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Statistical approach to mechanical behaviour of ceramic matrix composites based on Portland clinker

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Abstract

This work presents the results of the Weibull modulus of Portland clinker reinforced with several oxides like alumina, magnesia and silica. The Weibull modulus is a measure of the reliability of materials and it is used as an estimation of the probability of failure. The composite materials (3, 6 and 9% of oxides, by weight) were manufactured through Powder Technology processes including mixing in a ball mill, cold isostatic pressing and sintering at 1400°C in air. Sintering density for all the materials has been evaluated. Bending strength (three points) was the key property for measuring the Weibull modulus using at least 30 tests in all cases. Four different failure estimators were studied. These composite materials present a high Weibull modulus with good correlation coefficients. Some results are above typical values of the conventional and advanced ceramics. Microstructural analysis was carried out to explain the reliable behaviour of these composite materials.

Keywords: B. Composites; Oxides; Weibull statistics

1. Introduction

Reliability is highly related to the survival probability of a useful component and with the conservation of the properties of a material. Reliability would be related with the most probable value of the evaluated mechanical property, being higher with a lower quantity of abnormal values or with a narrow interval of values. Then it is an extension of the operational endurance during a period of time. As a function of the basic mechanical properties and the reliability of the material, the best application could be found.

In ceramic materials and ceramic matrix composites (CMCs), the mechanical values usually present a greater dispersion than those obtained for metallic materials, so their reliability is lower [1]. Normally, the results obtained from a test on a perfectly characterised material (composition, processing and purity level) present a certain dispersion [2], being not totally attributable to the test. The dispersion indicates a statistical behaviour of the material, due to intrinsic variables not entirely dependent on material characterisation.

New processes and technologies have made ceramic and CMCs more reliable [3–6]. They maintain their properties and are not so limited by reliability or elevated costs of production. However, it is necessary to know their strength values and their distribution of defects to control their reliability. The most common defects in ceramics are found on surface (such as roughness) and in volume (pores, inclusions). Volume defects produce more homogeneous strength values than the surface defects. Defects smaller than 50 μm are not easily detectable by non-destructive tests and could lead to the component failure [7]. The relationship between the final roughness of the part and its strength is very high. The strength decreases with the increase of roughness and relies on the polishing direction. In addition, the average strength depends on the size of the sample, since it is probable that large size defects exist in a greater volume.

The first probabilistic approach used to account for the scatter in fracture strength of brittle materials was introduced by Weibull [8]. His theory is based upon "weakest link" hypothesis that failure of the structure

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must occur when any one element fails. There are several studies about the reliability of different composite and ceramics materials: SiC (with Weibull modulus between 8 and 10) [9], Si_3N_4 [10], alumina [11], Al_2O_3/ZrO_2 [12] and alumina fibres [13]. Brittle fibres present a Weibull modulus between 2 and 5 with a high dispersion, while it oscillates between 5 and 15 (with low dispersion) in glass fibres. Monolithic and polycrystalline ceramics present Weibull modulus up to 25 [14]. Studies about reliability of composites based on clinker with Al_2O_3 and SiC [15,16] show that their Weibull modulus oscillates between 8 and 15, with a low dispersion.

Properties related to the strength of the materials need a great number of tests to establish their distribution and find a reliable survival parameter. The use of statistical models requires that samples reflect the distribution of defects of the material or component. The probability of failure of such components will therefore depend on the variability of the applied loads in addition to the variability of the defect populations [17]. The electronic components adjust to a Poisson distribution (random failures and constant failure rate with the time). However, not all the components possess a constant failure rate. In many cases, it increases and then this behaviour adjusts to a Weibull distribution. The Weibull distribution is the most commonly used and when exists a acceptable failure risk of the component. This function presents the best approach to the behaviour of mechanical and electromechanical components and it is often used to determine the dispersion of mechanical results in brittle materials [18].

A brittle material could have more than one defect; in fact, defects exist of different sizes, forms and orientations in relation to the applied load. In CMCs with brittle behaviour, a random distribution of defects could obey to statistical considerations. The Weibull distribution through the resistant properties of a polycrystalline composite material could be expressed by the simple Eq. (1):

$$F = 1 - \exp\left(-\alpha\, f(\sigma)^{\beta}\right) \tag{1}$$

where $f(\sigma)$ is function of the applied stress [Eq. (2)], F is the probability of failure, β is the dispersion or Weibull modulus and determines the shape of the curve or distribution, and α is a scale parameter. Eq. (2) is as follows:

$$f(\sigma) = \left(\frac{\sigma - \sigma_u}{\sigma_0}\right) \tag{2}$$

σ_0 being a characteristic strength or scale constant and it is the strength at $\sigma = \sigma_0$ in that the survival probability is reduced to 37%. The σ is the statistical utilised variable, in this case the three-point bending strength. The σ_u, localisation parameter or threshold strength [2], indicates the strength from which the distribution of defects is in accordance with Weibull. It usually takes a value equal to zero to simplify Eq. (3) [1,2]. The other two parameters (σ_0 and σ) can easily be found and depend on the material characteristics. From both Eqs. (1) and (2), it can be obtained:

$$\mathrm{Ln}\ \mathrm{Ln}\frac{1}{1-F} = \beta\ \mathrm{Ln}\ (\sigma - \sigma_u) - \beta\ \mathrm{Ln}\sigma_0 \tag{3}$$

