Investigation of thermal coating influence on the fire resistance of a multi-layer material

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Abstract. In modern practice, more and more multi-layered materials are emerging as response to the ever-changing demands of construction beneficiaries. One of these, a fire resistant multilayer material, is studied in this article. The multilayer material was made in two variants - on support of aluminium foam, respectively ceramic foam, and a ceramic fibre plate was used for the side exposed directly to the flame. On this plate, an Al₂O₃ ceramic coating with the role of thermal barrier was realized by atmospheric plasma spray method. His influence of was evaluated by comparing the recorded temperatures during the fire resistance tests for both the coated and the non-coated samples.

1. Introduction
Thermal spraying is one of the most versatile methods for producing coatings that interfere with modifying the functionality of various materials or components [1-7]. One of the applications that extend more and more the coverage area is the ability to protect components against high temperatures provided by thermal barrier coatings (TBC) [8]. A thermal barrier coating is typically a multilayer consisting of a high temperature resistant top coat and an oxidation-resistant bonding layer that is applied onto the substrate [9]. The top coat usually contains ceramic materials characterized by low thermal conductivity, necessary for thermal insulation, and the bonding layer serves to improve the cohesion between the upper layer and the substrate [10]. Typically, the top coat is obtained by thermal spraying in a plasma jet and is characterized by a layered structure of interconnected splats, pores and cracks between inlays. This unique microstructure is the one that is responsible for lowering the thermal conductivity over the thickness of the TBC [11]. Thermal barrier coatings were used at the beginning to increase the efficiency of gas turbine engines [12, 13], and more and more diverse uses were found: aircraft engines [14], fuel cells for energy systems[15], coating of different components of automotive engines[16-17],coating of polymer matrix composites (PMCs) reinforced by glass, carbon or aramid fibres [18-21]. In this paper we study the possibility of using thermal barrier coatings for obtaining a fire resistant multilayer material for use in the construction industry, for the construction of special purpose buildings.

2. Experimental setup
In this study, materials with different functionalities were used: the first category consists of the materials used as core for the multilayered material, the second category includes the materials that
reduce the heat transfer and ensure the resistance to fire, and the third category includes the materials needed for the assembly in the test stand. The components required to produce the studied multilayer materials are shown in Table 1 and their aspects are presented in Figure 1 a-c.

Table 1. Multi-layer component materials.

| Functional category | Function | Material | Observations |
|---------------------|----------|----------|--------------|
| 1                   | Aluminum core (Figure 1a) | Aluminum closed cells foam (Aluhab by Aluinvent Hungary) | Sample size of 50x50x10 mm, bubble size of about 5 mm |
|                     | Ceramic core (Figure 1b) | SiC-based open cell foam ceramic filter, (Vukopor S20) | Sample size of 50x50x10 mm, 20 ppi porosity, stable at high temperatures, heat shock resistance, $T_{\text{max}} = 1500^\circ\text{C}$ |
| 2                   | Insulation plate (Figure 1c) | Ceramic plate (Grafex CERA A by EtanșăriGrafex SRL Romania) | Produced from ceramic fibers, high temperatures resistance - $T_{\text{max}} = 1200^\circ\text{C}$, low thermal conductivity - $\lambda_{400} = 0.100$ W/mK, low density (0.9 g/cm$^3$), good resistance to heat shock |
|                     | Thermal barrier coating | $\text{Al}_2\text{O}_3$ powder (commercially available as MTS 7015) | The coatings are realized by atmospheric plasma spray and are hard, wear resistant, chemically inert and stable at high temperatures. |
| 3                   | Mounting materials | Ceramic paste (SuperPro by ProInvest Romania) | Special glazing for furnaces with $T_{\text{max}} = 1200^\circ\text{C}$ |
|                     | Textil material (Texxo by BIG Arbeitschutz GmbH, Germany) | Double knit Kevlar textile |

Figure 1. The aspect of some materials used for the multilayer samples: a) aluminum closed cells foam; b) SiC-based open cell foam; c) ceramic plate.

