Determination of the strength of dental phosphate-bonded investments by 2 and 3 parameter Weibull analysis

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Objective: Phosphate-bonded investments are being investigated for use as die materials for dental superplastic forming. The effects of handling technique on the strengths of these investments needs to be determined. The purpose was to use methods of Weibull analysis to fit measured 4 point bend strength data and determine strength and Weibull modulus, where modulus represents the scatter of the data. Materials and methods: Weibull parameters were determined using methods of regression and maximum likelihood using 2 and 3 parameters. The parameters were x_0 the lower bound of strength, m the Weibull Modulus, and θ the characteristic strength. x_0 is zero for the 2 parameter models. In addition to plots of the measured data and curves generated using the Weibull parameters, the fit of the models to the measured data was determined using correlation coefficient and the χ^2 statistic. Results: The results showed that characteristic strength was similar whether determined using regression or maximum likelihood, 2 parameter or 3 parameter models. However, Weibull Modulus was significantly lower for 3 parameter models than for 2 parameter models. Visually the 3 parameter models give the best fit for the majority the data sets. Correlation coefficient, r, showed whether the method of maximum likelihood or the method of regression was more appropriate. χ^2 and correlation coefficient did not necessarily give the same indication of goodness-of-fit of these data sets. Significance: Strengths of dental phosphate-bonded investment materials can be described well using weibull analysis which gives characteristic strengths and quantitative measures of scatter of the data in the form of Weibull Modulus. These may then be used to select a handling technique to produce dies for dental superplastic forming.

1. Introduction

Dental phosphate-bonded investment (PBI) materials are currently being used to manufacture dental prostheses by superplastic forming [1]. For successful superplastic forming of dental components, the strength of the PBI material is crucial if the fit of the formed prosthesis is to be accurate. Dies must, of course, be resistant to fracture but they must also be durable throughout the forming time, which is between 20 minutes and 3 hours. Such durability requires additional resistance to spallation.

The strength of dental investments is dependent on a number of factors that include the chemistry and volume fraction of refractory and binder, the ratio of investment powder to liquid, the use of proprietary mixing liquid and the handling technique [2]. Handling technique has been reported to account for the incidence of pores during investment preparation [3–5] but only recently has the effect of pores on strength been quantified [2]. The variables that affect pore size during handling include the method of mixing (by hand or using a mechanical spatula), and the use of reduced pressure and increased pressure for mixing and setting. In the same study entrapped air produced porosity that was measured for four materials using 6 different handling techniques. The porosity was shown to affect the cold strength of all PBI's but some were more markedly affected than others.

The strengths of the investments measured by Juszczyk and Curtis (6) were described using the three parameter Weibull distribution. The Weibull distribution is reported (7) to be a more accurate representation

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of strength for ceramics than the normal distribution and is often considered to be the most appropriate distribution to represent the weakest link model which is used to describe the influence of largest pore size on fracture when the stress intensity around a flaw is greater than the critical value (K_{IC}) of the material.

The three parameters determined using the Weibull distribution are the Characteristic strength (θ), the Weibull modulus m, and a scaling parameter x_0. The scaling parameter is set to zero if it is shown that the two parameters are sufficient to describe the data accurately. This is the case when the number of samples in the study is insufficient to describe the full shape of the distribution. There are various methods that have been adopted to determine the three parameters (8). The regression method and maximum likelihood method have been compared for other data sets (9) but not for strengths of PBI's.

The aim of this study was to use the 2 and 3 parameter Weibull distributions to describe the 4-point bend strengths of 4 investment materials and 6 handling techniques using the methods of regression and maximum likelihood and to determine which approach gave the best fit of strength for the majority of groups.

2. Method and materials

Four investment materials, Croform WB (Davis, Schottlander & Davis Ltd, Letchworth, UK), Rema Exact (Dentaurum, Phorzheim, Germany), Levotherm (Bayer Dental, Leverkusen, Germany) and Rematitan (Dentaurum, Phorzheim, Germany), were selected for investigation. The materials were mixed at the liquid to investment powder ratio recommended by the manufacturer using handling and setting conditions shown in Table I and described fully elsewhere (3).

50 specimens of dimensions $100 \times 15 \times 15$ mm^3 were prepared for each material and handling technique using polyvinylsiloxane duplicating material (Elite Double, Zhermack, Rovigo, Italy) for the mould. Specimens were left to set for one hour from the beginning of mixing and for a further 2 hours on removal from the mould prior to testing. Four-point bend tests at a crosshead speed of 1 mm min^{-1} were carried out at 20°C ± 2°C using an Instron 1193 testing machine (Instron Ltd, High Wycombe, UK). The strength at fracture of all test specimens was recorded.

2.1. Determining the Weibull parameters by the regression method

Values of strength for each group were ranked in increasing order. Median ranks were assigned to these values using the approximation:

$$\text{Median\_rank} = \left(\frac{i - 0.3}{n + 0.4}\right) \quad (1)$$

where i = failure order number, n = sample size

The data is plotted on scales equivalent to Weibull probability paper with strength on the abscissa and median ranks on the ordinate:

$$Y = \ln \ln \left(\frac{1}{1 - \text{Median\_rank}}\right) \quad (2)$$

$$X = \ln(\text{strength} - x_0) \quad (3)$$

The least squares method is used to fit a line through points to find the Weibull parameters. Corrections are made to this curve by incrementing the value of x_0 between zero and the lowest strength. The x_0 is subtracted from the original data set and plotted against median ranks as before. In order to find x_0 that corresponds to the best fit, the least squares method is used to fit the data for all values of x_0 and the correlation coefficient r, plotted to determine maximum r (Fig. 1). The Weibull parameters m and θ may be obtained from each of these curves using Equations 4–6.

$$m = \left[\frac{n \cdot \sum(X \cdot Y) - \sum X \sum Y}{n \cdot \sum X^2 - \left(\sum X\right)^2}\right] \quad (4)$$

$$\text{intercept} = \frac{\sum Y - m \sum X}{n} \quad (5)$$

$$\theta = \exp\left(-\frac{\text{intercept}}{m}\right) + x_0 \quad (6)$$

To obtain the coefficients for the 2 parameter model, $x_0 = 0$.

To obtain a further estimate of fit, the goodness of fit parameter χ^2, is determined for each data set by assigning all data for one sample into four bins. A histogram

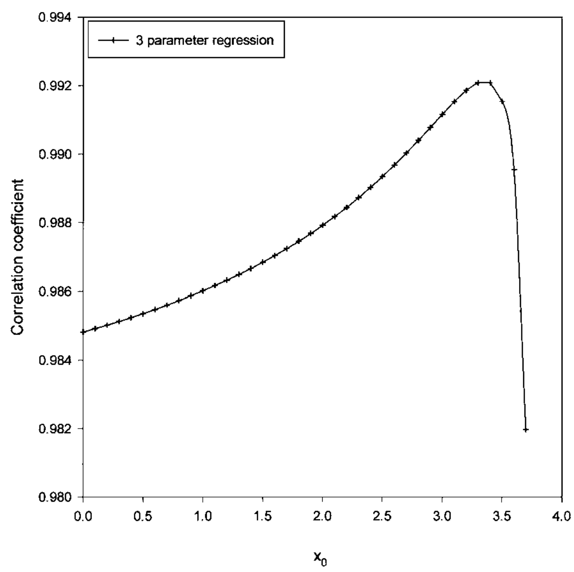


Figure 1 Variation of correlation coefficient with lower bound of strength x_0 using regression to fit the 3 parameter Weibull distribution to 50 measurements of strength of a dental casting investment.