If the same size is considered for all the samples, then β or Weibull modulus (slope) could be determined graphically by logarithms. A linear equation where failure probability is related to the applied load (4), where it is overestimated the failure probability:

$$\mathrm{Ln}\ \mathrm{Ln}\frac{1}{1-F_j} = \beta\ \mathrm{Ln}\ \sigma_j - \beta\ \mathrm{Ln}\sigma_0 \tag{4}$$

being σ_j the bending strength (or key property) value of the j experiment and F_j (estimator of the failure probability) [19].

To find reliability behaviour, it is necessary to arrange all the observations (experimental results) in ascending order. Next, the different values of F_j (estimator or cumulated probability of failure) are determined. When observations are few (20 or 30), it is necessary to extrapolate at a significant population (100 observations). In these cases, failure estimator F_j is employed to allow extrapolation of the experimental results to significant population and when the exact median values are not available. Through the failure estimators (or medium ranges), it is possible to estimate the real value of the key property.

2. Experimental procedure

Composite materials based on Portland clinker were manufactured using the typical methods of consolidation of conventional and advanced ceramics. Table 1 shows the characteristics of the clinker employed (supplied by Portland Valderrivas, Spain) and Table 2 presents the characteristics of the added oxides.

Portland clinker was dry mixed with the different oxides (Al_2O_3, MgO and SiO_2) in proportions of 3, 6 and 9 wt.%, in a ball mill during 30 min using stainless steel balls, being the ratio by weight material/balls as 1/5. The homogeneity of the different mixtures was checked.

The ceramic mixtures and the plain clinker were encapsulated in flexible moulds and subsequently degassed to avoid the formation of pores. Then, the samples were compacted at 180 MPa by cold isostatic pressing (CIP) in wet bag. Subsequently, the materials were sintered in air at 1400°C, optimised in previous work [20]. Other properties were evaluated, and are presented elsewhere [21]. Sintering density was evaluated by Archimedes principle [22]. Image analysis (Image ProPlus® v. 3.0) has been employed to estimate the porosity levels (in area). The samples were machined to adequate dimensions to realise the bending strength tests. Finally, a microstructural study was carried out to determine the influence of reactions between the reinforcements and the Portland clinker.

The Weibull modulus was evaluated using bending strength as the key property. The three-point bending strength was determined by MPIF 41 [23], the load was applied on the surface with lower roughness, being uniform in all the materials. All samples presents same dimensions to avoid volume factor effect on calculated Weibull modulus.

A number of methods gives a reliable approach to the Weibull modulus, most of which use one of the estimators of the failure probability [1,19]. In this work, four typical estimators were chosen:

$$F_j = j/(n+1)$$
$$F_j = (j-0.3)/(n+0.4)$$
$$F_j = (j-0.5)/n$$
$$F_j = (j-3/8)/(n+1/4)$$

where j is the rank of the experiment and n is the total number of experiments. The first estimator is more advisable in the case of a Gaussian distribution while the others are more suitable for an asymmetric distribution. For good linear adjustment of equations, a number of experiments are required. In this work, for each material at least 30 experiments were made.

Table 1
Characteristics of employed Portland clinker

Composition by oxides		Mineralogical composition	
$\%SiO_2$	20.24		
$\%Al_2O_3$	6.09		
$\%Fe_2O_3$	3.51	$\%C_3S$	63.18
%MgO	1.11	$\%C_2S$	12.13
%Sulphates	1.34	$\%C_3A$	10.20
%CaO	65.72	$\%C_4AF$	10.68
%CaO free	1.21		
%Fire losses	0.14		

Table 2
Characteristics of added reinforcements

Additives	Powder characteristics
α-Al_2O_3	Purity: 99.9%, 98% <2 μm. Density: 3.90 g/cm^3 (Alcoa, Brazil)
MgO	Purity: 90.0%. Size 95% <5 μm. Density: 3.60 g/cm^3 (Panreac, Spain)
SiO_2	Amorphous. Purity: 99%. Density: 2.65 g/cm^3 (Crosfield Chemicals, UK)

3. Results

The densities of these CMCs are summarised in Table 3. All materials present density values higher than plain clinker. All materials present levels of porosity lower than 4% and composites with silica additions have the lowest level of porosity.