The spraying of the thermal barrier coating was accomplished by the plasma spray deposition method in normal atmosphere using the SprayWizard 9MCE (Metco - Oerlikon, 2008) facility. The coating was performed on both sides of the ceramic plate and on one of the sides of aluminum, respectively ceramic foams, as can be seen in Figure 2. The aspect of the samples is shown in Figure 3, where the white color is given by the layer with the role of the thermal barrier coated from $\text{Al}_2\text{O}_3$. 
Figure 2. Aspect during the atmospheric plasma spray process used to obtain the thermal barrier coatings: 1- elements used for the multilayer material were mounted on the spraying support; 2 - robotic arm used to manipulate the 9MCE plasma gun; 3 - plasma spraying jet.

Figure 3. Aspect of the materials resulted after the thermal spraying process: a) aluminum closed cells foam; b) SiC-based open cell foam; c) ceramic plate.

The multilayer samples tested in this study were made by overlaying the insulating plates over the foam core, with the following configurations:
- Sample 1: ceramic plate + aluminum closed cells foam core;
- Sample 2: ceramic plate + ceramic open cells foam core (SiC);
- Sample 1C: ceramic plate coated with TB layer on both sides + aluminum closed cells foam core coated with TB layer on one side (the coated side was set to the area exposed to direct flame);
- Sample 2C: ceramic plate coated with TB layer on both sides + ceramic open cells foam core (SiC) coated with TB layer on one side (the coated side was set to the area exposed to direct flame).

The test stand was designed as a refractory brick wall in which a 50x50 cm hole was cut for sample placement [22-23]. The flame was obtained by burning a gaseous fuel (mixture of 87% butane and 13% propane) in a burner mounted at a fixed distance of 10 cm from the sample surface, as can be seen in Figure 4a, b. Throughout the whole test, for each of the four samples, the constant fuel flow pressure and therefore a constant temperature, between 600 - 650 °C, was assured. When assembling in the sample stand, the samples were assured from the point of view of maintaining the overlap by gluing them side by side with ceramic paste and textile material, as can be seen in Figure 5 a.

The recording of temperature variations was done with a digital thermometer equipped with a K-type thermocouple, mounted in direct contact with the surface that was not exposed directly to the flame at its center, as can be seen in Figure 5a - in the case of the core sample of aluminum foam, respectively Figure 5b - in the case of SiC core sample. Another temperature recording method used for the surfaces exposed to direct flame is remote measurement at certain points using an infrared thermometer, as can be seen in Figure 5b.
Figure 4. Aspects of the open flame test stand: a) component elements of the test stand: 1 - thermo-resistant brick wall; 2 - sample; 3 - gas burner; 4 - metal frame; 5 - metallic table; b) direct exposure of the sample S1 at open flame (minute 20).

Figure 5. Representative temperature registration aspects: a) thermocouple position in case of S1; b) thermocouple position in case of S2C; c) flame temperature registration with infrared thermometer (S2, minute 47).

Open flame exposure tests were performed over 60 minutes, temperature recording being performed at 1 minute in the first 5 minutes, then every 5 minutes. Three areas were observed: 1) the opposite face of the flame, at the centre of the sample (Figure 5a, b); 2) the face exposed directly to the flame in the central area of the samples (Figure 5c); 3) an area adjacent to the sample, on the face not exposed to the direct flame (marked in red on Figure 5b).

3. Results and discussions

The behaviour of the four samples following the direct flame exposure tests was evaluated in terms of the performance criteria provided in SR EN 1363-1 "Fire resistance tests. General Conditions": sealing and isolation. Sealing is the time in minutes in which the specimen continues to maintain its distinct functions during the test without resulting in sustained flame. Isolation is the time in minutes in which the specimen continues to maintain its distinct functions during the test without developing on the unexposed face temperatures which: a) increase the mean temperature above the initial mean temperature by more than 140 °C; or b) grow at any point above the initial mean temperature above 180 ° C [24-25].

