoxy)-phenyl]-propane) or urethane dimethacrylate are the most frequently used monomers. The filler phase is characterized by a wide variety of filler origin (quartz, pyrolitic silica, silicate glasses, or organic), shape (irregular, spherical), size (0.04 to 200 μm) and size distribution [1]. Usually this phase is composed of a well-engineered combination of these parameters. The coupling phase provides a chemical and physical link between the matrix and the filler phase.

Besides its composition, many other factors affect the clinical performance of a composite restoration, such as the method of manufacturing, the storage of the material, the handling by the dentist and the chemical and mechanical conditions during functioning [2].

In order to gain insight into the internal structure and bulk coherence of these composites, the Young's modulus, which gives the relation between elastic deformation and external load, is studied *in vitro*.

The present paper gives the results of a systematic study of the Young's modulus of dental restorative composites. These results were obtained by means of a dynamic non-destructive method based on the principles of oscillation. The dependence of Young's modulus with respect to the volumetric filler content is also discussed.

2. Experimental methods

2.1. Materials and sample preparation

Two types of composites were investigated: the self-cured composites (SCC), which polymerize by mixing a base and catalyst paste and the light-cured type (LCC), which polymerizes after irradiation with 400 to 500 nm visible light. This study covers both commercial and experimental materials. In total, 55 composites were investigated. In order to simulate a 100% and a 0% filled composite, respectively amorphous silica and the pure resin (unfilled matrix phase) were tested (Table I).

For each product ten rectangular samples (L = 35 mm; w = 5 mm and h = 1.5 mm) were polymerized in a dismountable brass mould at room temperature. The mixing and placing of the SCC was performed within 3 min. The mould was then covered with a glass plate and held under firm finger pressure for 5 min. The samples were released from the mould 10 min after mixing. The LCC were inserted in the presence of minimal environmental light. A device with four light tips was placed on the glass plate whereupon the composite was exposed for 60 sec and subsequently for an additional 60 sec on the bottom. All samples were finished on dry 600 grit abrasive paper and stored for 24 h at room temperature before testing.

2.2. The fundamental period test procedure

All measurements were carried out at room temperature. The samples were set in transverse vibration in order to determine the fundamental period of the first harmonic of the freely oscillating specimen. It is easier to excite transverse than longitudinal vibration in thin specimens. In this way transverse standing waves were created in the sample with nodal points situated at about 0.224 of the length of the sample from each end inwards [3, 4]. The sample rested on triangular supports at these nodal points.

This system was activated by a single pulse excitation

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TABLE I Products, initiation type (S, self-cured; L, light-cured), batch numbers, and manufacturers