Fig. 1 shows the results of average bending strength for all materials. In the studied interval, bending strength increases with the reinforcement amount and plain Portland clinker presents the lowest value. The statistical parameters of the calculated Weibull regressions for the four estimators are detailed in Table 4. A higher slope (β) represents a greater Weibull modulus. The most of the materials have correlation coefficients higher than 94%, only material with 9% alumina presents lower correlation and Weibull modulus for all estimators. Figs. 2–4 present the modulus as a function of the different estimators for composite materials with alumina, magnesia and silica, respectively. The most favourable estimator is $F_j=(j-0.5)/n$ for all materials, with good correlations. Weibull modulus values (between 26 and 29) for the 3% added magnesia is found.

Fig. 5 shows the microstructures of composite materials reinforced with alumina. Fig. 6 presents the porosity evolution of materials reinforced with magnesia and Fig. 7 shows the microstructural evolution of materials with silica addition. Fig. 8 illustrates a Weibull plot of

Table 3
Density of studied materials

Sintering density (g/cm^3)	Plain clinker	Al_2O_3	MgO	SiO_2
0%	3.08	–	–	–
3%	–	3.09	3.12	3.13
6%	–	3.11	3.17	3.16
9%	–	3.12	3.31	3.38

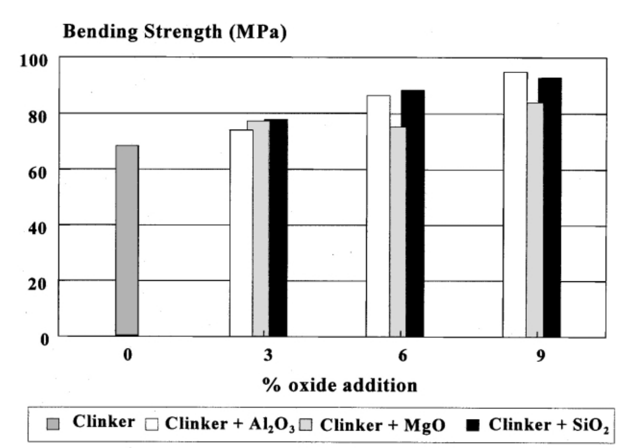

Fig. 1. Influence of the different additions on bending strength of the composite materials based on Portland clinker.

Table 4
Summary of statistical results of Weibull regressions for all analysed estimators

Material	σ_m	Estimators	β	$\beta.\ln(\sigma_0)$	σ_0	R2
Plain clinker	68.41	$F_j=j/(n+1)$	16.97	72.13	70.16	0.9710
		$F_j=(j-0,3)/(n+0,4)$	17.75	75.45	70.10	0.9720
		$F_j=(j-0,5)/n$	18.35	77.99	70.06	0.9726
		$F_j=(j-3/8)/(n+1/4)$	17.97	76.36	70.08	0.9722
$+3\%Al_2O_3$	74.1	$F_j=j/(n+1)$	8.73	37.89	76.67	0.9832
		$F_j=(j-0,3)/(n+0,4)$	9.40	40.76	76.52	0.9817
		$F_j=(j-0,5)/n$	9.97	43.25	76.41	0.9782
		$F_j=(j-3/8)/(n+1/4)$	9.60	41.62	76.48	0.9808
$+6\%Al_2O_3$	86.53	$F_j=j/(n+1)$	14.46	65.01	89.53	0.9824
		$F_j=(j-0,3)/(n+0,4)$	15.57	69.96	89.41	0.9860
		$F_j=(j-0,5)/n$	16.54	74.30	89.34	0.9873
		$F_j=(j-3/8)/(n+1/4)$	15.90	71.46	89.38	0.9866
$+9\%Al_2O_3$	95.04	$F_j=j/(n+1)$	6.29	29.01	100.39	0.9124
		$F_j=(j-0,3)/(n+0,4)$	6.59	30.34	100.13	0.9109
		$F_j=(j-0,5)/n$	6.81	31.37	99.96	0.9098
		$F_j=(j-3/8)/(n+1/4)$	6.67	30.71	100.07	0.9105
+3%MgO	77.45	$F_j=j/(n+1)$	25.76	112.50	78.85	0.9422
		$F_j=(j-0,3)/(n+0,4)$	27.72	121.11	78.92	0.9410
		$F_j=(j-0,5)/n$	29.43	128.55	78.87	0.9375
		$F_j=(j-3/8)/(n+1/4)$	28.31	123.69	78.91	0.9400
+6%MgO	75.38	$F_j=j/(n+1)$	12.24	53.37	78.40	0.9564
		$F_j=(j-0,3)/(n+0,4)$	13.09	57.07	78.31	0.9476
		$F_j=(j-0,5)/n$	13.82	60.24	78.25	0.9371
		$F_j=(j-3/8)/(n+1/4)$	13.34	58.17	78.29	0.9443
+9%MgO	84.18	$F_j=j/(n+1)$	15.43	68.85	86.7071	0.9543
		$F_j=(j-0,3)/(n+0,4)$	16.23	72.40	86.6055	0.9521
		$F_j=(j-0,5)/n$	16.84	75.13	86.5578	0.9497
		$F_j=(j-3/8)/(n+1/4)$	16.45	73.38	86.5899	0.9513
$+3\%SiO_2$	77.95	$F_j=j/(n+1)$	15.90	69.51	79.1979	0.9794
		$F_j=(j-0,3)/(n+0,4)$	17.05	74.54	79.1133	0.9756
		$F_j=(j-0,5)/n$	18.05	78.87	79.0594	0.9696
		$F_j=(j-3/8)/(n+1/4)$	17.40	76.04	79.1015	0.9739
$+6\%SiO_2$	88.47	$F_j=j/(n+1)$	13.20	59.63	91.5436	0.9546
		$F_j=(j-0,3)/(n+0,4)$	13.90	62.77	91.4466	0.9543
		$F_j=(j-0,5)/n$	14.44	65.212	91.36	0.9542
		$F_j=(j-3/8)/(n+1/4)$	14.09	63.645	91.41	0.9542
$+9\%SiO_2$	93.07	$F_j=j/(n+1)$	13.25	60.553	96.55	0.9539
		$F_j=(j-0,3)/(n+0,4)$	14.16	64.698	96.42	0.9473
		$F_j=(j-0,5)/n$	14.94	68.249	96.34	0.9392
		$F_j=(j-3/8)/(n+1/4)$	14.43	65.931	96.39	0.9448

