






# Novel Blast and Fire Resistant Composite Materials: Design and Preliminary Results

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**Abstract.** Today, very few materials are available for the protection of high-rise buildings and large constructions against blast and impact and practically, they belong to a new class of concrete, the Ultra High Performance Fiber Reinforced Concretes (UHPFRCs). UHPFRC exhibits high compressive and flexural strengths, along with high energy absorption capacity; however, it suffers from limited fire resistance, a property of critical importance as blast events are usually accompanied by fires. On the other hand, the traditional fire-resistant construction materials are practically unable to protect the structures against explosions, which is also critical, since fires in constructions are often accompanied by blast events. The present paper deals with a novel Hybrid Laminate Material (HLM), which is designed, developed and validated in the frame of an extended research, focused on the elimination of the above-mentioned risks. HLM consists of an outer layer of an optimized impact/blast-resistant UHPFRC and a superficial inner layer of a fire-resistant geopolymeric concrete (FRGC). Particularly, FRGC is based on the valorization of industrial wastes and by-products, in order to decrease the production cost and enhance sustainability and circular economy principles for the new HLM, in addition to the environmental and economic benefits of the geopolymerization technology. In this paper, the design of the novel HLM construction material is presented and the results of exploratory tests on the applicability of selected industrial waste and by-products for the FRGC development are given and the thermomechanical performance of the FRGC prepared by selected waste materials is discussed. According to the results, the fire-resistant layer of HLM can be based on waste materials providing the final product with viability and sustainability, without falling short of the properties required for such material.

**Keywords:** UHPFRC · Blast Resistance · Impact Resistance · Geopolymer · Fireproof Materials · Secondary Resources

## 1 Introduction

The recent trends in the construction industry for high-rise buildings and large constructions like super-span bridges require the use of materials with excellent safety, functionality and durability. Since such structures could be potential targets of terror attacks, a comprehensive effort is essential to protect them by minimizing the effect of impact and explosion through a rational selection of materials and design of structures. Therefore, the materials selected for these structures should also have, except for the ultra-high mechanical properties, blast- and impact-resistance capacity.

Today, very few materials of high performance are suitable to mitigate the impact of blast damages to structure or material. Practically, they comprise a new class of concrete, the Ultra High Performance Fiber Reinforced Concrete (UHPFRC), which is a combination of high strength concrete and fibers. UHPFRC was developed in 1990's in France and is characterized by ultra-high mechanical strengths and energy absorption capacity. It is defined as cementitious composite with high compressive strength, which is exceeding 150 MPa and high tensile strength, which may reach 15 MPa, good strain-hardening behavior under uniaxial tension and blast-resistance capacity [1–4]. The use of fibers (mainly steel) in the UHPFRC serves to achieve ductile behavior under tension, as well as to dispense with the use of conventional active or passive reinforcement. The incorporation of relatively high amounts of steel fibers in the UHPFRC, along with the high cost of different constituents, can increase its production cost to about 20–30 times over the cost of conventional concrete mixes [1]. During the last years, the use of supplementary cementitious materials, such as fly ash, slag and silica fume for replacing part of cement in UHPFRC, as well as the lowering of curing temperature resulted in the reduction of its production cost. Therefore, UHPFRC has attracted increasing interest over the past two decades for use in civil and military buildings and structures, as well as in aerospace and marine applications, due to its superior mechanical properties and excellent blast and ballistic impact performance [1, 2, 5]. However, the limited fire resistance of UHPFRC is an important drawback for its wide application.

Like all types of concrete, the UHPFRC is non-combustible. However, due to its extremely dense microstructure and impermeability, it is susceptible to cracks and explosive spalling in case of fire, which is one of the most dangerous mechanisms for damage of concrete structures [6]. Spalling of UHPFRC is mainly caused by the vapor pressure developed in the pores of the material due to the dehydration of hydrate phases, as well as by thermal stresses and can lead to deterioration of the concrete structure [6, 7]. To overcome this problem, polypropylene fibers are included in UHPFRC mixes, which create capillary pores at temperatures above 160 °C, when they melt and burn [6, 8, 9]. Although the addition of polypropylene fibers can improve the fire-resistance properties of UHPFRC, their industrial application remains under investigation. The mix proportion and geometry of the polypropylene fibers comprise important issues, as excessive fiber dosage can lead to a significant reduction in residual strength and elastic modulus values under elevated temperatures and thus, seriously jeopardizes the safety of UHPFRC structures [6].

