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Mechanical Behaviour and Reliability of Sintered Clinker Portland

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Abstract

In the present work, clinker Portland has been used, not as a component of cements manufacture, but like a raw material for making structural ceramics. The manufacturing process for this material includes blending for homogenization followed by cold isostatic pressing and sintering. The sinterability of clinker Portland has been evaluated. Also mechanical properties, microstructure and mechanical reliability through the Weibull modulus, were evaluated in all of the sintered materials.

Portlandschlacke, die normalerweise zur Herstellung von Zement verwendet wird, wurde auf ihre Verwendbarkeit als keramisches Grundmaterial hin untersucht. Der Herstellungsprozeß für dieses Material umfaßt das Mischen zur Homogenisierung sowie das anschließende kaltisostatische Pressen und Sintern. Die Sinterfähigkeit der Portlandschlacke, die mechanischen Eigenschaften, das Mikrogefüge und die mechanische Zuverlässigkeit, bestimmt mittels Weibull-Modulus, wurden für die gesinterten Proben ermittelt.

Dans cette étude, du clinker de type Portland a été utilisé, non pas en tant que composant de l'industrie cimentière, mais comme une matière première pour réaliser des céramiques structurales. Le procédé d'élaboration de ce matériau comprend des étapes de mélange pour homogénéisation, de pressage isostatique à froid et enfin de frittage. L'aptitude au frittage du clinker de type Portland a été évaluée. Ses propriétés mécaniques, sa microstructure ainsi que fiabililité mécanique par le module de Weibull ont été mesurées sur tous les matériaux frittés.

1 Introduction

Clinker Portland is defined[1] as an artificial material obtained from crudes with an appropriate chemical composition through a process called clinkering (calcinating + sintering). Accordingly,[2] clinker Portland is a material composed of at least 70% (by weight) of calcium silicates. The balance must be oxides, usually aluminium oxide and iron oxide. So it can be affirmed that clinker is composed of ceramic materials and as a consequence, it can be defined as a ceramic material. Usually, clinker has been used as a raw material in manufacturing cements and concrete, far from the usual applications of structural ceramics. However, through the present investigation, it can be demonstrated that clinker can be processed as a classical ceramic material using conventional methods related to powder technology and so, it could have another application as a structural ceramic, and not only for cements or concrete.

In order to study this feasibility, this work studies the sinterablity of clinker Portland, its mechanical properties and finally, its mechanical reliability through the Weibull modulus.

2 Experimental Procedure

2.1 Materials

Clinker Portland was delivered from Portland Valderrivas S.A. (Spain) with the following characteristics.

Chemical composition (% weight):

SiO_2	20·24
Al_2O_3	6·09
Fe_2O_3	3·51
MgO	1·11
Sulphates	1·34
CaO (total)	65·72
CaO (free)	1·21
Fire losses (%)	0·14
A/F ratio (flux modulus)	1·47
S/A ratio (siliceous modulus)	2·17
Standard of cal (%)	96·90

The chemical composition, from the point of view of the components, is the following (% by weight):

Tricalcium silicate (C_3S)	63·18
Dicalcium silicate (C_2S)	12·13
Tricalcium aluminate (C_3A)	10·20
Tetracalcium ferrite–aluminate (C_4AF)	10·68

The average particle size of the clinker Portland is 27 μm and 99% of particles are <45 μm. Other physical properties of the clinker are:

Apparent density (g/cm^3)	3·13
Specific surface (cm^2/g)	5 586·54

2.2 Methods

Clinker Portland was cold isostatically pressed (using a wet bag) at 250 MPa. Sintering was carried out in air up to 1420°C, during 30 min. The thermal behaviour of the powders was studied through the sinterability curve (sintered density versus sintering temperature). Density was measured by Archimedes' method.

Mechanical properties evaluated on sintered materials were flexural strength (in three points) and hardness (H_{V1}). Specimens for mechanical characterization were obtained through a high-precision cutting machine of diamond wire of 0·3 mm of diameter until final dimensions close to 3 mm × 3 mm ×12 mm were obtained.

The Weibull modulus of all the studied materials was determined through at least 20 results of the flexural strength test. For determining the Weibull modulus, the failure probability (F_j) was estimated through:

$$F_j = j/(n + 1)$$

where j is the rank of the experiment and n the total number of experiments. Having obtained the estimation of failure probability, a linear regression was adjusted as follows:

$$\ln \ln (1/(1 - F_j)) = A + m \ln \sigma_j$$

where σ_j is the bending strength and m the Weibull modulus.