plain clinker (similar to the rest of materials) and the material with 9% of alumina, showing the differences between both, that will explain the lower correlation obtained in this material.

4. Discussion

The reliability results obtained for these materials are very interesting. Most of the materials present medium-high values compared with conventional and advanced ceramic composites. The addition of alumina to the clinker increases the bending strength (Fig. 1). Its values increases with the amount of added oxide due to increment of density (Table 3). This is produced by the reaction between alumina and Portland clinker, which forms a greater amount of tricalcic aluminate phase, capturing CaO of the clinker and producing a greater amount of belitic $2CaO.SiO_2$ (C_2S) phase of small size (Fig. 5). All composite materials with alumina present similar shape and size of pore than plain clinker. However, alumina addition decreases the reliability with respect to plain clinker. Maximum values of reliability (about 15) are achieved with 6% of Al_2O_3 (Fig. 2) with good values of

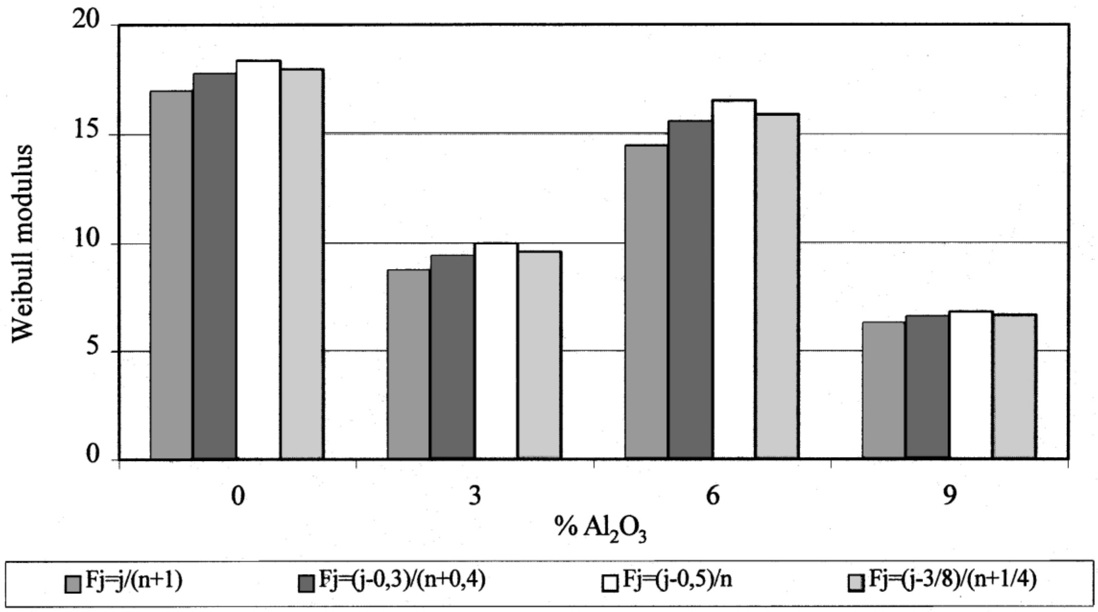

Fig. 2. Weibull modulus of the composite materials with alumina for the four estimators.