Therefore, improving the fire resistance of UHPFRC, while retaining ultra-high mechanical strength and excellent behavior against blast and ballistic impact, is more than necessary nowadays, given the basic requirements of modern constructions and

the increased terrorist threat. In this direction, an extended research is being conducted that aims at the design, development and validation of a novel Hybrid Laminate Material (HLM), consisting of an optimized blast- and impact-resistant UHPFRC that will serve as the outer layer of HLM and a fire-resistant geopolymeric concrete (FRGC) that will form the inner layer of the composite. The FRGC will be based on the utilization of industrial wastes and by-products, thus decreasing the production cost and enhancing sustainability and the circular economy principles. Furthermore, the low energy and CO<sub>2</sub> emissions technology of geopolymerization that is used for the FRGC production will provide HLM with additional environmental and economic benefits. In this paper, the design of the novel HLM construction material is presented and the results of exploratory tests on the applicability of different industrial wastes and by-products for the development of its fire-resistant layer are given. In addition, the results of a preliminary study on the thermomechanical performance of the FRGC prepared by selected waste materials are discussed.

## **2 The Novel Hybrid Laminate Material**

### **2.1 Conceptual Description**

The novel HLM aspires to fill in the gap that exists in the construction industry for materials that serve both, the passive fire protection of structures and their protection against explosion and impacts. Till today, few materials have been developed for the fire or the blast and impact protection of high-rise buildings and large industrial and military infrastructures. In fact, none of them can combine the advantages of a fire-resistant material with the high performance of UHPFRC against dynamic and violent explosions and impacts. UHPFRC's lack of fire resistance leads to explosive spalling phenomena when exposed at temperatures higher than 400 °C [10], resulting in the damage of structures and finally, in their collapse. Given that the spalling depth and velocity increase dramatically with the increase of the material's compressive strength [11], it is certain that a construction made by UHPFRC will collapse in a blast or impact event accompanied by fire. Accordingly, the lack of blast and impact resistance from the currently available fireproof materials makes it almost certain that a structure will collapse in the case of accompanied explosions. The absence of any reinforcement, along with the low compressive strength of today's fire-resistant materials (<8 MPa) [10] are the main factors that inhibit the development of the required blast resistance. In addition to the above mentioned technological shortcomings, the high cost and the increased environmental footprint of UHPFRC and the commercial fire-resistant materials render unviable their wide industrial application.

### **2.2 Design and Development**

The HLM is a composite material that aims at providing high-rise buildings and large constructions with ultra-high mechanical performance, as well as with two important functionalities: resistance against explosions and impacts and passive fire protection. This composite material is based on the concept of protective and sacrificial materials

for the safety of constructions. Particularly, it will comprise an outer layer of UHPFRC that will be capable to withstand the short and powerful blast and impact loadings and an inner layer of a fire-resistant geopolymer concrete (FRGC) that will protect the integrity of constructions against a subsequent fire. The UHPFRC layer of HLM will be designed to achieve high mechanical strengths (i.e. at least 150 MPa compressive and 20 MPa flexural), and increased energy absorption capacity to prevent its catastrophic failure under blast and impact loads. The FRGC layer accordingly, will be designed to withstand the typical heat load of building and construction elements that is determined by the ISO 834 standard fire curve (Cellulosic curve). The bonding of the two UHPFRC and FRGC layers will be designed to achieve minimum adhesion strength of 2 MPa.