TABLE I Description of conditions for handling investment materials for the measurement of 4-point bend strengths

Handling technique		Setting
ha	Hand spatulation	In Air
hp	Hand spatulation	Under pressure
hva	Hand spatulation in vacuum	In air
ma	Mechanical spatulation in air	In air
mvp	Mechanical spatulation in vacuum	Under pressure
mva	Mechanical spatulation in vacuum	In air

is plotted of measured frequency verses strength and a similar histogram may be plotted of estimated frequency verses strength using the estimated parameters in theWeibull density function:

$$f(x) = \left[\frac{m}{\theta - x_0}\left(\frac{x - x_0}{\theta - x_0}\right)^{m-1}\right] \times \left\{\exp\left[-\left(\frac{x - x_0}{\theta - x_0}\right)^{m}\right]\right\} \quad (7)$$

χ^2 is determined by comparing the measured frequencies and the estimated frequencies using Equation 8:

$$\chi^2 = \sum \frac{(\text{observed\_frequencies} - \text{estimated\_frequencies})^2}{\text{estimated\_frequencies}} \quad (8)$$

2.2. Determining the Weibull parameters by the maximum likelihood method

It is shown by Trustrum and Jayatilaka (8) that to obtain the equation for maximum likelihood for the 2 parameter model:

$$P_f = 1 - \exp(-b\sigma^m) \quad (9)$$

$$\text{Where} \quad b = \left\{\Gamma\frac{1 + \left(\frac{1}{m}\right)}{\sigma}\right\}^m \quad (10)$$

The probability density function

$$f(\sigma) = \frac{dP_f}{d\sigma} = bm\sigma^{(m-1)}\exp(-b\sigma^m) \quad (11)$$

from which it is shown that

$$\ln L = n \ln m + n \ln b + (m-1)\sum \ln\sigma_i - b\sum\sigma_i^m \quad (12)$$

If $\ln L$ is differentiated with respect to m and b and the partial derivates are equated to zero, the maximum likelihood estimates, m_L and b_L, are obtained which satisfy

$$\frac{n}{m_l} + \sum\ln\sigma_i - b_L\sum\sigma_i^{m_L}\ln\sigma_i = 0 \quad (13)$$

$$\frac{n}{b_L} - \sum\sigma_i^{m_L} = 0 \quad (14)$$

In Equations 13 and 14 b_L is eliminated giving the following equation for m_L, the Weibull Modulus.

$$\frac{n}{m_L} - n\frac{\sum\sigma_i^{m_L}\ln\sigma_i}{\sum\sigma_i^{m_L}} + \sum\ln\sigma_i = 0 \quad (15)$$

The root function (Equation 16) in a PC program, Mathcad (v8.03, Mathsoft Inc., Cambridge, MA 02142, USA), was used to solve Equation 15 giving a unique m_L for any specific value of x_0.

$$m(x0) = \text{root}\left[\left(\frac{n}{m_L} - n\frac{\sum\sigma(x0)_i^{m_L}\ln\sigma(x0)_i}{\sum\sigma(x0)_i^{m_L}} + \sum\ln\sigma(x0)_i\right), m\right] \quad (16)$$

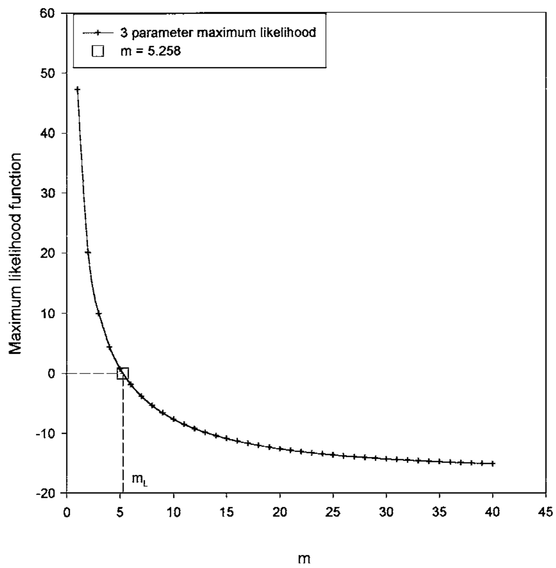


Figure 2 Plot used to determine Weibull Modulus for a data set using the method of maximum likelihood. The Weibull Modulus of this data set $m = 5.258$ which corresponds to maximum likelihood function = 0.

A plot of $f(m)$ (Equation 17) verses m shows the value of m_L (for $f(m) = 0$) that satifies Equation 15 (Fig. 2).

$$f(m) = \frac{n}{m} - n\frac{\sum\sigma_i^m\ln\sigma_i}{\sum\sigma_i^m} + \sum\ln\sigma_i \quad (17)$$

An $x_0 = 0$ gives the Weibull Modulus for the 2 parameter model. To determine the best fit Weibull Modulus for the 3 parameter model x_0 was varied between $0 < x_0 <$ minimum strength. Thus b was calculated from Equation 10 and the $\ln L$ function maximised using Equation 12 (Fig. 3).

The characteristic strength was determined from Equation 18.

$$\theta = x0 + \frac{\bar{\sigma}}{(b\bar{\sigma}^{m_L})^{\frac{1}{m_L}}} \quad (18)$$

The χ^2 statistic was determined for all data sets using the values of the parameters obtained using the maximum likelihood method and using Equations 7 and 8.

3. Results and discussion

3.1. Statistical analysis

Tables II and III show the coefficients obtained for the 2 parameter and 3 parameter models using the maximum likelihood method and the regression method and the degree of fit compared to the measured data using the correlation coefficient and χ^2 statistic.

TABLE II Coefficients for the 2 parameter models

	Maximum likelihood					Regression				
	x_0	θ	m	r	χ^2	x_0	θ	m	r	χ^2
cf_ha	0	4.763	14.787	0.997	2.924	0	4.76	14.74	0.997	2.828
cf_hp	0	5.478	10.998	0.994	4.215	0	5.462	11.648	0.994	3.999
cf_hva	0	5.843	14.04	0.982	1.848	0	5.818	15.756	0.985	0.698
cf_ma	0	4.942	13.099	0.982	2.767	0	4.912	15.557	0.985	3.077
cf_mvp	0	8.082	15.563	0.99	3.816	0	8.122	13.127	0.986	2.242
cf_mva	0	7.1	13.241	0.997	0.375	0	7.095	13.175	0.997	0.374
	x_0	θ	m	r	χ^2	x_0	θ	m	r	χ^2
le_ha	0	6.157	6.824	0.996	2.663	0	6.146	6.998	0.996	2.905
le_hp	0	6.62	9.351	0.982	7.306	0	6.58	10.684	0.98	8.002
le_hva	0	6.232	9.558	0.989	10.672	0	6.201	10.598	0.988	13.789
le_ma	0	5.311	9.39	0.986	1.416	0	5.278	10.721	0.989	1.542
le_mvp	0	7.154	12.953	0.996	0.845	0	7.158	12.521	0.996	0.644
le_mva	0	6.567	10.411	0.989	2.659	0	6.63	8.378	0.982	2.053
	x_0	θ	m	r	χ^2	x_0	θ	m	r	χ^2
re_ha	0	3.16	8.119	0.994	0.473	0	3.162	7.92	0.994	0.342
re_hp	0	3.648	11.27	0.996	0.996	0	3.667	9.757	0.997	0.661
re_hva	0	3.541	10.821	0.995	0.522	0	3.537	10.888	0.995	0.445
re_ma	0	3.358	10.91	0.992	9.413	0	3.358	10.698	0.992	8.863
re_mvp	0	4.128	13.475	0.99	9.322	0	4.136	12.521	0.991	8.213
re_mva	0	3.611	9.321	0.992	0.328	0	3.601	9.744	0.994	0.164
	x_0	θ	m	r	χ^2	x_0	θ	m	r	χ^2
rt_ha	0	3.747	8.625	0.981	5.059	0	3.721	9.954	0.98	6.621
rt_hp	0	4.393	7.172	0.996	0.793	0	4.377	7.562	0.997	1.063
rt_hva	0	3.975	6.804	0.983	1.594	0	3.945	7.818	0.989	0.72
rt_ma	0	4.421	8.94	0.976	5.747	0	4.387	10.59	0.978	4.983
rt_mvp	0	4.964	7.828	0.988	1.117	0	4.929	8.959	0.989	2.82
rt_mva	0	4.931	8.9	0.992	4.192	0	4.909	9.725	0.992	4.826