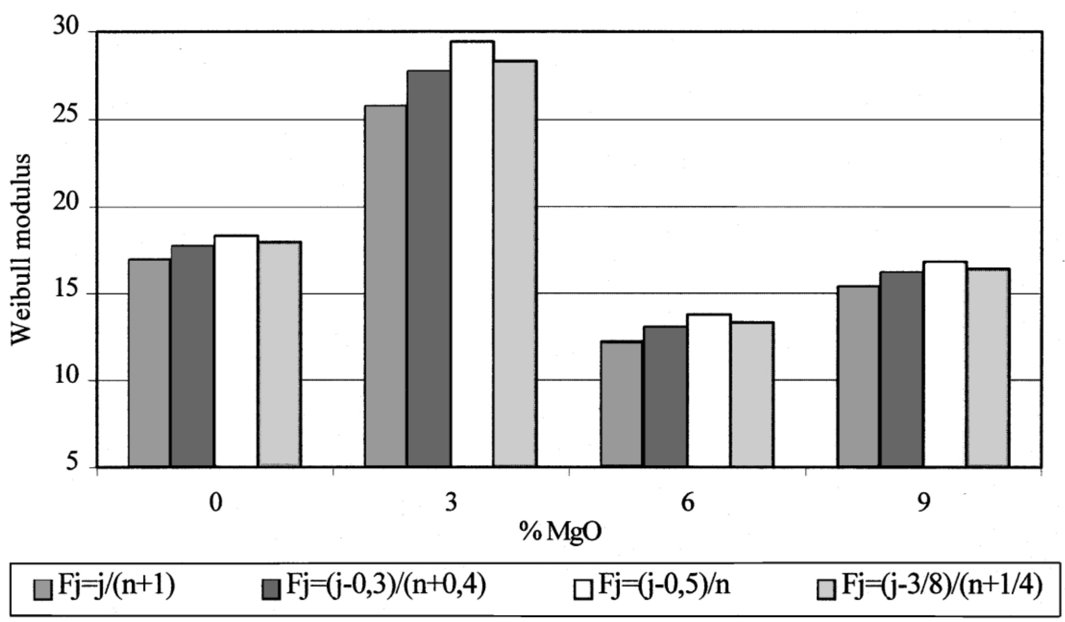

Fig. 3. Weibull modulus of the composite materials with magnesia for the four estimators.

correlation coefficient (Table 4) and bending strength (Table 3). On the other hand, the increase of narrow nets of $3CaO.Al_2O_3$ (C_3A) along all microstructure can affect negatively the reliability behaviour (material with 3% Al_2O_3). The presence of C_3A and C_2S has a positive influence on reliability of white Portland clinker [24]. This positive feature also occurs in Portland clinker, and so reliability should increase with Al_2O_3 amount. Material with 9% of alumina could be considered as an anomalous result, due to is low Weibull modulus and low regression but it is necessary to observe the differences between plain clinker regression and 9% of alumina composite (Fig. 8). In previous work [21], the formation of more C_3A and C_2S produces a reduction in brittle wear behaviour. From this point of view, material with 9% of alumina would be less brittle and hence more reliable, but apparently presents the lowest modulus and a low regression coefficients with all estimators, while porosity amount is similar to that in plain clinker. However, in Eq. (4), we consider that σ_u is zero to make simplifications. But the convex curve shape (Fig. 8) indicates that σ_u is not zero (only for this material). Then, the failure probability is overestimated and there is a survival period between $\sigma = 0$ and $\sigma = \sigma_u$.