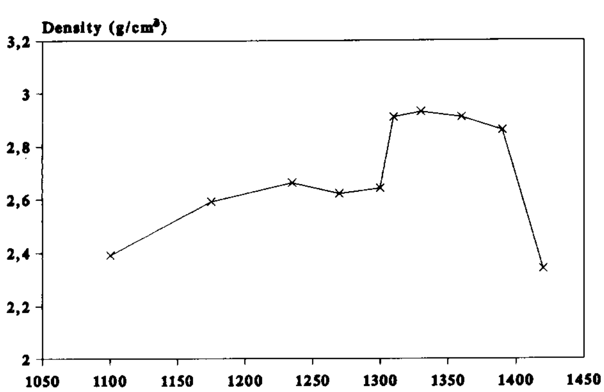

Fig. 1. Thermal answer of sintered clinker Portland.

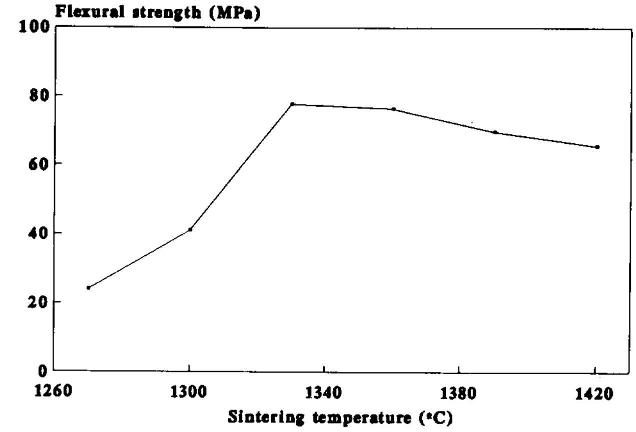

Fig. 2. Flexural strength of sintered clinker Portland.

Finally, a microstructural study of sintered materials was made using optical microscopy.

3 Results

Figure 1 shows the thermal behaviour of clinker Portland studied through the densification produced with increase of sintering temperature. As can be seen, an important increase of density occurs close to the sintering temperature of 1300°C.

Figures 2 and 3 show the results concerning the studied mechanical properties: flexural strength and hardness. In both curves a clear correlation between them can be observed, and also with the sinterability curve.

Figures 4 and 5 display the results concerning the reliability of these materials. In Fig. 4 the complete Weibull modulus slopes and in Fig. 5 the Weibull modulus is shown. Figure 6 overlaps the results of Weibull modulus with flexural strength results including not only the mean values but also the highest value obtained at each temperature. In this figure it can be noted that clinker Portland sintered at 1360°C has a Weibull modulus higher than 15 with some values in flexural strength close to 100 MPa.

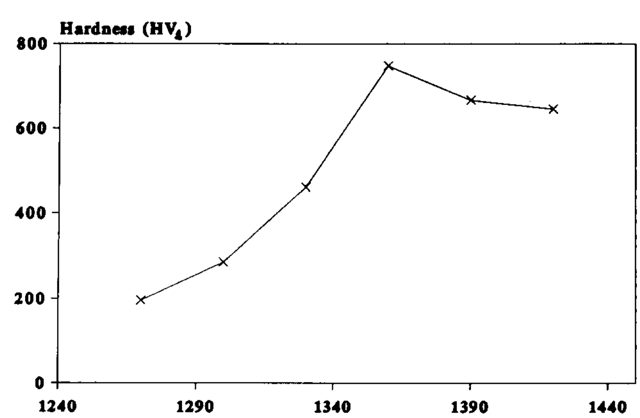

Fig. 3. Hardness of sintered clinker Portland.

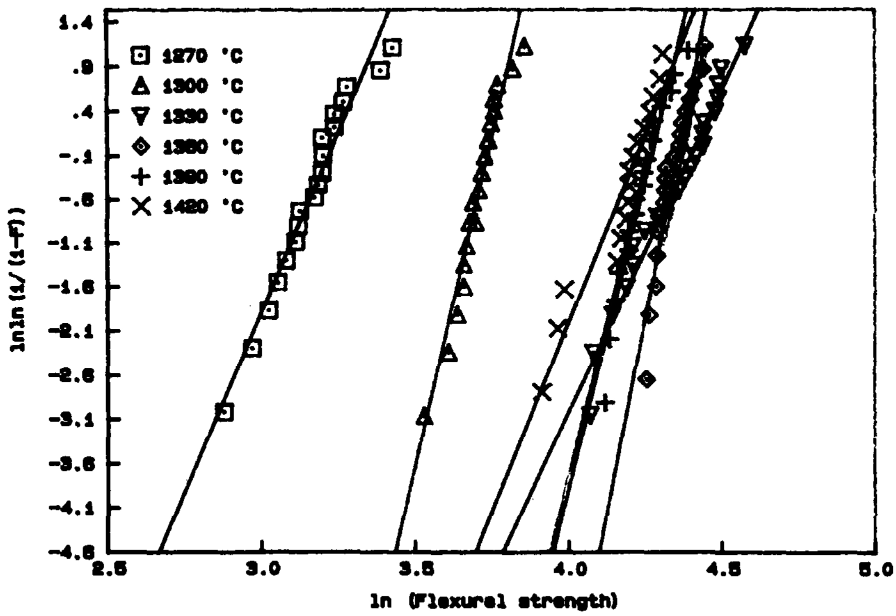

Fig. 4. Weibull modulus slopes of clinker Portland sintered at different temperatures.