In terms of the first criterion - the sealing - the behaviour of the four samples was considered satisfactory because none of them developed sustained flame during the 60 minutes of each test. In addition, the aspect of the component elements used to obtain the four samples was also analysed, being noteworthy that none of them underwent major structural changes, as can be seen from Figures
However, slight changes were noted in the case of ceramic tiles: the plates sides without TBC exposed to direct flame changed colour without loss of structural resistance (Figure 6c); the ceramic plates with TBC presented localized exfoliation of the layer (Figure 7c).

**Figure 6.** Sample aspects after the open flame test (open flame sides, without TBC): a) aluminium core; b) SiC core; c) ceramic plate.

**Figure 7.** Sample aspects after the open flame test (open flame sides, with TBC): a) aluminium core; b) ceramic plate from S1C; c) ceramic plate from S2C.

For the evaluation of the second performance criterion provided in SR EN 1363-1 - isolation - the temperature records were compared in the three considered points, the graphs being shown in Figures 8, 9 and 10.

It can be seen that in both cases the temperature recorded on the samples without TBC was higher, as expected. The influence of the deposited TB coating is evidenced by the differences between the temperatures recorded for the two types of samples. Thus, in the case of aluminium foam core samples, a difference of between 7 - 70°C was recorded, with an average $\Delta T = 49.87°C$ (Figure 8a). In the case of ceramic foam core samples, the temperature differences varied between 6 - 117°C, with an average $\Delta T = 48.12°C$ (Figure 8b). It can be noticed that the TB coating fulfilled the role of the thermal barrier: in both cases there was a similar average variation, the differences being stable over the entire duration of exposure to the open flame of the samples.

For a fuller interpretation of the behaviour of the studied samples were monitored the temperatures in other two areas: the face exposed directly to the flame and an area adjacent to the samples (Figure 5a). In the first case, that of the area adjacent to the samples, the temperature of the refractory brick wall recorded similar values, as can be seen in Figure 9a, the average temperature at the time of the experiment termination being 398°C. Another monitoring was performed on the surface exposed directly to the flame, where a similar behaviour was observed for all samples, the temperature after stabilization (after the first 10 minutes) ranging between 620-670°C, as can be seen in Figure 9b.
Figure 8. Temperature variation on the sides not exposed to direct flame of samples: a) S1 and S1C; b) S2 and S2C.

Figure 9. Temperature variation of: a) brick walls on the sides not exposed to direct flame (S1, S2); b) sample sides exposed to open flame (S1, S1C).

Figure 10. Temperature variation on the sides not exposed to direct flame of all samples.

Figure 10 shows the comparative behaviour of the four samples in relation to the temperatures recorded on the refractory brick wall. It can be noticed that the samples on the surface of which the TBC layer was deposited have recorded lower temperatures than those recorded in the case of refractory bricks. Regarding the comparative assessment of the influence of the core type, it can be seen that the core made of ceramic foam provided less heat transfer than the aluminium foam core.
Regarding the fulfilment of the second performance criterion provided in SR EN 1363-1 - isolation - it is observed that none of the analysed samples have the capacity to reduce the thermal transfer in such a way that a thermal gradient lower than 180 °C will be registered. However, an interpretation of the behaviour of the multilayer elements compared to the refractory bricks used for the construction of the test stand is favourable, so that the possibility of additional combinations of components with insulating role cannot be overlooked.

4. Conclusions

It was noted that all four samples had satisfactory behaviour in the case of direct flame exposure because none of them developed sustained flame.

The temperatures recorded for the multilayer aluminium foam core materials were higher than those recorded for ceramic core multilayer materials, indicating better thermal insulation for the latter.

Regarding the influence of TB coatings on the fire resistance of the projected multilayer material, it was observed that the presence of this type of layer caused a decrease in temperature by an average of 48 degrees in the case of multilayer materials with thermal barrier coating. We can take into account the production of fire resistant multi-layer elements in which one of the component layers is of the thermal barrier type obtained by thermal spraying.

5. References

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