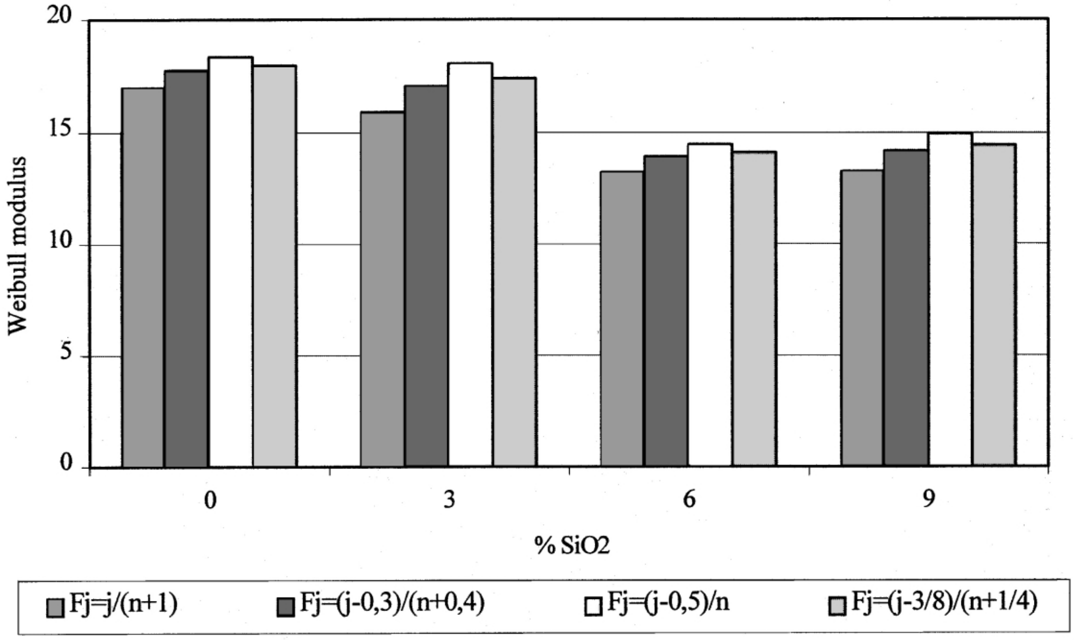

Fig. 4. Weibull modulus of the composite materials with silica for the four estimators.

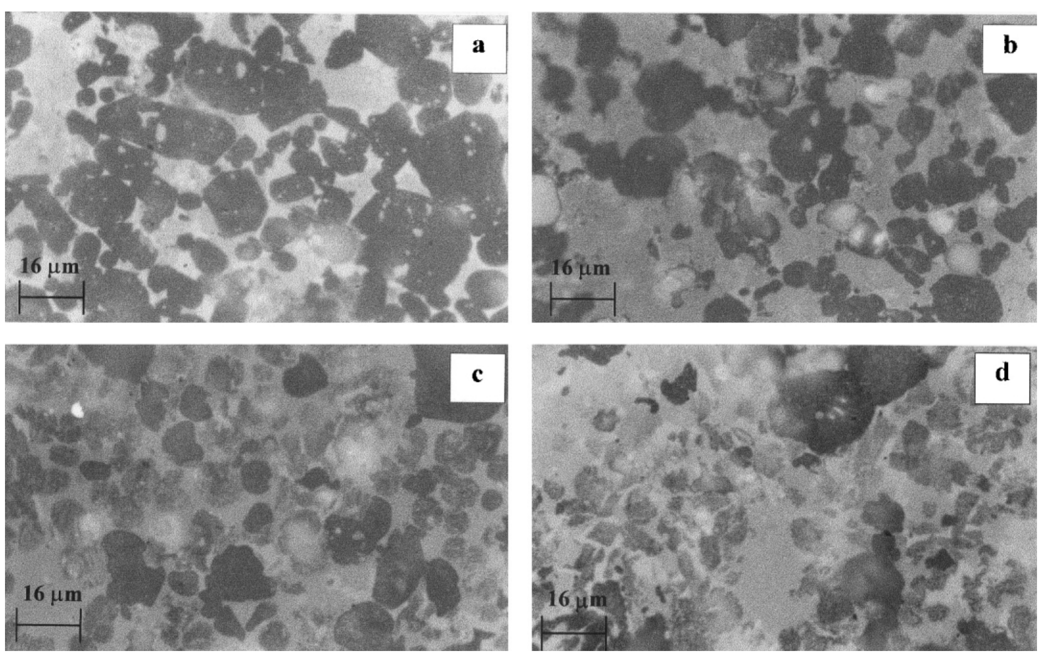

Fig. 5. Microstructures of composite materials reinforced with alumina. Detail of morphology phase evolution with increasing the alumina content: (a) plain clinker, (b) with 3%, (c) 6% and (d) 9% of alumina. Increment in belitic and aluminoferritic phases to form calcium aluminates. Etching 13% vol. acetic acid/ethanol.

Through several iterations, the estimated σ_u is about 75 MPa and its correlations are higher than 0.95. In this case, the calculation of the Weibull modulus must be carried out through numerical simulation methods.

The most favourable estimator respect Weibull modulus is $F_j = (j–0.5)/n$ for all alumina additions (Fig. 2), but all Weibull modulus and correlations values are very close for all estimators in the addition interval. Then, this estimator induces a good adjustment with lower alumina addition to explain the reliability behaviour.