Product	S/L	Batchnumber	Manufacturer
P-10	S	112983	3M Co, St. Paul, Minnesota, USA
P-30	L	Exp. Lot 5	
Concise	S	1994A + 1994B	
Silar	S	8601A + 8601B	
Silux	L	041183 5502 U 4Y3	
Adaptic	S	053183 3A001	Johnson & Johnson, East Windsor, New Jersey, USA
Adaptic radiopaque	S	840514 CHB4135 + CHB4135/1	
Miradapt	S	3D906 24051904	
Aurafill	L	L306159	
Answer	S	201804 21300	
Certain	L	02178P 3L1604	
J & J DPC	L	6459-81-1	
Occlusin	L	Lot SP06 Mar 84	ICI plc, Macclesfield, Great Britain
VU Resin T3000	L	0061/93B	
VU Resin T4000	L	0061/91B	
DPMA/WB14 T3000	L	0061/89B	
DPMA/WB14 T4000	L	0061/87B	
UDMA 1	L	UF434	
UDMA 2	L	UF372	
Estilux posterior XR1	L	061984 034	Kulzer & Co GmbH, Bad Homburg, West Germany
Estilux posterior Y	L	061984 182	
Estic microfill	S	0684 045P + 131C	
Durafill	L	061984 139	
Clearfil Posterior New Bond	S	11127 PPU-2206 + CPU-2106	Keur & Sneltjes Dental Mfg Co, The Netherlands
Clearfil	S	43005 BFXC-0204 + CFXC-0104	
Clearfil Experimental SV	L	SV	
Nimetic	S	0014 L157	Espe Dental Products, Lynbrook, New York, USA
Nimetic-Dispers	S	L139 009 + 012	
Epolite 100	S	081131 0021231 + E081131	GC Dental Industrial Corporation, Japan
Microrest AP	S	230241	
Biogloss	S	840522	De Trey AG, Zurich, Switzerland
DTY Experimental 828	S	840828	
DTY Experimental 717	S	840717	
Brilliant	S	150584-36	Coltène AG, Altstätten, Switzerland
Brilliant Lux	L	D3 120684-20	
Command Ultrafine	L	1 841286 BS U 30344	Kerr Mfg Co, Romulus, Michigan, USA
Pedo Posterior	L	L28027/28	
Ful-fil Compules	L	041983 0224831	L.D. Caulk Co, Milford, Delaware, USA
Prisma-fil Compules	L	041983 LYG 0306841	
Finesse	S	092183/6 83/12	
Amalux	L	40536	Pierre Roland, France
Scintilux 2	L	40412	
Lumifor	L	D632	Bayer AG, Leverkussen, West Germany
BYR Experimental	L	D558 B22	
D587 B22	S	WKM 6091-3B + WKM 6098-9C	
Isomolar	S	B551183 + C701183	Vivadent, Schaan, Liechtenstein
Heliomolar	L	050384	
Isopast	S	22 B430484 + C370484	
Heliosit	L	22 1C-1D-2B 020584	
Visio-Fil	L	L214 0096	ESPE, Seefeld, West Germany
Visio-Dispers	L	L188 0035	
Dentron Nano Lux 7	L	CH 40531 U	Dentron, Diepoldsan, Switzerland
Compolux	S	N118 B30838 + C30829	Septodont, Saint-Maur, France
Compolux molaire	S	B31122 + C31174	
Compolux molaire l.v.	L	40650	

by means of a small metal hammer attracted by an electromagnet. The sample started vibrating and the first harmonic was picked up by a microphone underneath the sample, after the overtones had died out. Therefore, the experiment was conducted in an anechoic test chamber (Type 4222, Brüel and Kjaer, Denmark). Note that no part of the measuring equipment was in contact with the sample. The captured signal was fed into a special signal analyser, the Grindo-Sonic (Lemmens Elektronika, Haasrode, Belgium). This apparatus measures eight periods of the oscillation and the time of duration of two periods is displayed in μ sec (Fig. 1). From this, the fundamental frequency of the sample under flexure can be calculated (f_F). As a function of this frequency, the dynamic Young's modulus under flexure (E, in MPa) is given by Equation 1 according to the Belgian Norm for the Concrete Industry [5]:

$$E = 4 \times 10^{-6} (\pi^2 L^4 / 4.73^4 i^2) f_F \varrho C \qquad (1)$$

where i is the radius of gyration (given by $i = h^2/12$), A the cross-sectional area, ϱ the density. The correction factor C depends on the radius of gyration and Poisson's ratio (ν) and is given by:

$$C = \frac{1}{2} + \frac{4.73^2}{2} \frac{i^2}{l^2} [1 + \tfrac{6}{5} 2(1 + \nu)]$$

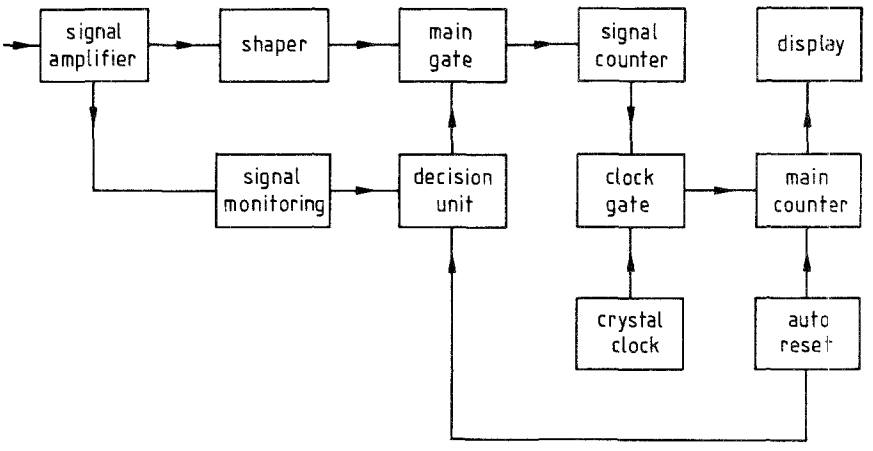

Figure 1 Scheme of the functioning of the signal analyser used.

