Development of laboratory techniques for assessment of operating properties of intumescent fireproofing coatings

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Abstract adhesion of a charred layer to the surface of constructions and materials is one of the main components of fire protective efficiency of intumescent charring compositions, especially for metal surfaces. The paper overviews literature sources concerning the investigation of adhesive-cohesive properties of charred layers. It is shown that there are no easily performable methods for assessment of these properties. The laboratory device based on the «back strike» method has been developed. Adhesive-cohesive properties of charred layers modified with carbon additives were assessed using «back strike» method and pushing method. It is shown that these methods provide equal results.

Keywords: methods of evaluation of performance properties, adhesive-cohesive properties of charred layers, safety, «back strike» method, pushing method, operational property

1. Introduction

Fire resistance is an important property of building constructions which must be stated in a building plan; a list of actions must be done to ensure required fire resistance limits. Nowadays one of the most effective ways of ensuring fire protection is to use thin fireproofing swelling coatings; they expand as a result of heating and form heat-isolating carbonaceous (charred) layer on a substrate. This layer acts as a physical barrier which prevents the surface from excessively fast heating, thus preventing the loss of operational properties of a building construction [1-3]. There is a need in information about fireproofing properties of the swelling coatings during whole time of their operating [4,5]. Field (full-scale) fire tests of fireproofing materials for steel constructions are conducted due to GOST 53295, but these tests are expensive and hardly performable; this doesn't allow using them freely for the assessment of fireproofing properties of intumescent materials.

According to the fact that a charred layer must stay on the substrate in any fire conditions (which is one of the most important parts of fire-protective efficiency of intumescent coatings) it is advisable to assess adhesive and mechanical properties of charred layers [6-8].

We have not found any methods of assessment of these properties which could be performed in a non-specific laboratory, only those which are stated in [9]:
- shear resistance method;
- pushing resistance method;
- group of striking methods.

The point of the first method is to measure the shearing force of a swollen (intumescent) layer using the special knife fixed at height h (fig. 1).
Figure 1. Device for shear resistance method:
1) a metal plate, 2) a charred layer, 3) a knife, 4) anti-rubbing device, 5) a plummet

There is an analog of this method – when a charred layer is blown off by air flux of determined speed or by water jet. Valuable data provided by these methods may be found in [10].

It is possible to measure the pushing effect in the following method (fig. 2) in two ways. Firstly, the depth of pushed charred layer can be measured; secondly, the weight of a plummet which pushed a charred layer to the defined depth can be measured. Experiments prove the second way to be more informative.

Figure 2. Measurement of charred layers mechanical properties using pushing resistance method.
Figure 3. Measurement of adhesive and cohesive properties of a charred layer

a) «back strike» method; a) 1) racks, 2) holders (pads), 3) charred layer, 4) a plummet (h is its initial height above charred layer)
b) 1) basis, 3) special box, 4) substrate, 5) charred layer

The group of striking methods is presented by back strike method and pendular strike method. The back strike method is performed the following way: a peen strikes the steel plate with charred from a non-covered side (fig. 3 a); adhesion is calculated as mass of fallen charred layer related to its initial mass. Variation of this method includes the steel plates strike by a falling frame (fig. 3 b) [11,12]. Some limitations of this method: a strike must be blown repeatedly right at the same area of the plate (it is solved – fig. 5); a kickback must also be taken into account – a peen strikes again by inertia, and these strikes are also valuable.

Figure 4. Measurement of adhesive and cohesive properties of charred layer by pendular strike method. Another variant of striking methods is a pendular strike method. It is similar to methods in which pendular hardness testers are used. Pendular strike method has its own limitations: a metal plate which is held from one side moves oscillatory, additionally destructing a charred layer; also, a there must be one strike blown to a certain area – a hard rod held by bearing is used for that.

Pushing resistance method and back strike method are considered the most easily performable is a non-specified laboratory.

The purpose of the research is to develop new technique for assessment of adhesive properties of charred intumescent layers basing on the methods mentioned above.
2. Materials and methods
To assess adhesive and cohesive properties of charred layers basing on the back strike method a device was made (fig. 5). Columns 2 are fixed in the basis 1; frame 3 with sample 4 fixed in it can move on them. A charred layer 5 is turned downside. A sample 4 is a steel plate (80×140×4 mm). A frame 6 with a peen 7 can also move and fall down to the sample. So, the device has two applications: sample fixed in a frame may fall down itself, or a peen may fall down and strike the sample.

![Figure 5. a) scheme; b) picture of a device for back strike method.](image)

Earlier it was shown [13,14] that modification of fireproofing compositions by carbon frame structures, such as nanotubes and intercalated graphite, leads to increase in fireproofing effect. Apparently, these additives affect charred layers microstructure (fig. 6) which was proven by data obtained with REM JSM 7001F (JEOL, Japan). An initial composition is a water soluble paint including an intumescent triad – melamine, pentaerythritol and ammonium polyphosphate.
Figure 6. Images of the microstructure of charred layers obtained by scanning electron microscopy; a) an initial sample; b) a sample modified by nanotubes; c) a sample modified by thermally expanded graphite.

Table 1: Additives used for modification

| Sample          | Additives               | Quantity, mass.% |
|-----------------|------------------------|-----------------|
| Initial sample  | –                      | –               |
| I               | Carbon nanotubes       | 0,7             |
| II              | Thermally expanded graphite | 0,7           |

Steel plates were cleaned from pollution and oxides in accordance with GOST 9.402. Compositions were applied to the plates. Thickness of dry layers was measured using MT-41NC device. Coated steel plates were placed to a furnace for 5 min at 600 °C. After that, adhesive and cohesive properties of formed charred layers were measured using the developed device. The mass of charred layer which stayed on the plate after a strike and the mass of whole charred layer were measured. Five parallel experiments were conducted for each sample, average values were calculated.

Table 2. Results of an experiment.

| No  | Modifying additives     | Adhesion coefficient (peens strike), % | Adhesion coefficient (frame falling), % | Critical pushing mass, g. |
|-----|-------------------------|----------------------------------------|----------------------------------------|---------------------------|
| 1   | Initial sample          | 45                                     | 9                                      | 90,3                      |
| 2   | Carbon nanotubes        | 93                                     | 17                                     | 104,2                     |
| 3   | Thermally expanded graphite | 64                                     | 12                                     | 97,8                      |

3. Conclusions
A new technique and a devise for assessment of mechanical properties of intumescent charred layers were developed.

Experimental data obtained by this technique shows that the back strike method and frame falling method provide correlative results. The pushing resistance method shows best result for the sample modified by carbon nanotubes. This method allows to measure the charred layers hardening degree
and is easily performable. The striking method requires special devices but proves to be more informative – as charred layers are exposed to big amount of pressure and strikes from turbulent gas flows in case of real fire.

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