The increasing in magnesia reinforcement has a positive influence on the bending strength compared with plain clinker (Fig. 1), and produces the opposite effect on the reliability (Fig. 3). The material with a low addition (Fig. 6b) presents a very high Weibull modulus

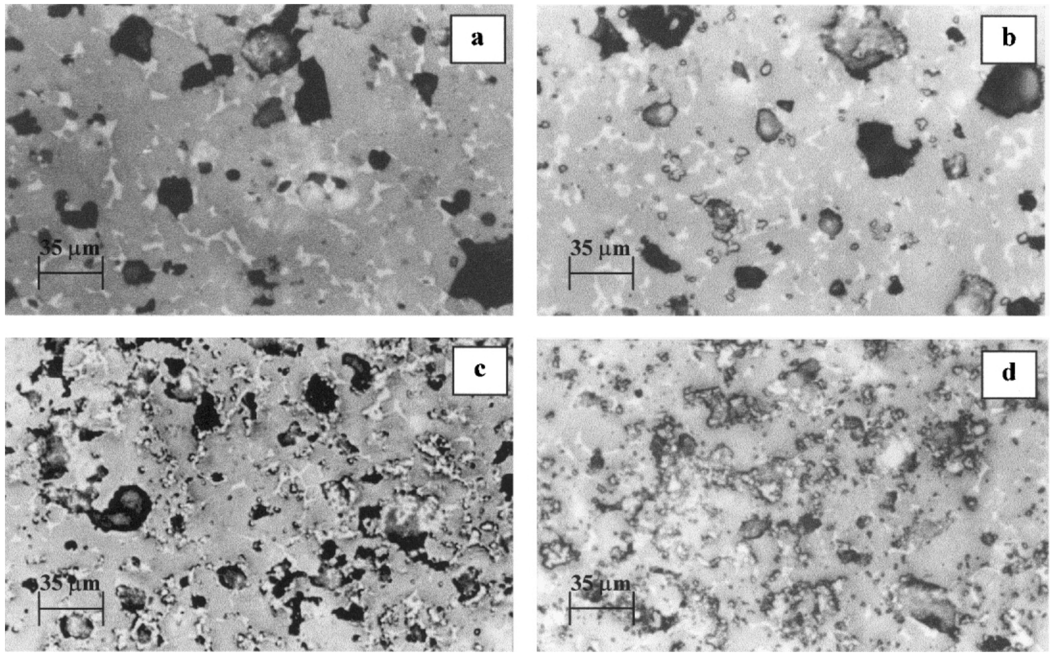

Fig. 6. Porosity evolution of composite materials based on clinker reinforced with magnesia: (a) plain clinker, (b) with 3%, (c) 6% and (d) 9% of magnesia. Without etching.