Given the extended research and significant technological achievements made in recent years in the field of blast and impact resistance of UHPFRC [1–5, 12, 13], FRGC is more challenging for the design and development of HLM. FRGC will be based on the innovative technology of geopolymerization, which achieves to transform a wide range of industrial waste and by-products into unique materials with excellent physical, mechanical, thermal and chemical properties, through the alkali-activation process at temperatures below 100 °C and ambient pressure [14]. For the production of FRGC, a variety of secondary aluminosilicate resources are selected to be investigated, including industrial waste and by-products generated in huge quantities every year worldwide, such as fly ashes of power production, slags of ferrous industry, waste glass and construction and demolition wastes. The utilization of waste materials in the production of FRGC, along with the low energy demand and carbon footprint of the geopolymerization technology, will provide the final HLM with viability and sustainability. As FRGC will serve for the passive fire protection of structures, specific properties related to this application will be investigated for this material, like fire resistance, thermal stability at high temperatures, compressive strength before and after fire exposure, apparent density and shrinkage. The mechanical strength at ambient conditions, apparent density and shrinkage are the first selection criteria of the most promising mix designs of raw materials for the FRGC development. The optimization of materials will take place at a second level, through the testing of their thermal stability and residual strength after exposure at temperatures up to 1050 °C and the evaluation of their fire resistance and load-bearing capacity when exposed to fire, according to the ISO 834 fire curve. Finally, the optimum FRGC will be validated on a lab-scale, in combination with the UHPFRC layered material of the novel Hybrid Laminate Material.

Since the chemistry of geopolymer systems is based on silicon, aluminum, calcium and alkali (Na or K) oxides, the fire resistance of FRGC can be designed through the engineering of forming refractory phases at high temperatures up to 1050 °C, which correspond to a cellulosic fire (ISO 834 fire curve). In order to achieve the desired refractory phases, the composition of FRGC will be designed based on the thermodynamics of ternary and quaternary phase diagrams of the FRGC main constituents ( $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{Na}_2\text{O}$  or  $\text{K}_2\text{O}$  and  $\text{CaO}$ ). Such phase diagrams are used to predict the properties of liquid slag in the different metallurgical processes [15]. The most important criterion for the FRGC composition design will be the solidus and liquidus temperatures of the different phases formed in the geopolymer matrix at temperatures up to 1050 °C. It is important for these phases to have high solidus and even higher liquidus temperatures (higher than

1200–1300 °C) and an essential gap between them, in order to safeguard the structures in cellulosic fire events, preventing their sudden collapse. The phase diagrams  $\text{Al}_2\text{O}_3\text{-Na}_2\text{O-SiO}_2$  and  $\text{Al}_2\text{O}_3\text{-K}_2\text{O-SiO}_2$  and  $\text{Al}_2\text{O}_3\text{-CaO-Na}_2\text{O-SiO}_2$  are the most important for the composition design of FRGC. In particular, the optimization of FRGC composition will be driven towards stable constituents of the aforementioned thermodynamic systems at temperatures higher than 1200–1300 °C, like leucite ( $\text{KAlSi}_2\text{O}_6$ ), which is stable up to almost 1600 °C.

### 3 Preliminary Results of the FRGC Development

#### 3.1 Evaluation of Raw Materials

A preliminary assessment of the different waste materials suitability for the development of the FRGC was based on the preparation of geopolymers by these raw materials, under different synthesis conditions. The shrinkage, density and compressive strength of the prepared geopolymers were evaluated as basic properties for the application of FRGC in the novel HLM. For this reason, cubic specimens of geopolymers were prepared and cured at ambient conditions for 7 days. The chemical composition of the used waste materials is summarized in Table 1. It concerns ground granulated blast-furnace slag (GGBFS), which is by-product of primary iron production, fly ash from power plants (FA), waste bricks and ceramic tiles (WB and WCT, respectively) from Construction and Demolition Waste (CDW) and waste glass (WG). As seen in Table 1, all the used waste materials are rich in silicon and aluminum, while some of them contain also large amounts of calcium (GGBFS, FA and WB) and iron (FA, WB and WCT). Among the used waste materials, GGBFS and WG are almost XRD amorphous, while FA and WCT contain a significant amount of an XRD amorphous Al-Si phase. On the contrary, WB is totally crystalline; however, it contains Al-Si phases easily dissolved in alkaline solutions, like feldspars.

