

## **TENSILE STRAIN HARDENING OF A METAKAOLIN BASED FIBRE REINFORCED COMPOSITE**

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### **ABSTRACT**

Portland cement concrete is the most used building material in the world. However, its manufacture is energy-intensive and it is susceptible to harsh environments. Alternative binder systems without ordinary Portland cement, such as geopolymers or alkali-activated materials, are recently new in the Civil Engineered world. These alternative binder systems seek, among other characteristics, improved durability and environmental efficiency. The attaining of strain hardening and multiple cracking typical of Strain Hardening Cementitious Composites (SHCC) using these alternative binder systems is very attractive from a conceptual point of view, since additional endurance to certain harsh or extreme environments, as well as enhanced durability, are usually expected as two of the main outcomes. In the present work, the behaviour of two different composites was studied: an existing Engineered Cementitious Composite (ECC) and a new composite based on an alternative binder prepared with metakaolin. Polyvinyl alcohol (PVA) fibres were used in both materials. A series of experiments, including compressive and direct tensile testing were carried out to characterize and compare the mechanical properties of both materials. The results showed that the alternative binder composite, when subjected to uniaxial tension, developed multiple cracks at steadily increasing tensile stress and strain, which is also typical of ECCs showing strain hardening behaviour. The development of fibre reinforced geopolymer or alkali-activated materials showing strain hardening ability in tension may still be considered as a novel research topic, with great potential for creating new and interesting developments for Civil Engineering and structural applications, particularly the ones subjected to harsh environments.

### **1. Introduction**

In general terms, geopolymers can be classified as inorganic polymers synthesised by the reaction of an alkaline solution and an aluminosilicate source, something which was first tried in 1978, by Davidovits [1]. The activator solution, the raw aluminosilicate and curing conditions are the main factors affecting the compressive strength and other mechanical properties of the geopolymers [2]. Strain hardening cementitious composites represent a class of fibre reinforced cementitious composites which have been developed with the aim of withstanding extreme tensile strains at moderate tensile stresses, departing from the typical behaviour of cementitious materials. Due to the complexity of the cracking processes established in these materials under tension, the presence of the fibres and the integral design of the material considering both the mechanical properties of the fibres, of the cementitious matrix and of the fibre-matrix interaction are essential to the attainment of the so-called pseudo-strain hardening in tension [3] [4]. The use of fibre reinforcement based on discrete microfibers distributed evenly in the matrix using geopolymers or alkali-activated binders was already investigated by other researchers. As an example, despite the high brittleness of the so-called AAS (Alkali-Activated Slag) mortar, strain hardening and high tensile ductility using polyvinyl alcohol (PVA) fibres as reinforcement was achieved by Lee et al [5]. From the test results they concluded that it was possible to obtain an ultimate tensile strain as high as 4.7%. The compressive strength ranged between 15 and 35 MPa. The attaining of strain hardening and multiple cracking typical of ECC materials using these alternative binder systems is very attractive from a conceptual point of view, since additional resistance to harsh environments and enhanced durability are expected as main outcome. This work presents the study that was carried out to develop a strain hardening cementitious composite resorting to an alternative binder system based on metakaolin and not containing Portland cement.

## 2. Experimental procedure

**Materials and procedures.** In the present work, two different compositions have been developed: an ECC mixture and an alternative mixture.

The composition of the ECC mixture is presented in **Erro! A origem da referência não foi encontrada.** The PVA fibres were added at a volume fraction of 2% to both mixtures. The PVA fibres used are 8 mm long and the diameter is 40  $\mu\text{m}$ , showing a tensile strength of 1600 MPa. The materials used to produce the geopolymer were the following (see **Erro! A origem da referência não foi encontrada.**): sand, metakaolin, activator prepared with sodium hydroxide and sodium silicate, VMA (Viscosity Modifying Agent) and super-plasticizer. The activator was prepared using a weight proportion of one third of sodium hydroxide and two thirds of sodium silicate or waterglass. In this research the metakaolin used had a maximum grain size below 5  $\mu\text{m}$ , the specific gravity of 2.5  $\text{g}/\text{cm}^3$  and the specific surface of 10150  $\text{cm}^2/\text{g}$ .