TABLE III Coefficients for the 3 parameter models

	Maximum likelihood					Regression				
	x_0	θ	m	r	χ^2	x_0	θ	m	r	χ^2
cf_ha	2.97	4.737	5.258	0.996	3.112	3.353	4.737	3.569	0.996	2.344
cf_hp	3.611	5.413	3.467	0.997	1.718	3.349	5.432	3.862	0.997	2.218
cf_hva	4.324	5.778	3.407	0.992	0.62	4.001	5.792	4.267	0.991	0.589
cf_ma	3.94	4.855	2.407	0.994	0.624	3.833	4.868	2.716	0.994	0.576
cf_mvp	−73.313	8.104	160.095	0.992	2.801	−57000	8.125	102800	0.99	1.93
cf_mva	3.072	7.077	7.346	0.998	0.186	0.796	7.094	11.567	0.997	0.356
	x_0	θ	m	r	χ^2	x_0	θ	m	r	χ^2
le_ha	3.436	6.04	2.702	0.993	0.915	3.617	6.054	2.144	0.99	0.264
le_hp	5.032	6.438	1.901	0.992	2.748	4.959	6.457	1.965	0.993	2.614
le_hva	4.634	6.082	2.071	0.995	5.91	4.577	6.099	2.05	0.995	4.666
le_ma	3.633	5.213	2.737	0.994	0.13	3.389	5.231	3.254	0.994	0.12
le_mvp	2.098	7.141	9.032	0.996	0.932	0.842	7.156	10.194	0.996	0.625
le_mva	−87.858	6.606	154.844	0.992	1.332	−13100	6.631	19710	0.99	1.027
	x_0	θ	m	r	χ^2	x_0	θ	m	r	χ^2
re_ha	0.752	3.149	6.106	0.994	0.786	−1.185	3.165	11.382	0.993	0.372
re_hp	−51.251	3.666	175.86	0.996	0.962	−3267	3.671	9986	0.996	0.774
re_hva	1.764	3.519	5.208	0.994	0.108	1.709	3.526	5.081	0.995	0.42
re_ma	1.888	3.333	4.462	0.992	9.464	2.235	3.33	2.807	0.992	7.037
re_mvp	−5.273	4.137	31.264	0.991	9.286	0.953	4.134	9.368	0.991	8.095
re_mva	1.896	3.577	4.257	0.993	0.131	1.282	3.594	5.869	0.993	0.142
	x_0	θ	m	r	χ^2	x_0	θ	m	r	χ^2
rt_ha	2.679	3.653	2.176	0.992	0.38	2.607	3.665	2.312	0.992	0.493
rt_hp	2.175	4.333	3.419	0.996	0.998	1.89	4.353	3.796	0.996	0.694
rt_hva	2.174	3.898	2.967	0.983	1.809	1.804	3.921	3.738	0.985	1.313
rt_ma	3.145	4.319	2.364	0.993	0.977	2.977	4.336	2.824	0.991	1.378
rt_mvp	3.172	4.859	2.606	0.996	0.325	2.952	4.879	2.968	0.995	0.245
rt_mva	3.531	4.814	2.137	0.996	2.738	3.504	4.828	2.007	0.996	2.502

The accuracy with which these models agree with the measured data is assessed using three methods: (1) visual plotting (2) determination of the correlation coefficient (3) determination of the χ^2 for goodness-of-fit.

3.1.1. Plotting the data and interpreting goodness-of-fit visually

Figs 4–9 show the measured data and best-fit curves for all handling techniques and the four investment materials. Excellent fit is observed visually for the 3 parameter

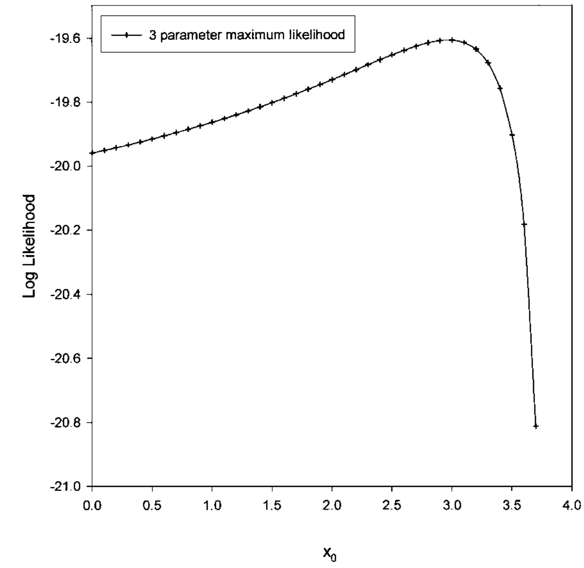
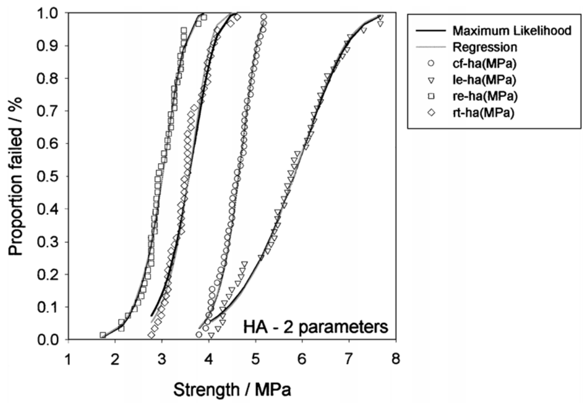
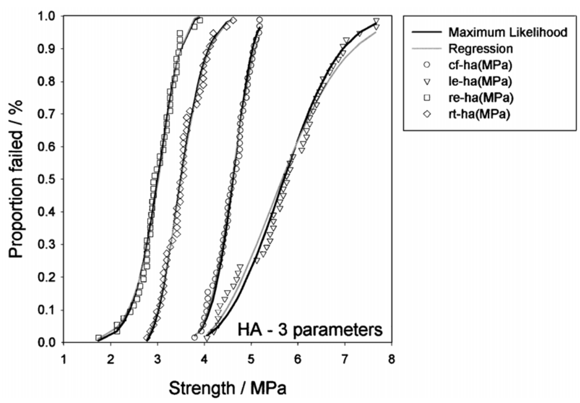


Figure 3 The variation of the log likelihood function with lower bound of strength x_0. Such a plot is used to determine the lower bound of strength x_0 where the log likelihood function is maximised.