$$+\left(\frac{1}{4}+\frac{4.73^2}{2}\frac{i^2}{l^2}[1+\tfrac{6}{5}2(1+\nu)]+\frac{4.73^4}{4}\frac{i^4}{l^4}[1-\tfrac{6}{5}2(1+\nu)]^2\right)^{1/2} \quad (2)$$

Poisson's ratio depends on the material itself but varies between 0.25 and 0.35 for dental composites, due to their composition. A constant value of 0.30 is chosen since it is found that a variation of ν by ± 0.05 resulted in changes of Young's modulus considerably less than the standard deviation. With this constant value of ν the values of the correction factor C for all 55 composites investigated, range from 1.01136729 to 1.01481367.

3. Results

The results of the measurements of the fundamental period, the calculated fundamental frequency and Young's modulus as a function of the volumetric filler fraction x, (compiled from the literature [6]) are given in Table II.

A linear regression analysis was made between the logarithm of Young's modulus and the volumetric filler fraction for the 57 data points $\{x_i, E_i\}$ (Table II) of the form:

$$y = a + bx \quad (3)$$

where

$$y = \ln E \quad (4)$$

resulting in an exponential function dependence of the calculated modulus $\boldsymbol{E}$ on x:

$$\boldsymbol{E} = \boldsymbol{E}_r \exp(bx) \quad (5)$$

where $\boldsymbol{E}_r$ is the *calculated* Young's modulus of the resin. It is found that $\boldsymbol{E}_r = 3{,}087$ MPa and $b = 2.968625$ with a correlation coefficient of $r = 0.948$ (Fig. 2). Table III gives the 95% confidence intervals (CI) of the *measured* Young's modulus E calculated with the following formula:

$$y \pm z_{\alpha/2} s_{y-y} \quad (6)$$

where

$$s_{y-y}^2 = 1 + \frac{1}{n} + \frac{(x_0 - \bar{x})^2}{(n-1)s_x^2} s_e^2 \quad (7)$$

and

$$s_e^2 = \frac{n-1}{n-2}(s_y^2 - b^2 s_x^2) \quad (8)$$

where n is the number of investigated composites, $\bar{x}$ the average filler fraction, s_x and s_y the standard deviations of the filler fraction and the logarithm of the measured Young's moduli, respectively, and s_e the unbiased estimation of the population standard deviation.