Table 1- ECC composition

<i>Materials</i>	M (by weight)
<i>Cement</i>	1
<i>Fly Ash</i>	2
<i>Sand</i>	0,35
<i>Filler</i>	0,35
<i>Tap Water</i>	0,8
<i>SP Sika 3002HE</i>	0,01
<i>VMA</i>	0,003
<i>PVA Fibre</i>	2% (by volume)

Table 2- Metakaolin based geopolymer composition

<i>Materials</i>	M (by weight)
<i>Metakoalin</i>	1
<i>Sand</i>	0,13
<i>Sodium Hidroxide</i>	0,22
<i>Sodium Silicate</i>	0,44
<i>SP Sika 3002HE</i>	0,01
<i>VMA</i>	0,003
<i>PVA Fibres</i>	2 % (by volume)

In the case of the geopolymeric mixture, a mixer with 3L capacity was used and the following procedure was adopted: firstly all the materials were collected and weighted. Solid ingredients, including metakaolin, sand and VMA were placed inside the bowl and mixed for one minute in slow speed. Subsequently, 90% of the activator and the super-plasticizer were added into the bowl while mixing for another 2 minutes. The mixer was restarted and all the fibres were then added to the mortar and mixed until the fibres were homogeneously distributed, for about 2 minutes. Then the remainder activator was added into the bowl and mixed for another 2 minutes. The ECC mixture was prepared using a similar mixing procedure: the solid ingredients were put in the bowl and mixed for one minute. Water and super-plasticizer were then added into the bowl and mixed for 5 minutes more. Then all the fibres were added into the mortar and mixed until the fibres became well distributed (about 2 minutes more). The fresh properties were studied before and after the fibres were added to the mortar according to EN 1015-3. The final diameter in two orthogonal directions ( $dxd$ ) of the spread mixtures was measured. The time taken by the mixture to reach a spread diameter of 20 cm was not measured because this spread diameter was not reached by any mixture. The specimens were cast in different moulds and then vibrated in the shaking table, in order to reduce the air entrapped by the mixture, and then covered with cling film. After one day of curing at 80° and relative humidity (RH) RH=0% the metakaolin specimens were demoulded and kept in dry atmosphere conditions, at about 20° +/- 2°C. The ECC specimens were kept in the mould at room temperature for 24 hours and then demoulded and moved into the climatic chamber for 28 days. The temperature and RH in the climatic chamber were, respectively, 20°C and 60%.

Table 3- Fresh properties of both mixtures

	<i>ECC</i>	<i>GP</i>
<i>dxd (cm)</i>	19x19	15x15
<i>dxd (cm) (with fibres)</i>	15x17	13x12

### Compression testing.

Compression tests were carried out using cubes measuring 50 x 50 x 50 mm<sup>3</sup>. In this study, one actuator with a 200 kN load cell and one LVDT (Linear Variable Differential Transformer) were used. The compressive tests were carried out at a constant compressive displacement rate of 0,02 mm/sec.

**Tensile testing.** The specimens for direct tension testing and for characterizing the tensile stress-strain responses were cast using dogbone-shaped moulds. These moulds were 20 mm thick, 370 mm long and 100 mm wide (the straight central part was 50 mm wide and 110 mm long). One actuator with a 200 kN load cell, two grips (one grip was connected with the actuator and the other was fixed in the reaction frame) and 3 LVDT's were used in the test set-up. During testing the specimens were subjected to a constant tensile displacement rate of 0,010 mm/s.

## 3. Results and discussion

### Compression Behaviour.

Three specimens of each mixture were tested. Fig. 1 shows the compressive stress vs strain response representing the compressive behaviour of both mixtures. The compressive results obtained for the mixtures are presented in Table 4. The results show that the ECC mixture achieved higher compression stresses when compared with the geopolymeric mortar.

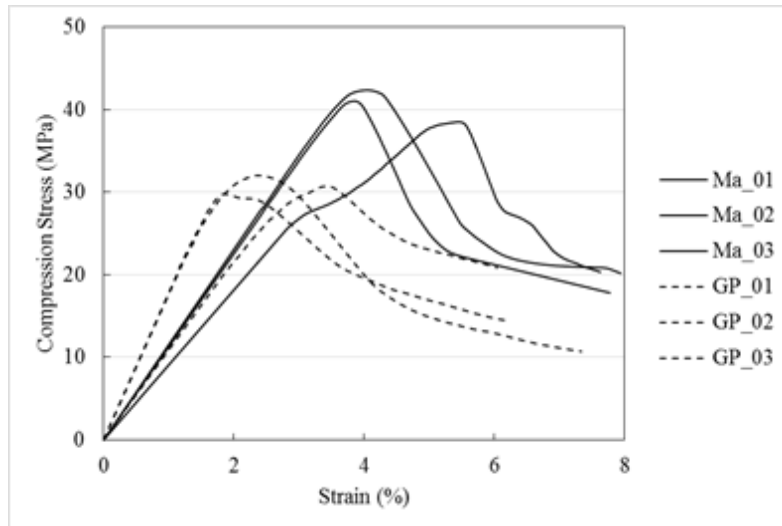


Fig. 1- Compressive behaviour of both mixtures

Table 4- Compression test results

<i>Mixture</i>	<i>Specimen</i>	<i>Compressive strength (Mpa)</i>	<i>Average (Mpa)</i>	<i>Standard Deviation (Mpa)</i>	<i>Coefficient of Variation (%)</i>
<b>GP</b>	1	30,70	30,82	0,95	3
	2	32,04			
	3	29,72			
<b>ECC</b>	1	38,52	40,64	1,60	4
	2	42,37			
	3	41,04			