**Table 1.** Chemical composition of the solid raw materials.

| Chemical Species        | GGBFS      | FA    | WB    | WCT   | WG    |
|-------------------------|------------|-------|-------|-------|-------|
|                         | mass, % wt |       |       |       |       |
| $\text{SiO}_2$          | 36.9       | 47.42 | 53.57 | 62.4  | 76.56 |
| $\text{Al}_2\text{O}_3$ | 8.81       | 19.16 | 14.33 | 14.68 | 10.34 |
| CaO                     | 44.75      | 16.85 | 7.71  | 1.48  |       |
| FeO                     | 0.41       | 7.64  | 10.19 | 8.58  | 0.24  |
| $\text{K}_2\text{O}$    | 0.32       | 1.67  | 3.74  | 3.76  | 0.92  |
| MgO                     | 7.04       | 2.68  | 4.07  | 3.68  | 3.96  |
| $\text{Na}_2\text{O}$   |            | 0.61  | 0.66  | 0.98  | 8.76  |
| $\text{TiO}_2$          | 0.59       | 1.25  | 1.46  |       |       |

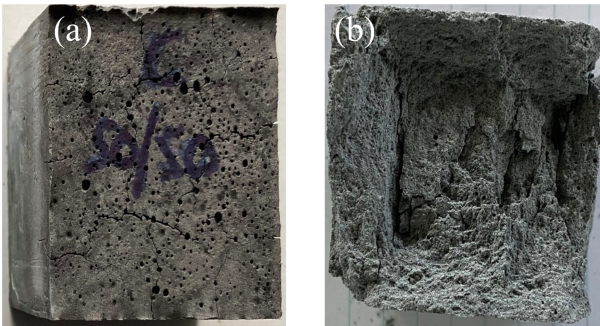
Except for the solid raw materials, solutions of alkali hydroxides, NaOH and KOH prepared in the laboratory, and commercially available alkali silicate solutions, Na<sub>2</sub>SiO<sub>3</sub> (MERCK; molar ratio Na/Si = 0.57) and K<sub>2</sub>SiO<sub>3</sub> (MULTIPLASS S.A.; molar ratio K/Si = 0.65), were also used for the preparation of the geopolymers. Specifically, NaOH was combined with Na<sub>2</sub>SiO<sub>3</sub> (Na-based system), while KOH was combined with K<sub>2</sub>SiO<sub>3</sub> solution (K-based system). Silica fume was also used as additive in certain cases. Table 2 presents the mix designs of the geopolymers prepared in this study, along with the studied properties. The volume ratio of alkali silicate to alkali hydroxide solutions was kept constant and equal to 0.45 in all the experiments performed. The compressive strength of geopolymers was determined using a 2000 kN electro-hydraulic mechanical testing machine. The apparent density of geopolymer samples was determined as the mass of materials per unit volume. For each studied property, the average of three measurements is given in Table 2. The ratio S/L concerns the proportion of solids to liquids used in the synthesis of each geopolymer.

**Table 2.** Mix designs of geopolymer binders.

| Geopolymer Mix ID   | [Na/KOH]<br>(M) | S/L<br>(g/mL) | Shrinkage<br>(%) | Density<br>(g/cm <sup>3</sup> ) | Compressive<br>Strength (MPa) |
|---------------------|-----------------|---------------|------------------|---------------------------------|-------------------------------|
| FA100- N7           | 7               | 2.8           | 1.82             | 1.41                            | 17.73                         |
| FA90-BFS10-N7       | 7               | 2.8           | 2.42             | 1.43                            | 17.47                         |
| FA70-BFS30-N7       | 7               | 2.8           | 3.80             | 1.60                            | 17.29                         |
| FA50-BFS50-N7       | 7               | 2.8           | 5.30             | 1.71                            | 16.38                         |
| FA90-BFS10-SF1-N7   | 7               | 2.8           | 3.16             | 1.37                            | 18.14                         |
| FA90-BFS10-SF2.5-N7 | 7               | 2.8           | 0.80             | 1.31                            | 13.02                         |
| FA90-BFS10-SF5-N7   | 7               | 2.8           | 0.08             | 1.32                            | 13.82                         |
| FA90-BFS10-SF10-N7  | 7               | 2.8           | 0.04             | 1.31                            | 14.60                         |
| FA90-BFS10-SF15-N7  | 7               | 2.8           | 1.14             | 1.28                            | 11.14                         |
| FA90-BFS10-N7       | 7               | 3.8           | 1.08             | 1.50                            | 25.39                         |
| FA90-BFS10-SF1-N7   | 7               | 3.8           | 1.72             | 1.47                            | 22.02                         |
| FA90-BFS10-SF1-N8   | 8               | 3.8           | 0.62             | 1.55                            | 17.14                         |
| FA90-BFS10-SF1-N10  | 10              | 3.8           | 0.06             | 1.58                            | 16.71                         |
| FA90-BFS10-SF1-K7   | 7               | 3.8           | 0.16             | 1.57                            | 18.17                         |
| FA90-BFS10-SF1-K8   | 8               | 3.8           | 0.98             | 1.56                            | 17.66                         |
| FA90-BFS10-SF1-K10  | 10              | 3.8           | 0.50             | 1.62                            | 12.58                         |
| WB100-KOH8-NWG      | 8               | 2.5           | 0.26             | 1.55                            | 19.25                         |
| WCT100-KOH8-NWG     | 8               | 3.4           | 0.48             | 1.74                            | 33.10                         |