The exponential regression curve given by Equation 5 also implies that

$$b = \ln \boldsymbol{E}_s / \boldsymbol{E}_r \quad (9)$$

where $\boldsymbol{E}_s$ is the Young's modulus calculated by

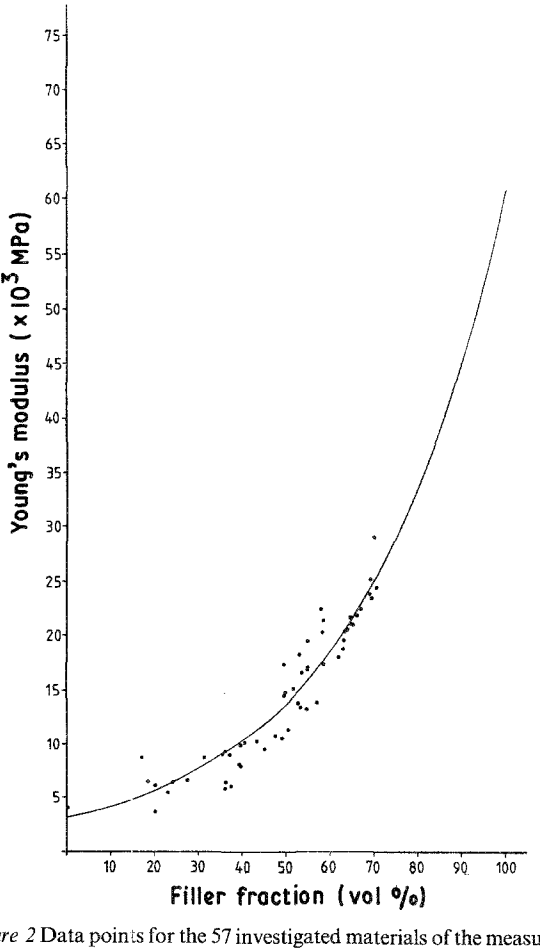

Figure 2 Data points for the 57 investigated materials of the measured Young's modulus against the volumetric filler fraction. The solid line represents the exponential regression analysis with $r = 0.948$.

TABLE II Products, volumetric filler content (VFC), fundamental period (T_F), fundamental frequency (f_F), and Young's modulus (E)

Product	VFC (%)	T_F (μsec)	f_F (Hz)	E (MPa)
Silica	100.0	142 ± 4	7064 ± 187	77145 ± 442
Clearfil Posterior SV	69.8	219 ± 2	4567 ± 42	29104 ± 237
P-10	69.1	242 ± 4	4141 ± 56	25117 ± 429
Johnson & Johnson DPC	70.6	253 ± 5	3961 ± 74	24425 ± 280
Occlusin	69.0	255 ± 2	3930 ± 28	23774 ± 225
P-30	69.6	248 ± 3	4030 ± 50	23385 ± 223
Concise	57.9	243 ± 3	4124 ± 58	22531 ± 305
DPMA/WB14 resin T3000	67.0	261 ± 2	3837 ± 24	22425 ± 247
Estilux posterior XR1	66.2	255 ± 3	3924 ± 40	21805 ± 238
Visio-Fil	64.4	256 ± 3	3911 ± 47	21723 ± 158
Adaptic	58.4	244 ± 3	4101 ± 47	21412 ± 230
Clearfil Posterior New Bond	64.8	252 ± 2	3975 ± 32	21073 ± 475
VU resin T3000	65.0	267 ± 4	3744 ± 58	21009 ± 473
DPMA/WB14 resin T4000	64.0	250 ± 4	4009 ± 68	20528 ± 201
Clearfil	58.1	254 ± 4	3952 ± 30	20373 ± 282
Miradapt	63.2	267 ± 4	3742 ± 58	20320 ± 196
Adaptic radiopaque	55.0	260 ± 2	3848 ± 31	19616 ± 365
Nimetic	63.0	264 ± 6	3825 ± 78	19550 ± 552
VU resin T4000	63.0	260 ± 2	3846 ± 31	18773 ± 272
Epolite 100	53.0	270 ± 8	3740 ± 56	18206 ± 498
Aurafill	62.0	273 ± 8	3668 ± 101	17985 ± 537
Estilux posterior	58.1	275 ± 6	3645 ± 75	17408 ± 476
De Trey 828	55.0	285 ± 5	3504 ± 64	16873 ± 276
De Trey 717	55.0	300 ± 4	3326 ± 41	16854 ± 223
Brilliant	53.9	289 ± 4	3476 ± 44	16586 ± 276
Biogloss	51.9	297 ± 6	3369 ± 64	15190 ± 385
Command Ultrafine	49.9	311 ± 4	3219 ± 42	14803 ± 168
Brilliant Lux	49.8	312 ± 5	3207 ± 52	14451 ± 176
Pedo Posterior	57.1	319 ± 5	3139 ± 43	13849 ± 571
Ful-fil	52.8	317 ± 2	3157 ± 22	13842 ± 208
Amalux	39.0	286 ± 4	3492 ± 53	13372 ± 221
Prisma-fil	53.2	325 ± 3	3075 ± 31	13362 ± 210
Lumifor	54.8	325 ± 6	3090 ± 56	13208 ± 204
Scintilux 2	50.5	305 ± 6	3283 ± 62	11360 ± 304
Visio-Dispers	47.6	328 ± 7	3047 ± 61	10786 ± 187
Heliomolar	49.1	316 ± 4	3169 ± 43	10612 ± 240
UDMA 43	43.4	321 ± 6	3115 ± 59	10340 ± 252
Nimetic-Dispers	40.5	324 ± 6	3088 ± 55	10147 ± 175
Answer	39.7	319 ± 4	3132 ± 42	9932 ± 275
Isomolar	45.3	328 ± 5	3049 ± 49	9619 ± 307
Silux	36.3	332 ± 7	3013 ± 62	9382 ± 155
Silar	35.4	332 ± 3	3011 ± 29	9075 ± 167
UDMA 37	37.2	340 ± 3	2941 ± 29	9012 ± 150
Certain	31.4	339 ± 5	2954 ± 46	8770 ± 123
Microrest AP	17.1	331 ± 4	3015 ± 36	8679 ± 250
Compolux molaire l.v.	39.5	357 ± 7	2800 ± 55	8142 ± 126
Compolux molaire	39.7	371 ± 4	2682 ± 45	7964 ± 102
Compolux	27.4	390 ± 11	2567 ± 71	6691 ± 104
Estic microfill	36.1	377 ± 6	2653 ± 44	6473 ± 58
Finesse	18.5	376 ± 3	2662 ± 21	6437 ± 157
Heliosit	24.2	378 ± 6	2648 ± 40	6401 ± 142
BYR Experimental	20.2	385 ± 7	2598 ± 44	6108 ± 94
Durafill	37.5	393 ± 6	2545 ± 39	6085 ± 88
Dentron Nano Lux 7	36.1	413 ± 9	2422 ± 48	5860 ± 242
D587B22	20.2	385 ± 7	2597 ± 50	5724 ± 113
Isopast	23.2	409 ± 15	2453 ± 88	5436 ± 268
Unfilled resin	00.0	479 ± 10	2090 ± 43	4004 ± 61

