METAL FIBRE-REINFORCED CERAMIC MATERIALS

Alejandro Saburit Llaudis[(1)], Mónica Vicent Cabedo[(1)], Fco. Javier García Ten[(1)], Rubén Niñerola González[(2)], Elkin Martínez Díaz[(2)]

[(1)]Instituto de Tecnología Cerámica (ITC). Asociación de Investigación de las Industrias Cerámicas (AICE). Universitat Jaume I (UJI), Castellón, Spain.

[(2)]Instituto Tecnológico Metalmecánico (AIMME), Paterna (Valencia), Spain.

1. ABSTRACT

The advances in raw materials, technology, and process control have led to upgraded ceramic materials, which nowadays need to provide new properties and functions to address increasingly innovative applications.

However, the brittleness of ceramics in general and of porcelain tile, in particular, constitutes a drawback. The enhancement of toughness in advanced ceramic materials is an important field of research, from which hardly any advances have been transferred to traditional ceramics. One line of work aimed at improving toughness in ceramic materials is the introduction of metal particles, giving rise to metal-ceramic composites with enhanced properties compared to those of a ceramic matrix. However, combining such different materials is no simple matter, as a certain compatibility is required to create interfaces that provide the resulting composites with appropriate mechanical properties. Careful study is therefore needed of the microstructure of the new ceramic matrix composite (CMC).

This work was conducted using a porcelain tile matrix to which different types of metal fibres were added. Different materials mixing and preparation methods were used with a view to obtaining homogeneous test pieces and avoiding the segregation of the materials making up the pieces. Once the materials had been appropriately mixed, this was followed by pressing and sintering in electric laboratory kiln. The resulting composite material was microstructurally characterised.

2. INTRODUCTION

Metal–ceramic composites are homogenously mixed, synthetic materials that are designed to bring together the best properties of each starting material. In the case of composite materials with a ceramic matrix, the qualities of ceramic materials, such as wear resistance, corrosion resistance, and stability at high temperatures, are combined with the properties of metal materials, such as toughness, high thermal and electric conductivity, and thermal shock resistance. In addition, the use of metals as a strengthening phase has also been studied to improve not only the toughness of ceramic materials, but also to offset the loss of properties owing to the presence of porosity after sintering [1],[2].

This work investigated the interface of a porcelain tile matrix reinforced with stainless steel fibres. Two types of fibres and the influence of kiln atmosphere were studied.

3. EXPERIMENTAL PROCEDURE

A typical spray-dried powder for manufacturing porcelain tile was used as a ceramic matrix, together with two types of stainless steel fibre, between 2 and 4 mm long, as reinforcement.

The mixtures, with a 5 wt% strengthening phase, were formed by uniaxial pressing at a pressure of 35 MPa. The test pieces were then thermally treated in an electric laboratory kiln at 1200 °C for 6 minutes using different atmospheres: an oxidising atmosphere (with air) and an inert atmosphere (with nitrogen).

The resulting test pieces were then observed by scanning electron microscopy and their chemical composition was determined by energy dispersive X-ray (EDX) microanalysis.

4. RESULTS

4.1. INFLUENCE OF THE TYPE OF FIBRE

The influence of the type of fibre was studied, first, in air atmosphere. Table 1 details the chemical composition of the two fibres, and figures 1 and 2 show their surface appearance. It may be observed that both fibres consisted of iron as major element and chromium as secondary element. Fibre 1 contained nickel and molybdenum as minor elements, whereas fibre 2 only contained molybdenum as minor element. With regard to the fibre surface, fibre 1 exhibited a smoother surface than fibre 2.

The micrographs of the fired test pieces (Figures 3 and 4) show that metal–ceramic contact (interface) was not homogeneous, as there were areas in which chromium had diffused towards the ceramic matrix, forming an aureole with a lighter colour, and others in which such an aureole was not found. Observation in greater detail of the interface revealed areas that displayed good contact (Figure 7), as well as areas in which the strengthening phase was not in contact with the ceramic matrix (Figure 5 and 6). In this last case it may be observed that chromium had sometimes not diffused into the ceramic matrix (Figure 5) whereas in others, though there was a separation between the phases, chromium diffusion was noted (Figure 6). In the first case, phase separation must have taken place at low temperature (during forming or in the first thermal treatment stages), when the liquid phase had not yet developed in

the ceramic matrix. However, in the second case, it was evident that both phases had been in contact at high temperature and had then separated.