FA = Fly ash; BFS = Ground granulated blast-furnace slag; SF = Silica fume; N = NaOH and Na<sub>2</sub>SiO<sub>3</sub> alkali activator; K = KOH and K<sub>2</sub>SiO<sub>3</sub> alkali activator; KOH = KOH solution; NWG = Na<sub>2</sub>SiO<sub>3</sub>.

According to the results given in Table 2, the geopolymers based on WB, WCT and FA with the addition of GGBFS 10% wt. Presented the higher compressive strength. Especially that one based on the WCT raw material developed the highest compressive strength. The increasing replacement of FA by GGBFS did not improve the compressive strength of geopolymers at all, while it affected slightly their shrinkage and increased their density. In addition, the materials with the highest replacement of FA (50% wt.) presented an extensive surface cracking after curing (Fig. 1a). The addition of SF in FA/GGBFS geopolymers improved drastically their density and resulted in almost negligible shrinkage, without enhancing their compressive strength. The addition of SF favored the amorphous gel phase formation in the matrix of geopolymers, which resulted in the materials swelling (Fig. 1b). The increase of SF addition intensified the materials swelling and the formation of internal voids (Fig. 1b), leading to the decrease of their density, as well as of their compressive strength (Table 2). Furthermore, the increase of S/L ratio increased drastically the compressive strength of geopolymers without increasing substantially their density (Table 2), since the water reduction in the geopolymer systems accelerates the polymerization reactions [16].



**Fig. 1.** (a) Specimens of the FA50-BFS50-N7 geopolymer (FA = 50% wt.; GGBFS = 50% wt.; alkali activator = 7 M NaOH and Na<sub>2</sub>SiO<sub>3</sub> solution) and (b) Specimens of the FA90-BFS10-SF2.5-N7 geopolymer (Geopolymer precursor = 90%wt FA and 10% wt. GGBFS; SF addition = 2.5% of solids; alkali activator = 7 M NaOH and Na<sub>2</sub>SiO<sub>3</sub> solution).

As shown in Table 2, the increase of alkali concentration in the alkali activator decreased importantly the compressive strength of the resulted materials. In particular, the highest compressive strength for the Na-based geopolymer system is achieved at the lower investigated concentration of NaOH solution (7 M), while the compressive strength of the materials in the K-Based geopolymer system remains almost unchanged for the highest concentrations of KOH solution investigated in this study (7 and 8 M) and decreased drastically for the lower investigated concentration. The concentration of the alkali hydroxide solution in the activator of a geopolymer system presents an optimum, which is strongly related to the extent of the geopolymeric network formed in the matrix of materials [16]. As it is well-known, the alkali ions balance the excess of the negative charge created during the incorporation of the Al IV-coordinated atoms into the geopolymeric network. The use of Na or K in the alkali activator did not affect so much the properties of the produced geopolymers (Table 2), but it will definitely affect