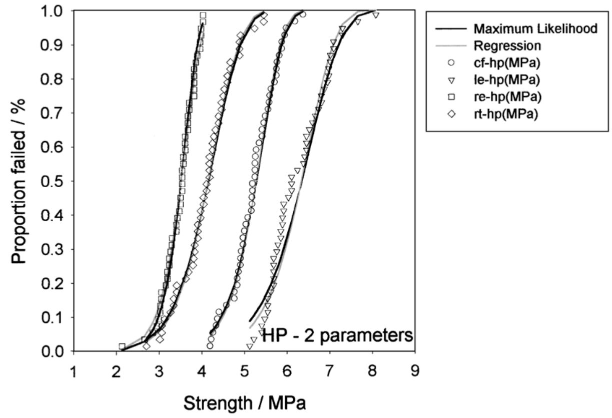
(a)



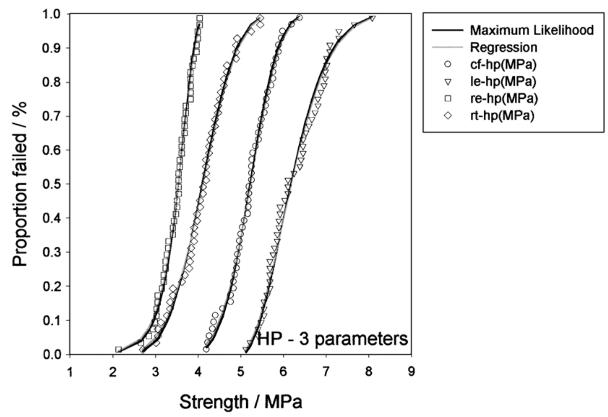
(b)

Figure 4 Plot of measured strength for all materials using the HA handling technique and curves generated from the measured data points using (a) 2 Weibull parameters (b) 3 Weibull parameters. The parameters of the distribution function were determined using methods of regression and maximum likelihood.

Weibull model determined by maximum likelihood and regression in most cases and clearly indicates that visually the 3 parameter distributions are the more appropriate models to describe the data.



(a)



(b)

Figure 5 Plot of measured strength for all materials using the HP handling technique and curves generated from the measured data points using (a) 2 Weibull parameters (b) 3 Weibull parameters. The parameters of the distribution function were determined using methods of regression and maximum likelihood.

Examples of data sets that fit poorly by visual inspection are indicated in Table IV and are mainly for the 2 parameter models. Poor fit may mean that the models fail to pass through the data points for the lowest strengths and include the data of le-ha and rt-mva. Poor fit is also exhibited for rt-ha, le-hp and others for which the models neither fit the lowest strengths nor the mid-range strengths. There are still others such as rt-hva that are not fit well at the higher strengths.

For the 3 parameter models, x_0 is determined in addition to characteristic strength and Weibull modulus. In most cases 3 parameters should result in significantly better fit than for 2 parameters and this is clearly observed in most cases. In addition, x_0 may be interpreted to have a physical significance and represents a lower bound of strength. However, if x_0 is determined to be negative, shown by the symbol $-x_0$ in Table IV for data sets cf-mvp, le-mva, re-hp and re-mvp, this sometimes indicates a shelf-life for the material, specimen or component. Since the majority of data sets do not display this same characteristic then in these cases a negative x_0 is taken to be an anomaly and may fit the data only partially better than the 2 parameter models as for cf-mvp and re-mvp. Of course, despite a negative x_0, the models may still fit the data well as shown for cf-mvp (ml), le-mva (ml) and re-mvp (ml).

TABLE IV Summary of fit of data sets (1) visual inspection—poor fit is indicated by a tick, (2) correlation coefficient <0.9855—poor fit is indicated by an r (3) $\chi^2 < 3.841$ (3 parameter) and $\chi^2 < 5.991$ (2 parameter)—poor fit is indicated by χ^2. The symbol $-x_0$ represents values of $x_0 < 0$. The symbol $\otimes$ represents the most appropriate model to fit the data

		2 parameter models		3 parameter models	
Material	Handling technique	Maximum likelihood	Regression	Maximum likelihood	Regression
Croform (cf)	ha	$\otimes$	$\otimes$		
Croform (cf)	hp			$\otimes$	$\otimes$
Croform (cf)	hva	$\surd r$	$\surd r$	$\otimes$	
Croform (cf)	ma	$\surd r$	$\surd r$	$\otimes$	$\otimes$
Croform (cf)	mvp		r	$\otimes - x_0$	$-x_0$
Croform (cf)	mva			$\otimes$	
Levotherm (le)	ha	$\otimes\surd$	$\surd$	$\surd$	$\surd$
Levotherm (le)	hp	$\surd r\chi^2$	$\surd r\chi^2$		$\otimes$
Levotherm (le)	hva	$\surd\chi^2$	$\surd\chi^2$	$\otimes\chi^2$	
Levotherm (le)	ma	$\surd r$	$\surd$	$\otimes$	$\otimes$
Levotherm (le)	mvp	$\otimes$	$\otimes$	$\otimes$	$\otimes$
Levotherm (le)	mva	$\surd$	$\surd r$	$\otimes - x_0$	$-x_0$
Rema Exact (re)	ha	$\otimes$		$\otimes$	
Rema Exact (re)	hp		$\otimes$	$-x_0$	$-x_0$
Rema Exact (re)	hva		$\otimes$		$\otimes$
Rema Exact (re)	ma	$\otimes\chi^2$	$\otimes\chi^2$	$\otimes\chi^2$	$\otimes\chi^2$
Rema Exact (re)	mvp	χ^2	$\otimes\chi^2$	$\otimes\chi^2 - x_0$	$\otimes\chi^2$
Rema Exact (re)	mva		$\otimes$		
Rematitan (rt)	ha	$\surd r$	$\surd r\chi^2$	$\otimes$	$\otimes$
Rematitan (rt)	hp		$\otimes$		
Rematitan (rt)	hva	$\surd r$	$\otimes\surd$	$\surd r$	$\surd r$
Rematitan (rt)	ma	$\surd r$	$\surd r$	$\otimes$	
Rematitan (rt)	mvp	$\surd$	$\surd$	$\otimes$	
Rematitan (rt)	mva	$\surd$	$\surd$	$\otimes$	$\otimes$
No. best fit according to highest correlation coefficient		5/24	9/24	16/24	11/24
No. Visually poor		12/24	12/24	2/24	2/24

3.1.2. Goodness-of-fit by determining the correlation coefficient the chi-squared statistic

The correlation coefficient and chi-squared statistic can be used to compare the distribution functions of the actual data and the 2 and 3 parameter Weibull distribution functions. They may be plotted for each data set as shown in Fig. 10 (2 parameter models) and Fig. 11 (3 parameter models). There seems to be greater scatter for the 2 parameter models than the 3 parameter models. For the 3 parameter models all correlation coefficients are greater than 0.9855, apart from the data set for rt_hva and all values of χ^2 are less than 3.841 (2 degrees of freedom) and 5.991 (3 degrees of freedom), respectively, apart from the data sets for le-hva, re-ma and re-mvp.