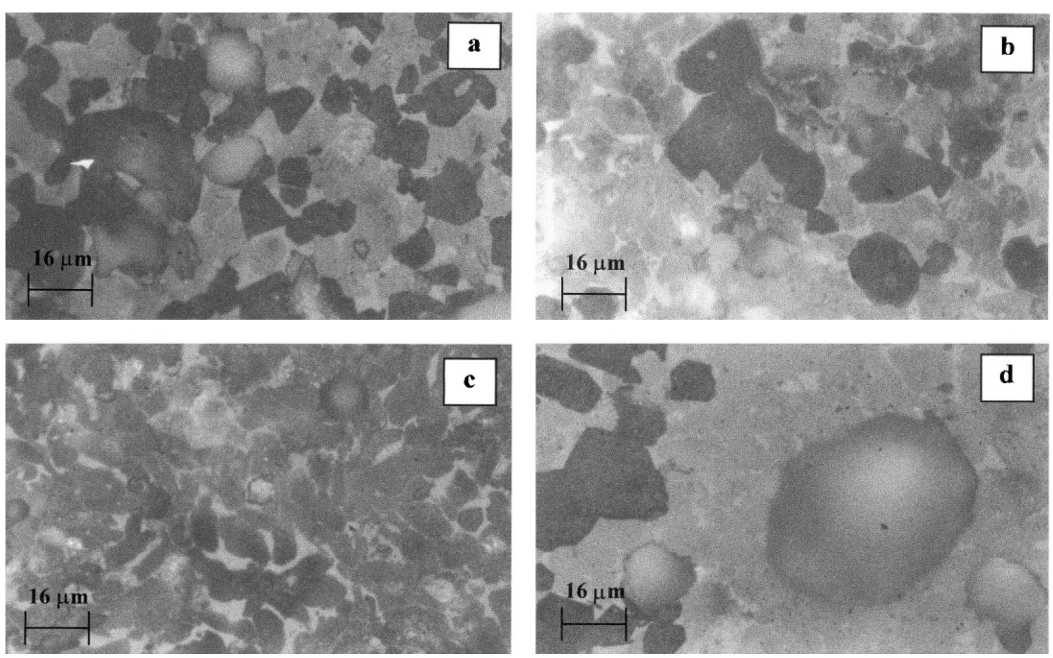

Fig. 7. Microstructures of composite materials base clinker reinforced with silica. Detail of evolution morphology of phases with increment of silica to form rich phases silica: (a) clinker with 3% of silica, (b) with 6%, (c) with 9% and (d) detail of a rich-silica phase around of large-size pore. Etching 13% vol. acetic acid/ethanol.

(between 26 and 29) due to lower magnesia amount with enough coherence with base material thought C_3A phase [25]. Magnesia particles have a uniform distribution (Fig. 6b) and the same probability to break then they do not affect Weibull modulus. A increment of magnesia content provoke an increase of clusters of magnesia (Fig. 6c and d) with associated porosity that decreases reliability. They produce an amplified stress tension that provoke the random crack of sample when a load is applied, but the obtained Weibull modulus is similar or higher than advanced ceramics and some sintered steels [19]. The best estimator on Weibull modulus is again $F_j = (j-0.5)/n$, more suitable for an asymmetric distribution. Correlation coefficients do not differ

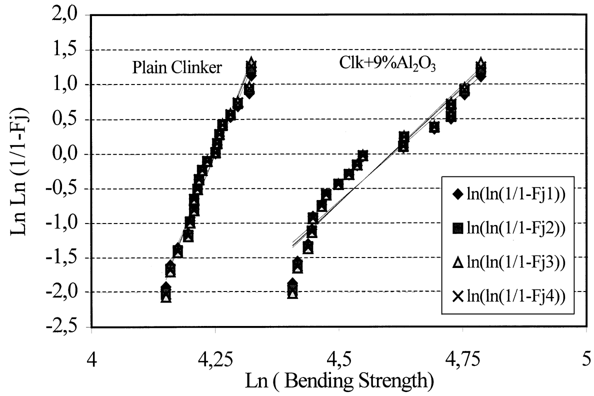

Fig. 8. Weibull plots of plain clinker and material with 9% of alumina.

between the estimators for 3 and 9% of magnesia, while for medium addition the best adjustment is with $F_j=j/(n+1)$, employed for a gaussian distribution [1]. In these materials, reliability is affected by the distribution of magnesia particles and non coherent cluster formation.

The addition of silica produces an increase in the bending strength with respect to that of clinker (Fig. 1), due to reactions between the clinker and the reinforcement. The added silica reacts with the Portland clinker producing an increase in rich-silica phases or C_2S (Fig. 7) and a reduction of the amount of porosity (about 1%). However, some segregation appears in belitic areas (Fig. 7d), which does not positively affect on the Weibull modulus. The Weibull modulus slightly decreases with the increasing of silica addition as compared with plain clinker, due to the increment of belitic segregation and associated large size-porosity, with a maximum for 9% silica. This behaviour could be intensified due to a morphological change from more rounded shape to flake shape of the phases (Fig. 7a–c). The most favourable estimator is the same that for the others additions, with good adjustments (Table 4). Morphological changes of phases and isolated large-size porosity have a negative influence on reliability.

Comparing scale constant σ_0 with the experimental average value for bending strength σ_m (Table 4), only material with 9% of alumina shows significant deviation (about 5), while the other oscillates between 2 and 3.5.

Most of these composite materials have Weibull modulus values above 14, as shown in earlier studies with similar materials [15,16,26–28] and comparable values with multiphase sintered alloyed steels [19].

These multiphase materials, very cheap and processed by CIP, present good reliability with reproducibility near advanced materials, although they present a great amount of different phases and impurities due to fabrication process. So, this work can predict different structural applications (e.g. low temperature refractories and construction tiles) on account of their reliability.

5. Conclusions

Approximation is acceptable with all estimators, except for material with 9% of alumina. The best estimator of Weibull modulus is $F_j=(j-0.5)/n$, with good correlation coefficients. Then, is possible the fast evaluation of the mechanical behaviour and reliability of these materials.

The studied additives provide strengthening and hardening, due to the increase of belitic phase (alumina and silica) and due to particle dispersion with magnesia.

Most composite materials present reliability higher than conventional ceramic showing a Weibull modulus above 14, reaching maximum values between 26 and 29.

In the material with 9% of alumina, σ_u can not taken equal to zero due to a existence of a possible survival period that induce a lower brittle behaviour. It is necessary to evaluate the statistical approach to its mechanical properties through other numerical method.

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