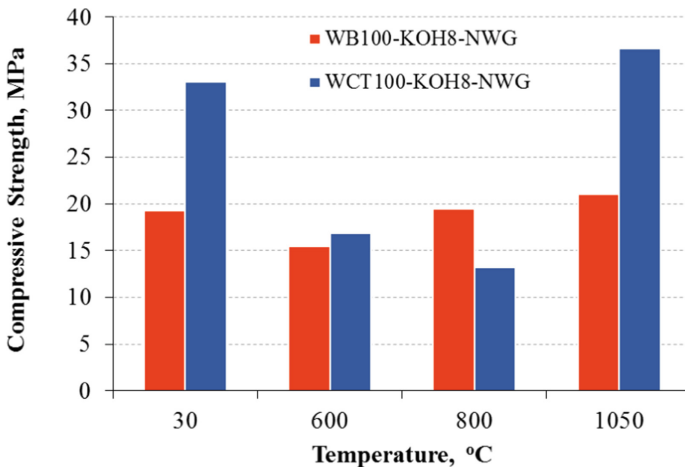


their thermal stability at high temperatures. The selection of the alkali in the activator will determine the crystalline phases that will be formed at high temperatures and their melting points, which in turn determine the temperature of the geopolymers mechanical failure [17]. Among the geopolymers given in Table 2, the WB100-KOH8-NWG and WCT100-KOH8-NWG were selected for further investigation during their exposure at high temperatures.

### 3.2 Thermal Stability at High Temperatures

In Fig. 2, the residual compressive strength of the geopolymers WB100-KOH8-NWG and WCT100-KOH8-NWG, after their exposure at 600, 800 and 1050 °C for 2 h, is presented. For reasons of comparison, the compressive strength of the same materials after curing (30 °C, 7 d), is also given.

The thermal treatment of geopolymers took place in a lab-scale electric furnace. In each experiment, the geopolymers' specimens were placed in the furnace that started from room temperature to reach the desired temperature, operating with a heating rate of 4.4 °C/min. The specimens were left at the desired temperature for 2 h and then, the furnace was turned off. After each experiment, the compressive strength, density and mass loss of geopolymers were measured. In all the experiments, cubic specimens of 50 × 50 × 50 mm, which were cured for 7 d at 50 °C and hardened for another 7 d at ambient temperature and dry conditions were used.



**Fig. 2.** Residual compressive strength of the geopolymers WB100-KOH8-NWG and WCT100-KOH8-NWG, after their exposure at high temperatures for 2 h (curing at 30 °C for 7 days).

As shown in Fig. 2, the exposure of both geopolymers at high temperatures resulted in changes of their compressive strength, which are more significant in the case of the WCT100-KOH8-NWG one. More precisely, the residual compressive strength of this material was substantially reduced up to 800 °C and then, it was sharply increased as temperature raised to the highest value tested (1050 °C). At this temperature, the residual



compressive strength reached a slightly higher value than the initial one. The residual compressive strength of the WB100-KOH8-NWG geopolymer followed a similar trend, but with less abrupt changes (Fig. 2).

The decrease of compressive strength observed at 600 and 800 °C for both of the studied geopolymers (Fig. 2) is strongly related to the softening of the geopolymeric matrix, which happens between 550 and 650 °C [18]. At 800 °C, the amorphous phase of geopolymers could be transformed to a crystalline one, which in the case of sodium (nepheline) starts to melt, resulting in a losing structure with decreased mechanical strength. Above 800 °C, sodium acts as flux, causing partial sintering of the unreacted solid particles in the contact zone, between them and the melted geopolymeric binder, which results in more durable structures [19]. This process improved the residual compressive strength of the developed materials at 1050 °C.

During the exposure of both the studied materials at the investigated temperatures, there were no signs of spalling or deformation phenomena. Only small size cracks appeared on the specimens' surface at 800 °C, which diaped after their heating exposure at 1050 °C. The viscous gel phase formed at such high temperatures achieves the healing of cracks [20].

## 4 Conclusions and Further Work

The design of a novel Hybrid Laminate Material (HLM), which aims at protecting structures against explosions, fires and impacts, was presented and discussed. Preliminary experiments performed for the development of the fire-resistant layer (FRGC) of HLM, according to the geopolymerization technology, have proved the potential of waste materials valorization, leading to a viable and more sustainable final material.

The fly ash and the construction and demolition wastes were proved as the most promising waste materials among the investigated for the development of the FRGC layer. The geopolymers based on the waste bricks and ceramic tiles presented a good thermal stability and adequate mechanical performance after exposure at high temperatures. However, further research and intensive lab-scale experimental work are necessary in order to optimize the fire-resistant layer of HLM. The thermodynamic analysis of the geopolymer systems and the use of phase diagrams will assist the final design, development and optimization of FRGC.

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