Equation 5 of the amorphous silica, and also that:

$$\ln E = x \ln E_s + (1 - x) \ln E_r \qquad (10)$$

or

$$E = (E_s)^x (E_r)^{1-x} \qquad (11)$$

This formula can be interpreted as a generalization of the Voigt model where, instead of a linear mixing of the moduli of the two phases, one has a linear mixing of the logarithms of the moduli. Accurate knowledge of the Young's moduli of the resin and the pure silica thus suffices to predict the value for any arbitrary composite in between.

4. Discussion

The above described dynamic non-destructive test has several advantages over the dynamic method where the sample is activated by a series of impulses with a frequency close to the resonance frequency or one of its harmonics as described by Spinner and Tefft [3].

One major advantage is the absolute absence of mechanical contact between the test equipment and

the sample, which automatically eliminates the uncontrollable influence of such contacts on the measurements.

Second, since the fundamental period is measured directly and the fundamental frequency is thus known immediately, there is no need to scan a whole frequency range as has to be done with the driven mechanical resonance frequency method of the quoted method [3].

Third, the strain applied to the sample is extremely low even compared with a low strain static non-destructive test. This guarantees that the response of the sample is completely within the elastic region so that highly non-linear plastic effects are avoided.

The value measured for the amorphous silica corresponds well with the literature value of about 70 000 MPa [7]. For a more detailed discussion of the results of the composite materials and their implication for dentistry, the reader is referred to Braem [6] and to Braem *et al.* [8].

As one can see from the results given in Table III, all 57 experimental data points but two (Microrest