The correlation coefficient and chi-squared statistic are used here to compare the distribution functions of the data sets with the 2 and 3 parameter Weibull distribution functions. The 2 and 3 parameter distribution functions are generated using lower bound of strength X_0, characteristic strength θ, and weibull modulus m shown in Tables II and III.

Using regression the residuals indicate how all values of measured strengths deviate from the Weibull distributions, whereas using χ^2 only four residuals are obtained (Tables V and VI) and the data may not be distributed in the expected manner over the range of strengths that represent the four bins of a histogram. Large χ^2's do not necessarily indicate that the overall fit is poor e.g. two handling techniques, namely cf-mva and re-ma show good overall fit of the models (Figs 7, 9) but once the data is grouped into 4 bins small residuals for cf-mva result in a low χ^2, whereas for re-ma, large residuals result in a high χ^2 (Fig. 12). In the case of re-ma this may indicate that the data would be better fit using an alternative distribution.

3.1.3. Differences between characteristic strength and Weibull Modulus

By examining characteristic strength in Tables II and III it is apparent that characteristic strength does not change significantly when determined by the 2 or 3 parameter models (Fig. 13). However, there is a considerable disparity between Weibull Modulus computed for the 2 and 3 parameter models (Fig. 14). It is essential that modulus is determined with confidence since it gives an indication of the reliability of the material. High values of Weibull Modulus are generally obtained for engineering ceramics for which the fracture strengths show less scatter due to the ability of the processing route to remove defects such as porosity. The 2 parameter regression and maximum likelihood models generate Weibull modulii for the investments that would be abnormally high and would indicate a better material (less scatter in strength) than is actually the case e.g. Compare Wiebull Modulus for cf-ha at 14.74 (2 parameter regression) with cf-ha at 3.569 (3 parameter regression). Greater confidence in the 3 parameter

TABLE V Residuals for 2 parameter models

	Maximum likelihood				Regression			
	cf	le	re	rt	cf	le	re	rt
ha	3.407	4.049	0.948	4.612	3.350	4.167	0.783	5.139
	−1.995	−0.524	−1.251	2.396	−2.071	−0.746	−1.309	0.476
	−1.061	−3.230	−0.406	−6.533	−1.045	−3.479	0.067	−7.085
	−0.351	−0.296	0.709	−0.476	−0.235	0.058	0.459	1.470
hp	−0.150	7.610	−0.078	1.983	0.078	7.934	−0.673	2.299
	6.439	−3.319	1.676	−0.811	5.855	−5.287	0.592	−1.156
	−5.722	−2.651	−2.992	−0.317	−6.329	−2.691	−1.834	−1.052
	−0.567	−1.641	1.394	−0.855	0.396	0.044	1.914	−0.091
hva	−0.328	8.390	−0.433	0.863	0.250	8.758	−0.431	1.734
	2.832	−5.052	2.054	0.448	2.225	−6.180	1.953	−0.845
	0.888	−4.065	−0.362	1.503	−0.945	−4.876	−0.473	−0.001
	−3.391	0.726	−1.258	−2.814	−1.529	2.297	−1.048	−0.888
ma	0.422	0.107	5.010	1.612	1.003	0.796	4.866	2.354
	5.083	3.742	−1.427	6.700	2.832	2.616	−1.433	4.808
	−4.595	−2.746	−8.011	−6.333	−5.421	−4.332	−7.784	−7.658
	−0.910	−1.103	4.428	−1.979	1.585	0.920	4.350	0.497
mvp	2.127	1.599	0.451	0.701	1.277	1.375	0.020	1.378
	−3.074	−0.917	5.098	2.606	−3.797	−1.053	4.690	0.971
	−1.172	−0.239	−10.195	−3.543	1.576	0.212	−9.471	−4.442
	2.119	−0.443	4.646	0.235	0.943	−0.534	4.760	2.094
mva	−0.791	1.357	0.263	5.341	−0.843	0.232	0.494	5.657
	1.228	−2.963	1.185	−2.946	1.135	−4.060	0.848	−3.884
	−0.061	−1.162	−0.165	−0.912	−0.024	2.120	−0.791	−1.624
	−0.375	2.767	−1.283	−1.483	−0.268	1.709	−0.551	−0.150

TABLE VI Residuals for 3 parameter models

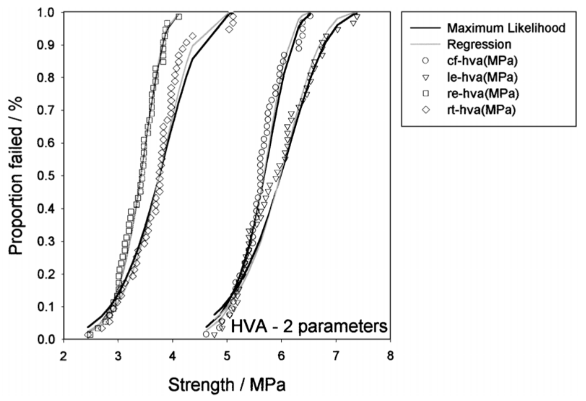
	Maximum likelihood				Regression			
	cf	le	re	rt	cf	le	re	rt
ha	3.110	2.102	1.101	1.276	1.966	0.191	0.882	1.420
	−3.622	−2.535	−1.844	0.039	−4.534	−1.622	−0.847	0.534
	−0.770	−0.399	−0.288	−1.664	0.908	1.197	−0.695	−2.033
	1.282	0.832	1.031	0.349	1.660	0.233	0.660	0.078
hp	−1.391	2.498	−0.346	1.622	−1.487	2.638	−0.573	1.380
	4.137	−4.530	2.079	−3.002	4.939	−4.102	1.739	−2.278
	−3.092	3.174	−1.676	1.244	−3.204	2.873	−1.255	1.276
	0.346	−1.142	−0.057	0.136	−0.248	−1.409	0.089	−0.378
hva	−0.478	4.633	−0.466	0.491	−0.472	4.195	−0.926	0.786
	−0.590	−7.770	0.594	−2.462	0.313	−7.009	0.772	−1.665
	2.438	0.892	0.376	3.753	1.850	1.007	1.247	2.700
	−1.370	2.245	−0.504	−1.782	−1.692	1.807	−1.093	−1.822
ma	−2.270	−0.815	4.213	−0.594	−1.913	−0.565	2.239	0.097
	1.724	0.160	−3.035	3.317	2.168	0.825	−3.660	3.593
	0.258	0.176	−7.087	−2.312	−0.389	−0.608	−4.888	−3.293
	0.289	0.480	5.908	−0.410	0.135	0.348	6.308	−0.397
mvp	1.972	1.665	0.407	−0.924	1.539	1.327	−0.057	−0.649
	−2.545	−1.335	5.511	−0.149	−2.856	−1.199	4.354	0.369
	−0.704	−0.333	−9.805	0.095	0.867	0.298	−9.486	−0.569
	1.276	0.055	3.887	0.978	0.451	−0.426	5.187	0.849
mva	−0.626	1.085	0.037	1.617	−0.863	0.675	0.056	0.758
	0.483	−2.183	−0.732	−5.334	1.004	−2.517	0.288	−4.357
	−0.262	−0.365	1.158	3.740	0.083	1.077	0.569	4.181
	0.406	1.464	−0.464	−0.023	−0.224	0.765	−0.913	−0.581

models is proposed since fit for the majority of data sets is visually excellent and this is confirmed quantitatively using correlation coefficient as the measure of goodness-of-fit.