### Tensile Behaviour.

Three dogbone-shaped specimens of each mixture were tested. The responses represented in Fig. 2 were obtained with the ECC mixture and show high stiffness before the formation of the first crack. After the first crack formation, all the specimens exhibited the appearance of several micro-cracks at increasing tensile stresses, reaching ultimate tensile stresses that ranged between 3,9 and 4 MPa. The specimens tested reached ultimate tensile strains between 3,4% and 3,8%.

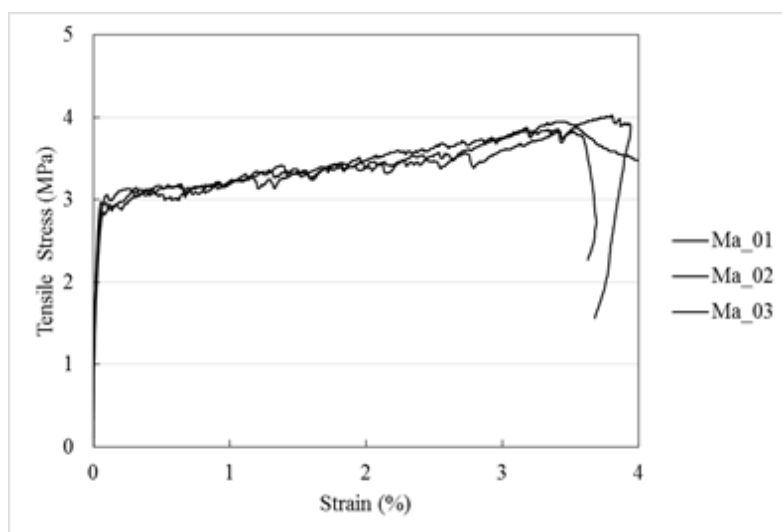


Fig. 2- Tensile response of the ECC mixture

Regarding the geopolymeric specimens, the tensile responses obtained revealed the development of multiple cracks at steadily increasing tensile stresses and strains, somewhat resembling the tensile strain-hardening behaviour which is typical of ECC. Similarly, high stiffness was observed until the first crack was developed, and after, multiple cracks were formed until the maximum value of tensile strain was achieved, see Fig. 3.

The crack pattern obtained in the metakaolin based geopolymer is shown in Fig. 4. The white color of the matrix does not facilitate the visualization of all the cracks formed, except the wider ones which eventually lead to the failure of the specimen. However, a closer look allows the visual identification of several narrow and closely spaced cracks. This feature is promising regarding the improvement of the durability of this material.