TABLE III Measured (E) and calculated (E) Young's moduli (MPa) with 95% CI of E

Products	x	E	E	CI of E
Silica	1.000	77 145	60 096	(41 376, 87 284)
Clearfil Posterior SV	0.698	29 104	24 518	(17 233, 34 883)
P-10	0.691	25 117	24 014	(16 883, 34 156)
Johnson & Johnson DPC	0.706	24 425	25 107	(17 641, 35 733)
Occlusin	0.690	23 774	23 943	(16 834, 34 053)
P-30	0.696	23 385	24 373	(17 132, 34 674)
Concise	0.579	22 531	17 221	(12 146, 24 417)
DPMA/WB14 resin T3000	0.670	22 425	22 563	(15 874, 32 064)
Estilux posterior XR1	0.662	21 805	22 033	(15 507, 31 305)
Visio-Fil	0.644	21 723	20 887	(14 709, 29 659)
Adaptic	0.584	21 412	17 479	(12 327, 24 784)
Clearfil Posterior New Bond	0.648	21 073	21 136	(14 883, 30 017)
VU resin T3000	0.650	21 009	21 262	(14 970, 30 198)
DPMA/WB14 resin T4000	0.640	20 528	20 640	(14 537, 29 306)
Clearfil	0.581	20 373	17 324	(12 218, 24 563)
Miradapt	0.632	20 320	20 156	(14 199, 28 611)
Adaptic radiopaque	0.550	19 616	15 801	(11 149, 22 393)
Nimetic	0.630	19 550	20 036	(14 116, 28 441)
VU resin T4000	0.630	18 773	20 036	(14 116, 28 441)
Epolite 100	0.530	18 206	14 890	(10 508, 21 098)
Aurafill	0.620	17 985	19 450	(13 706, 27 601)
Estilux posterior	0.581	17 408	17 324	(12 218, 24 563)
De Trey 828	0.550	16 873	15 801	(11 149, 22 393)
De Trey 717	0.550	16 854	15 801	(11 149, 22 393)
Brilliant	0.539	16 586	15 293	(10 792, 21 671)
Biogloss	0.519	15 190	14 412	(10 172, 20 419)
Command Ultrafine	0.499	14 803	13 581	(9 586, 19 241)
Brilliant Lux	0.498	14 451	13 541	(9 558, 19 183)
Pedo Posterior	0.571	13 849	16 817	(11 863, 23 841)
Ful-fil	0.528	13 842	14 802	(10 446, 20 973)
Amalux	0.390	13 372	9 826	(6 927, 13 936)
Prisma-fil	0.532	13 362	14 979	(10 571, 21 224)
Lumifor	0.548	13 208	15 707	(11 083, 22 260)
Scintilux 2	0.505	11 360	13 825	(9 758, 19 587)
Visio-Dispers	0.476	10 786	12 684	(8 953, 17 971)
Heliomolar	0.491	10 612	13 262	(9 361, 18 789)
UDMA 43	0.434	10 340	11 198	(7 901, 15 869)
Nimetic-Dispers	0.405	10 147	10 274	(7 246, 14 566)
Answer	0.397	9 932	10 033	(7 075, 14 226)
Isomolar	0.453	9 619	11 847	(8 361, 16 787)
Silux	0.363	9 382	9 070	(6 391, 12 870)
Silar	0.354	9 075	8 830	(6 221, 12 534)
UDMA 37	0.372	9 012	9 315	(6 566, 13 216)
Certain	0.314	8 770	7 842	(5 518, 11 144)
Microrest AP	0.171	8 679	5 129	(3 584, 7 340)
Compolux molaire l.v.	0.395	8 142	9 973	(7 033, 14 143)
Compolux molaire	0.397	7 964	10 033	(7 075, 14 226)
Compolux	0.274	6 691	6 964	(4 892, 9 912)
Estic microfill	0.361	6 473	9 016	(6 353, 12 795)
Finesse	0.185	6 437	5 347	(3 739, 7 645)
Heliosit	0.242	6 401	6 333	(4 443, 9 027)
BYR Experimental	0.202	6 108	5 624	(3 937, 8 033)
Durafill	0.375	6 085	9 398	(6 625, 13 333)
Dentron Nano Lux 7	0.361	5 860	9 016	(6 353, 12 795)
D587B22	0.202	5 724	5 624	(3 937, 8 033)
Isopast	0.232	5 436	6 147	(4 311, 8 767)
Unfilled resin	0.000	4 004	3 087	(2 129, 4 477)

AP, and Durafill) are within the 95% CI as one would expect from a correlation coefficient of 0.948. A possible explanation for the misfit of the two materials could be that their volumetric filler fraction as reported in the literature is incorrect. Indeed, especially in this type of composite, which is filled with organic-resin based filler particles, the procedure for the determination of the filler content is subject to uncertainties, especially for the burning out of the matrix phase.