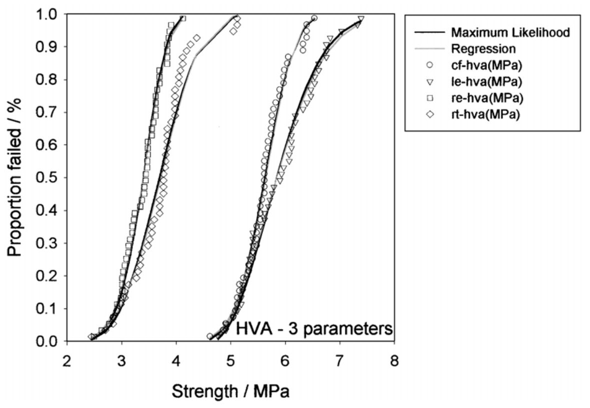
3.2. Strength and scatter of data

Since it has been shown that the characteristic strength may be determined reliably using either maximum likelihood or regression techniques then some comment should be made about the differences in strength that were observed when comparing handling techniques and materials. The following trends are discussed using the 3 parameter models since these have been shown to give better fit overall.

During mixing, air bubbles are introduced that create voids in these materials. The voids result in bubbles within the investment and on the wax pattern. On the

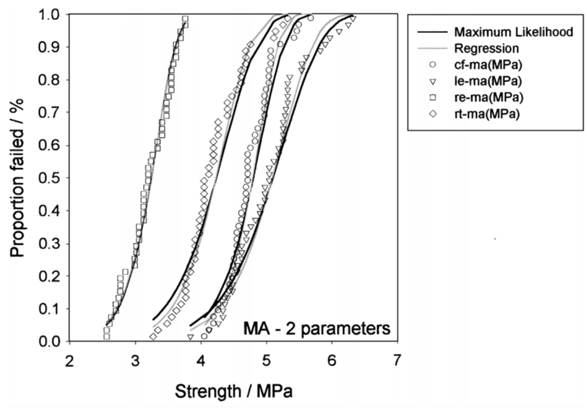


(a)

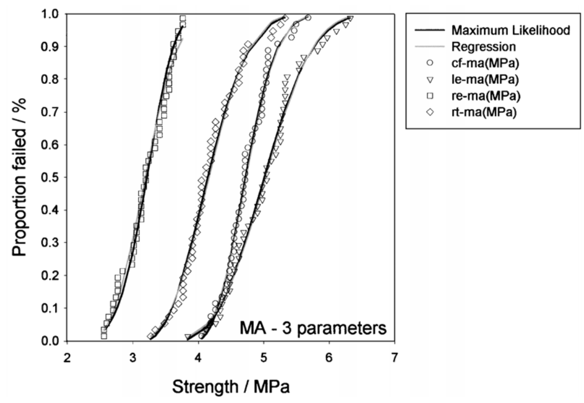


(b)

Figure 6 Plot of measured strength for all materials using the HVA handling technique and curves generated from the measured data points using (a) 2 Weibull parameters (b) 3 Weibull parameters. The parameters of the distribution function were determined using methods of regression and maximum likelihood.



(a)



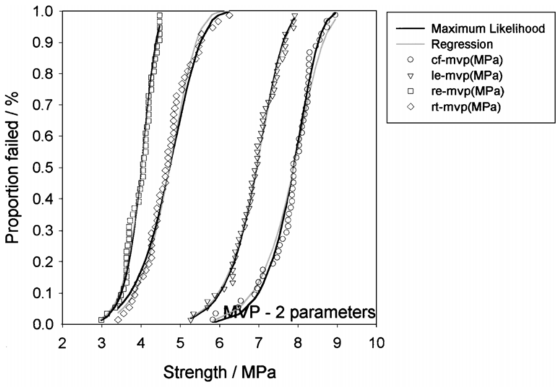
(b)

Figure 7 Plot of measured strength for all materials using the MA handling technique and curves generated from the measured data points using (a) 2 Weibull parameters (b) 3 Weibull parameters. The parameters of the distribution function were determined using methods of regression and maximum likelihood.

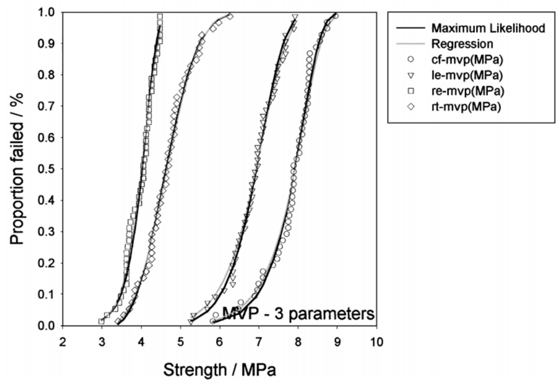
cast metal these voids result in nodules that must be removed by hand during finishing. The MVP and MVA handling techniques are the most effective methods of removing these bubbles. An additional benefit of removing these bubbles is an increase in the strength of the as-cast investment at room temperature. This increase in strength is not a feature of all the investments tested. Croform WB shows a marked increase in strength when processed by the MVP and MVA techniques but the Levotherm, Rema Exact and Rematitan investments seem to be more insensitive to handling technique despite MVP resulting in highest strength for all materials. Similar trends may be observed for the scatter of the data. This is shown by the values of Weibull modulus. In the case of Croform WB, Weibull Modulii of 7.1 and 8.1 for MVP and MVA were determined compared with values below 5.7 for other handling techniques. Similar trends were observed for Levotherm but not for Rema Exact or Rematitan. The scatter of data for Rematitan seemed most insensitive to handling technique and Weibull Modulus remained low and relatively constant below 3.8. Higher values of Weibull Modulus are desirable for engineering applications since reliability is greater at higher Modulus.

Furthermore, it is important to speculate about the causes of the scatter of the data. The discussion has concentrated on the presence of pores or voids introduced during handling. It has been demonstrated that pore sizes can be reduced by the use of mechanical spatulation in a vacuum coupled with setting under pressure. The effect of this reduction in pore size was an increase in strength and a reduction, on the whole, in the scatter of the data. However, there are other possible causes of scatter. Two such causes are, on the one hand, the number of pores in the sample, and on the other the position of a pore in the sample.

In the four point bend test the stress on the sample between the two central loading knife-edges is constant but the top of the specimen above the midline is in compression whilst the lower portion below the midline is in tension. Since pores do not have an effect on the strength of the material in compression, pores above the midline of the specimen may be disregarded as crack initiators. However, in the lower section of the specimen pores will result in fracture due to critical pore size effects. Similar size pores on or near the lower surface will probably be more instrumental in initiating cracks than those closer to the midline of the specimen. The probability of fracture due to the presence of pores would be that much greater the more pores of a critical size that there are present in the sample, although only one is necessary to actually cause fracture. Thus, apart from the presence of pores of different sizes, an additional cause of the scatter would be the depth of the largest pore from the surface in one specimen compared with another.