Finally, in Fig. 7 some microstructures of the studied sintered materials can be seen.

4 Discussion

Concerning the sintering behaviour, the obtained results are consistent with those expected for these materials: an increase of density with the sintering temperature. Over 1300°C the density of these materials has risen, showing that a further increase in temperature will not improve densification too much, and the high levels of density remain constant up to 1400°C. If the DTA analysis of this material made in a previous work[3] is reviewed, an endothermic peak at 1323°C can be seen which can be due to a possible transient liquid phase at this temperature that confirms the results shown in Fig. 1. These results show that clinker Portland can be processed as a conventional ceramic material.

The mechanical behaviour of clinker Portland is quite congruent with its thermal behaviour, and so, the most densified materials are the better ones from the mechanical point of view. At 1330°C, maximum values for flexural strength close to 100 MPa (mean value close to 75 MPa) have been obtained. From the microstructural study it can be noted that when sintering temperatures higher than 1330°C are used large rounded areas appear in the microstructure. These areas are large pores produced by liquid-phase sintering. It can be seen that the appearance of these gaps in the microstructure coincide with the fall in mechanical properties. The values obtained are slightly higher than the results obtained by other researchers[4] for ceramic matrix composites reinforced with C_2S obtained through a process more complicated (and using higher sintering temperatures) than the one used in the present work. The results also are better than those obtained with SiO_2-based ceramic matrix composites.[5] From the point of view of hardness, clinker sintered at 1369°C has the highest level of hardness. In this work the phenomenon called

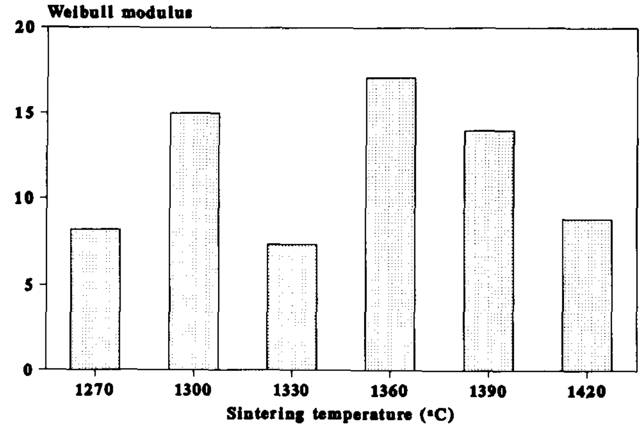

Fig. 5. Weibull modulus of clinker Portland sintered at different temperatures.

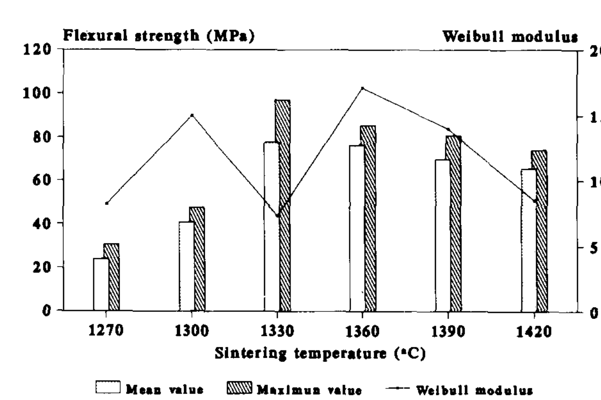

Fig. 6. Mean and maximum values of flexural strength and Weibull modulus of clinker Portland sintered at different temperatures.