It should be noted that a linear regression analysis between Young's modulus and the volumetric filler fraction yields a correlation coefficient of 0.817, which is significantly smaller than the 0.948 value obtained from the exponential regression. Furthermore, according to the linear regression, the Young's modulus of the matrix phase, i.e. for $x = 0$, is equal to -8503 MPa. This means that for small concentrations of the filler phase a negative value for Young's modulus is obtained, which is physically impossible. This leads to the conclusion that linear regression has to be rejected.

The phenomenological model given by Equations 5 and 10 shows an exponential dependence of Young's modulus against the volumetric filler fraction. This model is valid for composites consisting of an organic resin matrix phase and a filler phase of size between 0.04 μm and 200 μm. Futhermore, these particles are homogeneously embedded in the matrix so that the composite can be considered to be isotropic. Finally, all particles are assumed to be linked with the matrix phase through the coupling phase. It must, however, be noted that for small filler particles, Young's modulus is less dependent on particle size than on the maximum particle packing fraction, a ratio determined by the particle shape and the size distribution [9]. This probably is why some composites have Young's moduli that do not correspond completely with the values predicted from their filler percentage.

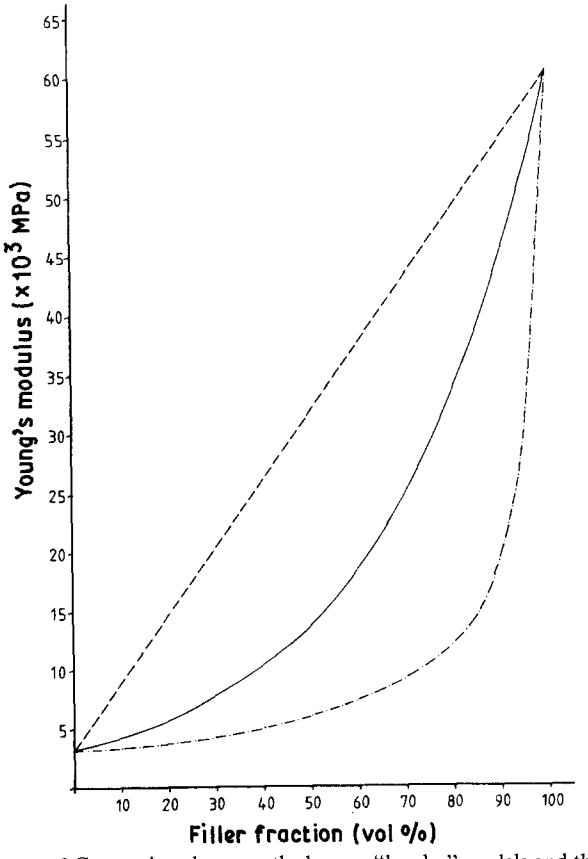

Figure 3 Comparison between the known "border" models and the phenomenological model developed from Equation 5. (– – –) Parallel model, (–·–·–) series model, (——) present study.

When the results for Young's moduli calculated on the basis of Equation 5 are compared with the results obtained from the uniform strain model of Voigt and the uniform stress model of Reuss [10] for the same limiting values of E_r and E_s, the phenomenological model is situated between them, as it should be. This is shown in Fig. 3.

It should be noted that such an exponential dependence of Young's modulus is not found in unidirectional composites where the tensile modulus in the direction of the fibres is given by the linear rule of mixtures, i.e. the Voigt model. This rule also seems to be obeyed experimentally for the unidirectional composites [11], as the exponential rule is obeyed by isotropic dental composites as shown by the results of Table III and Fig. 2.

5. Conclusions

The present paper describes a dynamic non-destructive method with which the Young's modulus of 55 isotropic dental composites is determined. For such composites, an exponential rule of mixtures is derived phenomenologically and shown to be satisfied by the majority of the investigated composites within acceptable statistical limits. This model can, therefore, be used for predictive purposes.

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