This phase separation might be due to the different thermal expansion of the two materials. Thus, the metal exhibits a greater thermal expansion (α=11–14·10^{-6} °C^{-1}) than the ceramic matrix (α=8.4·10^{-6} °C^{-1}) and during cooling, once the ceramic matrix has become rigid (T<700 °C), the greater shrinkage of the metal could generate a compressive stress on the ceramic matrix, which, if excessive, might lead to separation of the two materials.

Table 1 also details the semi-quantitative analysis of the fibres in an area close to the interface. It shows that, in both cases, the chromium content decreased, which confirms the migration of this element into the ceramic matrix during thermal treatment.

Element	Original		Oxidising atmosphere	
	Fibre 1	Fibre 2	Fibre 1	Fibre 2
Fe	70.5	82.5	71.5	84.5
Cr	19	16.5	18	14.5
Mn	1	---	1	---
Ni	9.5	---	9.5	---
Mo	---	1	---	1

Table 1. *Semi-quantitative analysis (%) of the original and thermally treated fibres.*

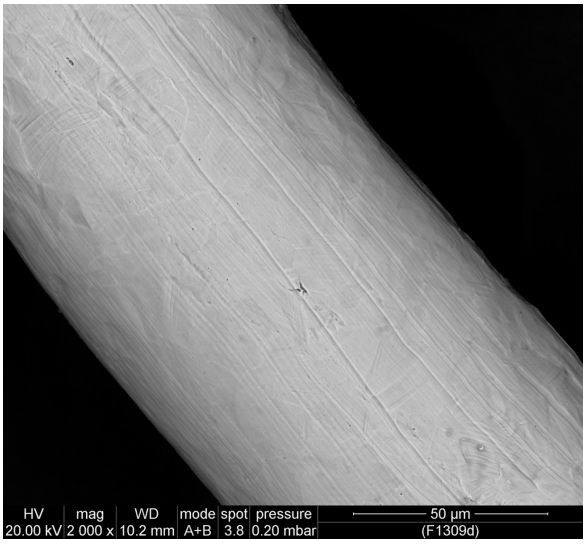

Figure 1. Micrograph of fibre 1.

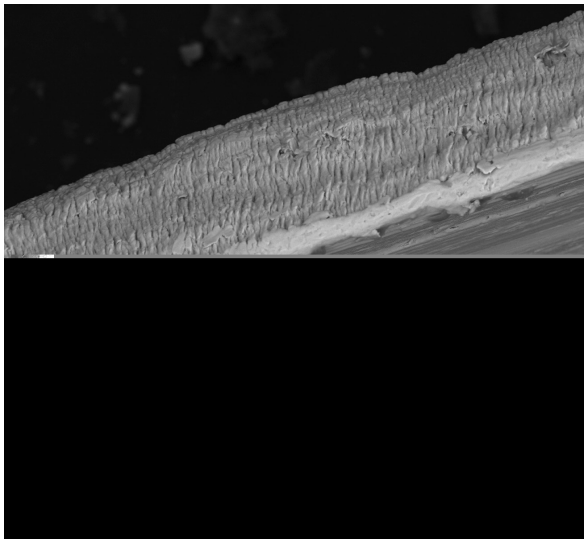

Figure 2. Micrograph of fibre 2.