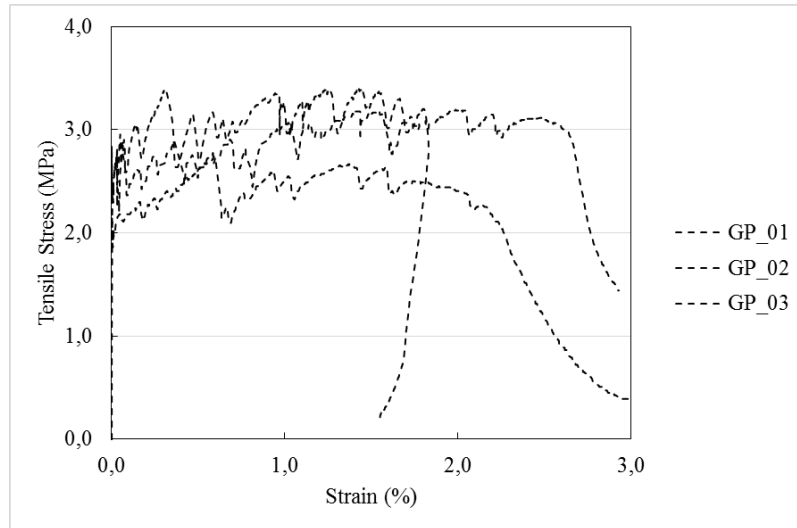


Fig. 3-Geopolymer tensile response



Fig. 4- Crack pattern of a geopolymeric and ECC specimen

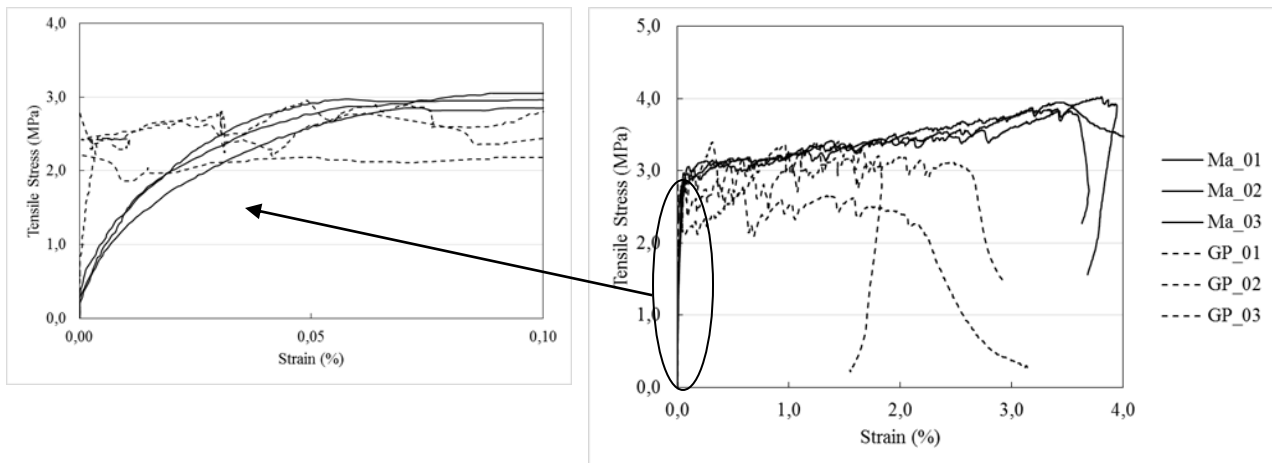


Fig. 5- Comparison of tensile results of both mixtures

Although both materials showed a tensile strain hardening response, as shown in Fig. 5, the behavior of both composites is quite different: both showed high stiffness until the formation of the first crack but resulted in different strain hardening behaviors that led to distinct crack patterns. The crack pattern of the ECC material showed more cracks with a small width when compared with the metakaolin based composite, showed in Fig. 4. The smaller crack widths resulted in a smoother strain hardening phase and high ductility in the ECC tensile response. The crack opening behavior obtained in the geopolymeric specimens was associated with a greater scatter and a less reliable strain hardening behavior, which reveals the need to further optimize the micromechanical design of the composite. The maximum tensile stress reached was 3.4 MPa and the maximum tensile strain was 1.8%.

#### 4. Conclusions

ECC materials, which typically contain ordinary Portland cement in their composition, have special behaviour when subjected to uniaxial loading, which is characterized by a moderate tensile hardening for a substantial increase in tensile strain while multiple cracks form. In general, this type of tensile behaviour is considered as very attractive for a wide multiplicity of applications, including the ones needing improved damage tolerance and enhanced durability. The main goal of this study was to try to obtain the same type of behaviour but with an alternative binder system, in this case a metakaolin based geopolymer. That behaviour was somewhat reached by resorting to a metakaolin based geopolymeric matrix. The geopolymeric composite can be considered as a promising solution due to the potentially higher endurance to certain types of harsh environments. After overcoming the technical challenge of designing these materials to perform satisfactorily in tension, the next challenge will be to determine their real potential in terms of durability, mainly considering especially harsh environments.

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## References

- [1] N. Saidi, B. Samet e S. Baklouti, “Effect of Composition on Structure and Mechanical Properties of Metakaolin Based PSS-Geopolymer,” *International Journal Of Materials Science*, 2013.
- [2] Z. Li, Z. Ding e Y. Zhang, “DEVELOPMENT OF SUSTAINABLE CEMENTITIOUS,” Department of Civil Engineering, Hong Kong University of Science and Technology, Hong Kong, 2004.
- [3] E. B. Pereira, G. Fischer e J. A. Barros, “Description of near-tip fracture processes in strain hardening cementitious composites using image-based analysis and the compact tension test,” em *Proceedings of the 8th International Conference on Fracture Mechanics of Concrete and Concrete Structures, FraMCoS 2013*, pp. 1037-1048., 2013.
- [4] E. B. Pereira, G. Fischer e J. A. Barros, Hybrid fiber reinforcement and crack formation in cementitious composite materials.(2012) RILEM Bookseries, 2, pp. 535-542.
- [5] B. Y. Lee, C.-G. Cho, H.-J. Lim, J.-K. Song, K.-H. Yang e V. C. Li, “Strain hardening fiber reinforced alkali-activated mortar – A feasibility study,” *Construction and Building Materials*, 2012.
- [6] D. Bondar, C. J. Lynsdale, N. B. Milestone, N. Hassani e A. A. Ramezaniapour, “Enhineering Properties of Alkali Activated Natural Pozzolan Concrete,” em *International Conference on Sustainable Construction Materials and Technologies*.