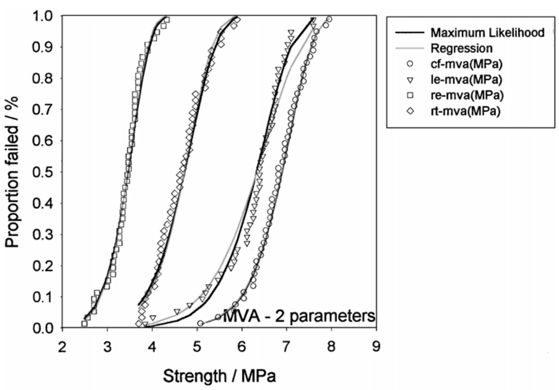


(a)

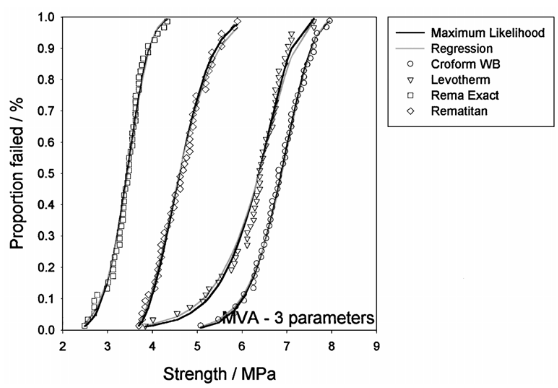


(b)

Figure 8 Plot of measured strength for all materials using the MVP handling technique and curves generated from the measured data points using (a) 2 Weibull parameters (b) 3 Weibull parameters. The parameters of the distribution function were determined using methods of regression and maximum likelihood.



(a)



(b)

Figure 9 Plot of measured strength for all materials using the MVA handling technique and curves generated from the measured data points using (a) 2 Weibull parameters (b) 3 Weibull parameters. The parameters of the distribution function were determined using methods of regression and maximum likelihood.

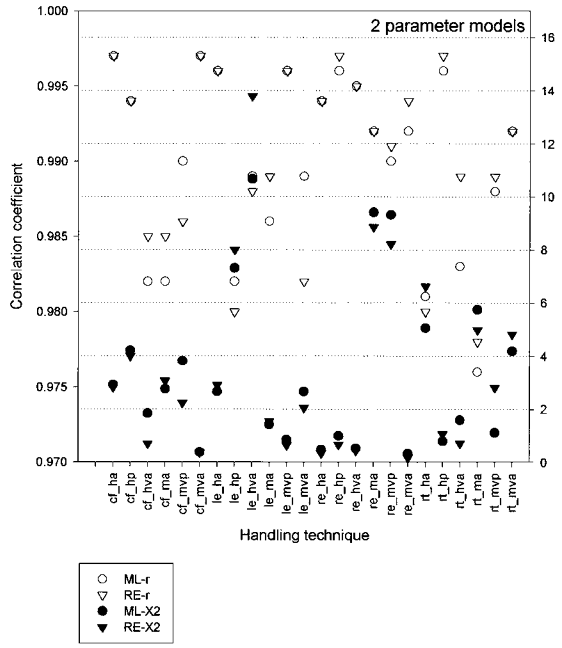


Figure 10 Correlation coefficients and χ^2's of all materials and handling techniques indicating the degree of goodness-of-fit of the 2 parameter Weibull distribution to the measured data.

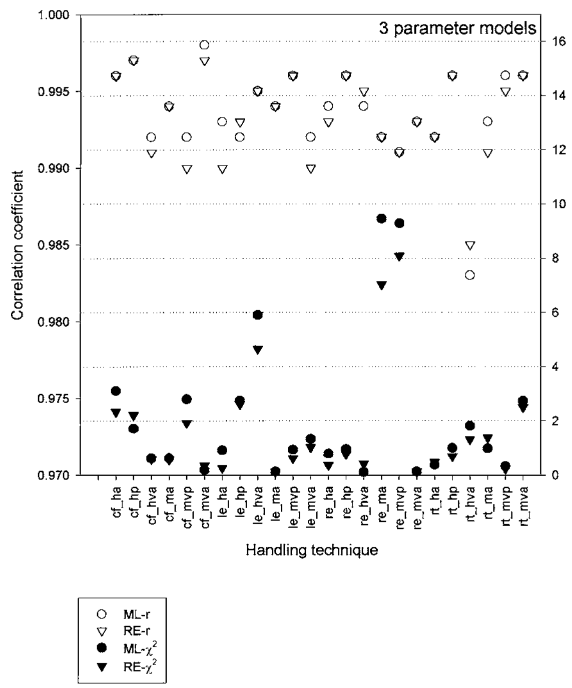


Figure 11 Correlation coefficients and χ^2's of all materials and handling techniques indicating the degree of goodness-of-fit of the 3 parameter Weibull distribution to the measured data.

The arguments outlined above may also be applied to the 'holes' created in the materials by the refractory particles. The constituents of phosphate-bonded investments are refractory particles of silica and a matrix of magnesium ammonium phosphate. The sizes of the refractory particles in the four investments were measured using sieves of mesh sizes ranging from 45 μm to 710 μm. Although the majority of particles in all investments were found to be between 125 μm and 150 μm, a small percentage (<2.5 %) were larger than 700 μm. The largest voids found in specimens