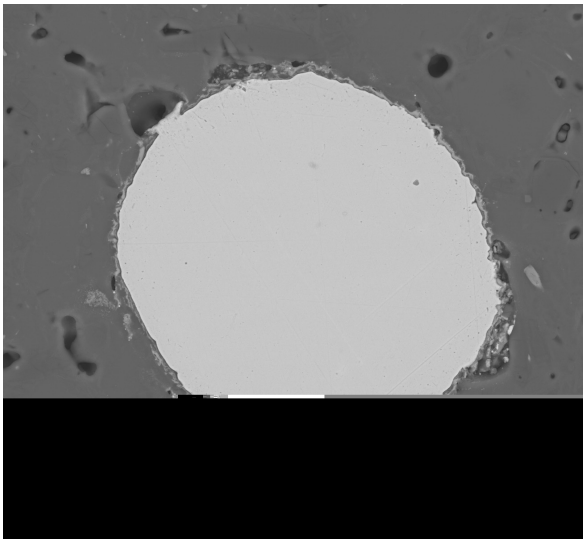

Figure 3. Micrograph of fibre 1 in the ceramic matrix.

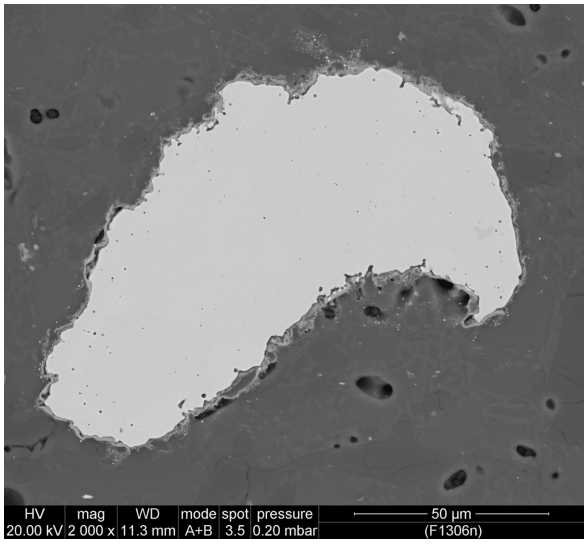

Figure 4. Micrograph of fibre 2 in the ceramic matrix.

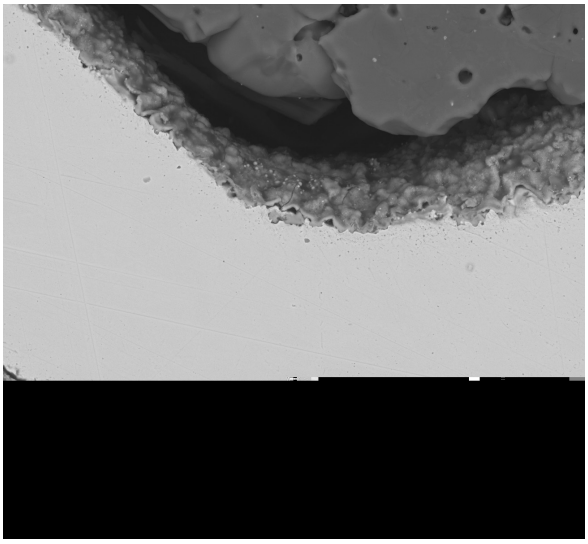

Figure 5. Micrograph of the interface.

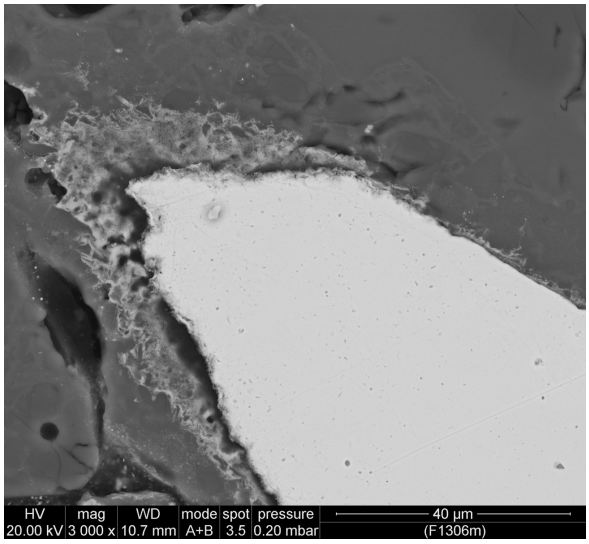

Figure 6. Micrograph of the interface (II).