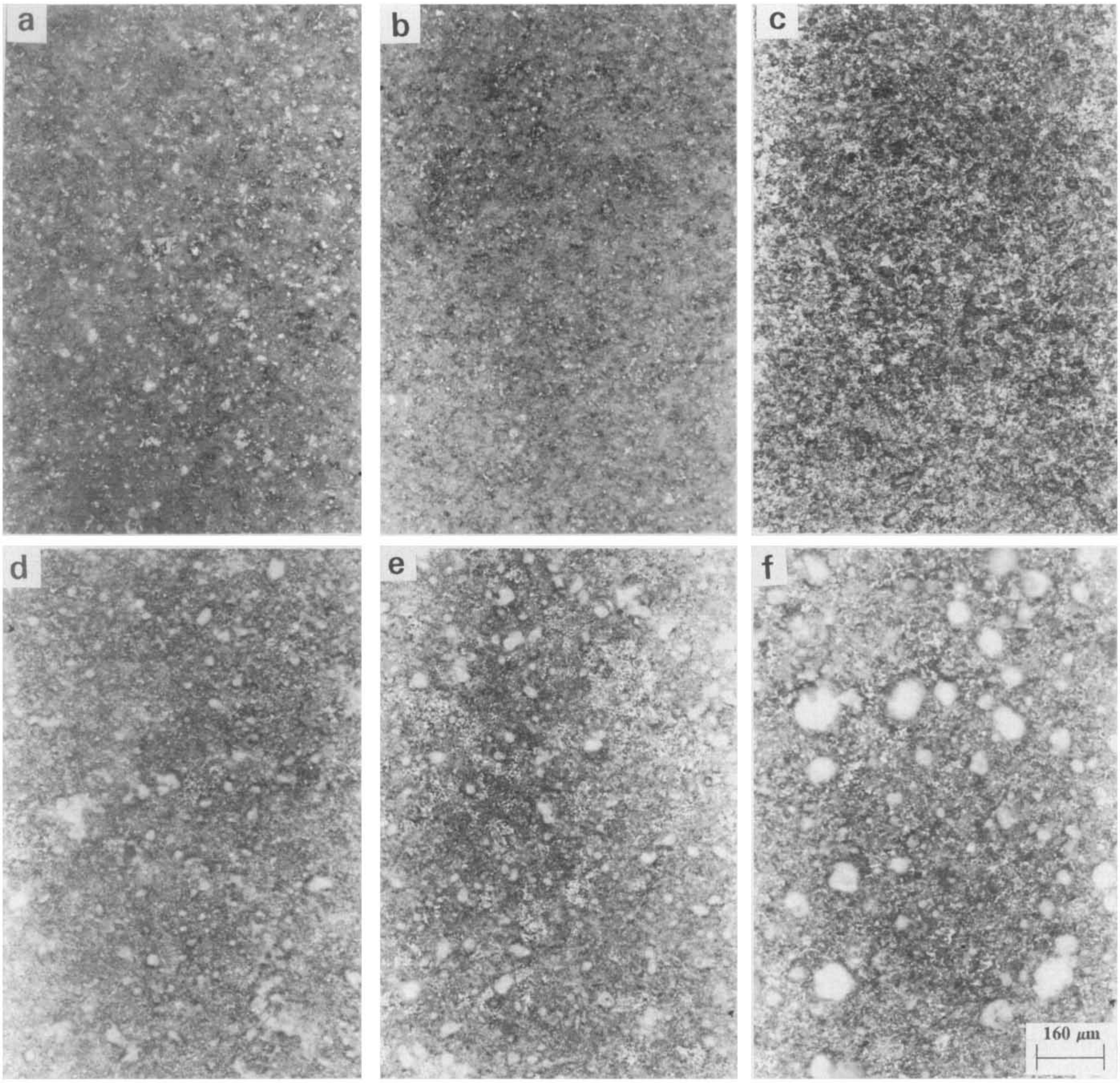

Fig. 7. Microstructure of clinker Portland, sintered at (a) 1270, (b) 1300, (c) 1330, (d) 1360, (e) 1390 and (f) 1410 °C.

'dusting',[6] produced when β-C_2S changes to γ-C_2S at 670°C[7] due to the higher specific volume of the γ-phase, has not appeared, thanks to the presence of other impurities in the C_2S that inhibit this transformation.

From Fig. 6 the results for the reliability of these materials can be discussed. It can be seen that for three different sintering temperatures the value 10 is surpassed and for two sintering conditions the values for flexural strength are close to 80 MPa. It can be emphasized that in these cases the results show a good strength/reliability ratio. The best result is for clinker sintered at 1360°C, that exhibits the highest value for the Weibull modulus (17) with a mean flexural strength result close to 90 MPa. This value of the Weibull modulus is higher than those considered as the maximum for conventional ceramics (around 15)[8] and close to 20, the goal for advanced ceramics.[9,10]

5 Conclusions

The main conclusion of this work is that clinker Portland, as a powder material, conveniently ground, can be used as raw material in manufacturing structural ceramics, using manufacturing methods and conventional techniques used in powder technology. In this way, clinker Portland could be considered as a new family of structural ceramics.

From the point of view of the mechanical properties of the sintered materials studied, considering only the results of flexural strength, the best sintering temperature, in which the highest values for this property are detained, is 1330°C. If the hardness and the reliability of sintered clinker are considered, the best sintering temperature is higher because the best results have been obtained for 1360°C.

Acknowledgements

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