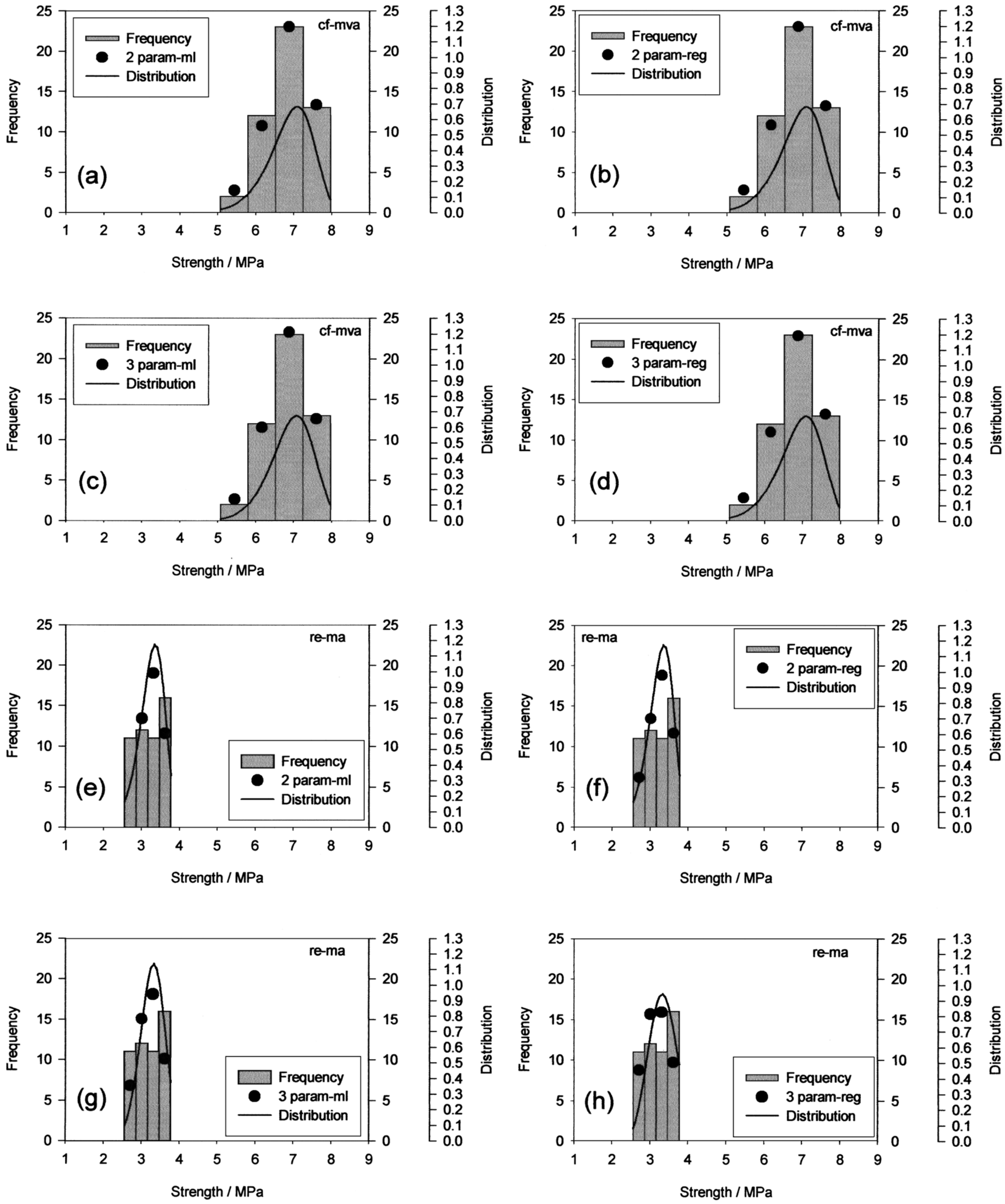


Figure 12 Histograms for the mva handling technique for Croform WB (a–d) and the ma handling technique for Rema Exact (e–h) generated by dividing the measured data into 4 bins. Estimated frequencies using the weibull distributions are indicated and the shapes of the corresponding distributions drawn. 2 and 3 parameter Weibull distributions are shown and parameters were determined using methods of regression and maximum likelihood.

processed by the HA handling technique were larger than any refractory particle and up to 2500 μm in diameter. On setting and prior to heating the refractory particles in phosphate-bonded investments are not believed to create a bond with the matrix phase (10). Thus, the interface between refractory particle and matrix is not capable of transferring stress. This means that the refractory particles act as stress raisers in the materials, much like the voids. Thus, it is of interest to compare pore or void sizes with refractory particle sizes. It has already been noted that, for the HA handing techniques, pore sizes are greater than refractory particle sizes but when pores are removed through mechanical spatulation and setting under pressure, refractory particles are larger than the pores that remain. In fact, it has been shown that for pore sizes less than 400 μm the mechanism of fracture may change and could be attributed to refractory particle size rather than pore size (2).

On heating this scenario would be expected to change since bonds form between the matrix reaction products and the refractory particles and only air bubbles would be expected to contribute to the critical hole size effect. Thus, if pores are virtually eliminated through use of

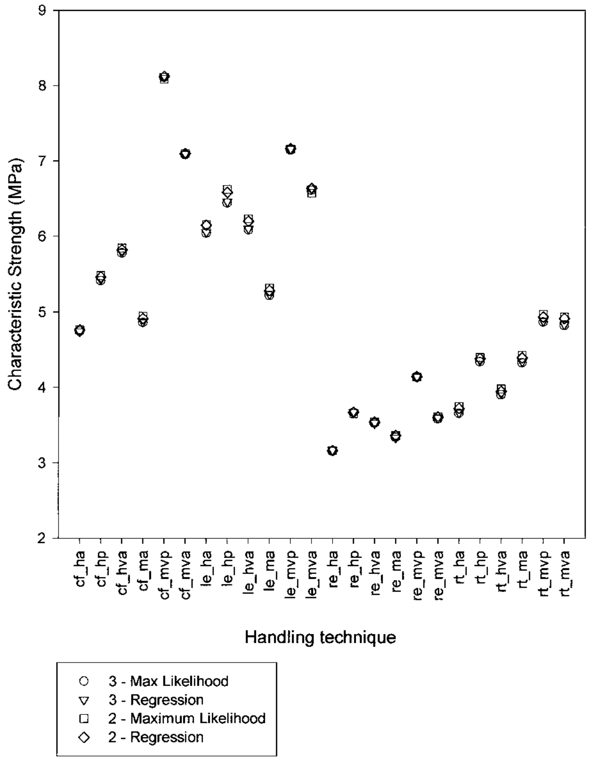


Figure 13 Characteristic strengths for all materials and handling techniques determined using methods of regression and maximum likelihood and 2 and 3 parameter Weibull distributions.

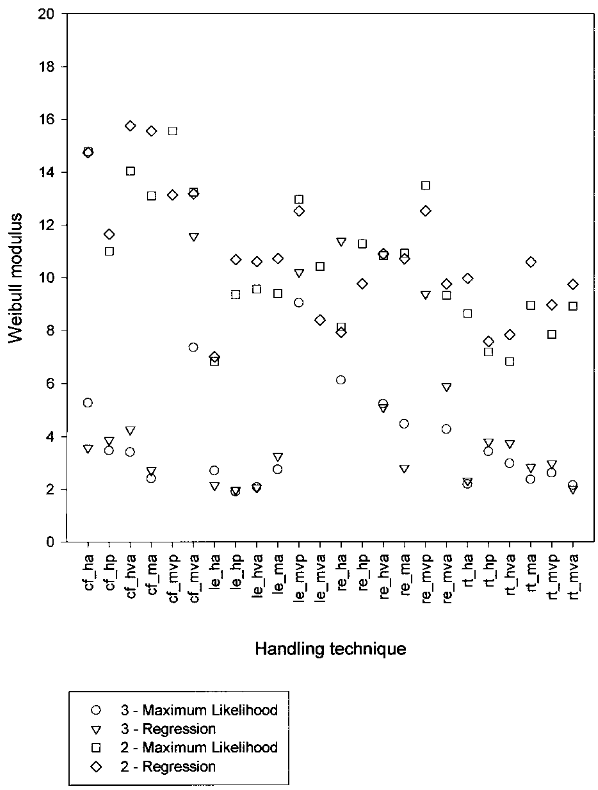


Figure 14 Weibull modulii for all materials and handling techniques determined using methods of regression and maximum likelihood and 2 and 3 parameter Weibull distributions.

an appropriate handling technique, then strength would be limited by the resistance of the matrix to crack initiation and propagation. The refractory particles would still have a role to play since they could arrest cracks propagating through the matrix. Such mechanisms have been used in dental composites to improve resistance to crack propagation.

Preliminary tests of hot strength have been carried out at 900°C and it was found that the Croform and Levotherm materials retain the pores that were incorporated during processing. There does not seem to be a sintering affect over the time periods that these mould materials would be used for casting or superplastic forming. A difference in strength between the HA and MVP processes materials was still observed. Likewise, the Rematitan materials seemed to be as insensitive to the presence of pores as at room temperature. However, in the case of Rema Exact, in particular, the hot strength recorded for the two handling techniques was identical, showing that the presence of pores had become insignificant at these void concentrations.

4. Conclusions

1. Two and three parameter Weibull distributions have been shown to be appropriate descriptions of the 4-point bend strength of four dental casting investments prepared using six different handling techniques.

2. Visually the 3 parameter distributions show a better fit than the 2 parameter distributions.

3. Correlation coefficient has been used to identify the distribution that best describes each data set.

4. The introduction of air bubbles or pores during mixing and setting has a significant effect on strength at room temperature for all materials whereas at high temperatures the strength of some materials is not apparently influenced by the presence or otherwise of the same pores.

5. The MVP handling technique has the effect of increasing strength in all materials at room temperature by the removal of air bubbles or pores but in some materials it also has the effect of markedly reducing the scatter in the results. In other materials scatter is unaffected by handling technique and remains relatively high.

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