4.2 INFLUENCE OF KILN ATMOSPHERE

Experiments were conducted in two kiln atmospheres (oxidising and inert), using fibre 2 as strengthening phase. Figure 7 shows the interface microstructure of a piece fired in an oxidising atmosphere, while figure 8 shows that of a piece fired in an inert atmosphere. In both micrographs, the strengthening phase is surrounded by an aureole, in which chromium was detected as foreign element in the ceramic matrix. The aureole exhibited waves owing to the high quantity of liquid phase developed in the ceramic matrix.

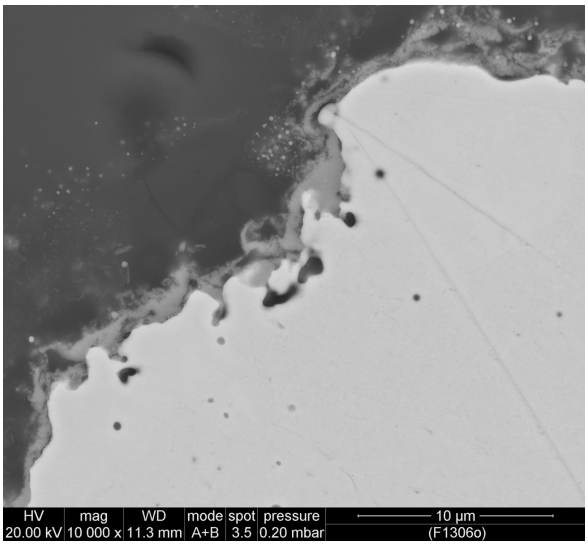

Figure 7. *Appearance of the interface in an oxidising atmosphere.*

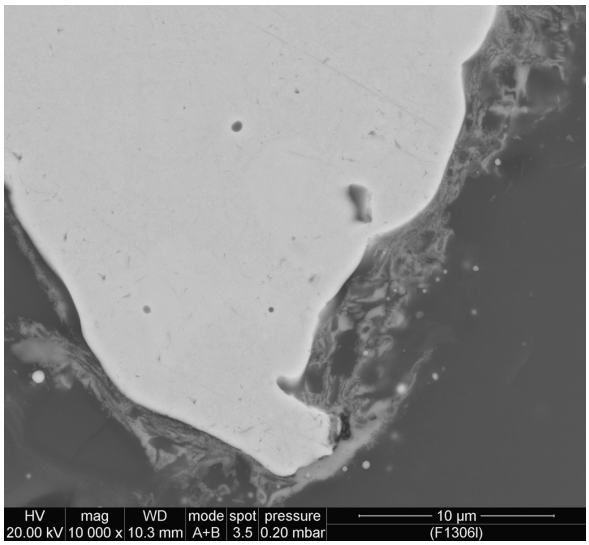

Figure 8. *Appearance of the interface in an inert atmosphere.*

Table 2 details the semi-quantitative analysis of the fibres in an area close to the interface, as well as that of the original fibre. The results show that, when an inert atmosphere was used, the percentage of chromium that migrated from the fibre to the matrix decreased with respect to that which migrated in an oxidising atmosphere. As a result, the fibres underwent greater degradation in an oxidising atmosphere. This is consistent with the literature [3], which recommends performing thermal treatment of these materials in a reducing or an inert atmosphere to avoid oxidation of the metal phase.

Element	Original fibre	In an oxidising atmosphere	In an inert atmosphere
Fe	82.5	86.5	84.5
Cr	16.5	12.5	14.5
Mo	1	1	1

Table 2. *Semi-quantitative analysis (%) of fibre 2 before and after thermal treatment.*

5. ACKNOWLEDGEMENTS

The study was conducted thanks to a project co-funded by IVACE and the European Regional Development Fund (ERDF), within the ERDF Operational Programme of the Valencia Region 2014–2020.

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