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Study of influence of filler composition in composite systems on emissivity

ANNOTATION OF DISSERTATION THESIS

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Content

Summary

The research work is focused on the possibility to influence the emissivity of an inorganic composite coating by changing the filler composition. These coatings can also be used in power-producing equipment and they can increase the efficiency of this type of equipment thanks to high emissivity. The intention of our project is to develop a system which will provade a very high normal spectral emissivity (0.75 - 0.99) within a short and medium wavelength range of infrared spectrum of electromagnetic radiation (1,5 – 8,0 µm) and at temperatures above 700 ^{o}C where most of the thermal energy is transfered. For example lining materials usually have the emissivity of 0.20 – 0.40 in this spectral zone, steel has the emissivity of 0.60 – 0.80. This provides sufficient space for the emissivity improvement by the developed emissivity coatings. The coatings have to be resistant to the given conditions which occure in the thermal equipments (very high temperature and different level of abrasion). BG HitCoat® is one of these protective coatings. Chromium Oxide is the active ingredient which is contained in the filler of its basic composition. Other active ingredients were also built into the matrix of the composite. Some of the active ingredients had high value of emissivity (Iron, Silicon Carbide), others had high value of melting temperature (Magnezite, Magnetite, Kaolin, Boron Carbide, Boron Nitride, Zirconium Oxide, etc.). Emissivity comparison of new samples then followed.

Fourrier Transform Infra-Red (FTIR) Spectrometry was applied for measurement of normal spectral emissivity. Research required measurements carried out mainly at high temperatures. Radiation of researched materials was compared with radiation of the comparative high temperature coating based on Cr_2O_3 and with radiation of the black body. Influence of filler composition on the emissivity in monitored range of infrared spectrum was demonstrated. The least emissive active ingredient (Zirconium Oxide) had the values of 0.09 – 0.60 in the range, the most emissive one (mixture of nitrides) had the values of 0,75 - 0,93.

The material with the most emissive active ingredient was then subjected to comparison with the original formulation. All important parameters were compared (thermal resistance, thermal conductivity, adhesion, morphology) and the new material was comparable in all the tests.

Keywords: inorganic composites, IR spectrometry, emissivity.

1. Introduction

During the last decade inorganic composite materials have been used for protection of heat transfer surfaces which are pelted with heat, clinkering, abrasion and high temperature corrosion. If the materials also have a high emissivity, they can be used in various heating plants (industrial furnaces, combustion chambers, heat exchangers, etc.) to improve energy efficiency. Other research and development works in this area deal both with new materials and technologies for their application [1-4], and with theoretical analysis [5, 6] and practical use of these coatings [7-9]. Development of new methods for measuring emissivity is needed [10, 11].

The project FR-TI1/273 "Research and development of high emissive composite coatings for high temperature aplications" had similar topic. The project was realized with the financial support from the state budget of the Czech Republic through the Ministry of Industry and Trade. Company BG SYS HT Ltd., which is the producer of innorganic composite material BG HitCoat® [12], also co-financed the project. The aim of the project was to find more suitable active ingredient which improves the emissive properties of the material.

Analyses of uses status of emissive coatings in heating units was realized at the beginning. At first suitable active ingredients were searched for and samples for emissivity measurement were prepared. Then several phases of spectral normal emissivity measurement were realized.

The most suitable active ingredient, which improved emissive properties of the original material, was found. The new material fully complied with the comparative tests of the original material.

2. Experimental part

2.1. Overview of active ingredients

The substances were selected according to two basic criteria: emissivity higher than 0.75 and/or melting temperature higher than 1,500 ^{o}C. These two substances were chosen from the table of known emissivites [13]: Iron (Fe) and Silicon Carbide (SiC). The emissivity of other substances was not known, only suitable parameter was a higher melting temperature. The substances were selected: Chromium Oxide (Cr_2O_3), Carbonyl Iron Powder (C-Fe), Cerium Oxide (CeO_2), Magnetite (Fe_3O_4), Barite ($BaSO_4$), Magnesite (MgO), Kaolin, Boron Carbide (BC), Boron Nitride (BN), Aluminium Nitride (AlN) and Zirkonium Oxide (ZrO_2).

2.2 Preparation of individual formulations

Mixture of fillers including the appropriate active ingredient was inserted into the binder solution. Dispergation with using propeller stiller (500 min^{-1}) and ultrasonic equipment was executed (10 minutes).

Composition of the fillers for the individual formulations came from basic formulation of commercially exploited composite BG HitCoat® (see Tab. 1). Chromium oxide was used as the active ingredient in basic formulation. This active ingredient was replaced by the above mentioned substances in other formulations. Overview of all the prepared samples including active ingredients is shown in Table 2. The percentage of the original active ingredient in the filler (25 vol. %) was maintained in the first stage. Active ingredients with the best emissive results were chosen in the second stage (the third and the fourth phases). The proportion of these active ingredient was increased to 50 and 75 vol. %. The effect of the amount or combination of these active ingredients on spectral normal emissivity was then researched.

Tab. 1 Basic formulation

Raw material	**Weight proportion [%]**
binders	45 - 55
fillers	45 - 55
active ingredient – chromium oxide	25 vol. % (in filler)
Pigment volume concentration (%)	50 - 60

Tab. 2 Overview of prepared samples

Sample Nō.	Active ingredient	Coating thickness [μm]	Proportion of act. ingredient in filler [vol. %]	Pigment volume concentration[b)] [%]
S01	steel [a)]	-	-	-
S02	Cr_2O_3	153 ± 12	25	50 - 60
S04	C-Fe	148 ± 10	25	50 - 60
S05	BN	145 ± 8	25	50 - 60
S06	C-Fe + Cr_2O_3 (1:1)	156 ± 11	25	50 - 60
S07	AlN	150 ± 13	25	50 - 60
S08	SiC	153 ± 10	25	50 - 60
S09	Fe_3O_4	158 ± 11	25	50 - 60
S10	nano-Fe	148 ± 9	25	50 - 60
S11	$BaSO_4$ (Barite)	153 ± 10	25	50 - 60
S12	CeO_2	159 ± 13	25	50 - 60
S13	B_4C	146 ± 10	25	50 - 60
S14	MgO	149 ± 10	25	50 - 60
S15	Kaolin	155 ± 11	25	50 - 60
S16	Carbides (SiC + B_4C, 1:1)	159 ± 9	25	50 - 60
S17	Nitrides (BN + AlN, 1:1)	148 ± 11	25	50 - 60
S18	ZrO_2	151 ± 12	25	50 - 60
S21	Nitrides (BN + AlN, 1:1)	156 ± 15	50	50 - 60
S23	C-Fe	149 ± 8	50	50 - 60
S24	C-Fe	146 ± 10	75	50 - 60
S25	C-Fe + Nitrides (2:1:1)	144 ± 11	50	50 - 60
S26	Nitrides (BN + AlN, 1:1)	150 ± 10	75	50 - 60
S28	C-Fe + Nitrides (2:1:1)	153 ± 15	75	50 - 60
S29	C-Fe + Nitrides (2:1:1)	156 ± 13	25	50 - 60

a) heat resistant steel (ČSN 41 7153)

b) equal value in all samples

2.3. Testing of composite properties

2.3.1 Spectral normal emissivity

2.3.1.1 Measurement method of spectral normal emissivity in high temperatures

Measurement was performed at the New Technologies Research Centre of the University of West Bohemia in Pilsen. Several measurement methods including functional samples and software for processing the results were developed for the research [14 - 22].

23 diferent formulations were prepared (see Tab. 2), at least 20 pieces of each sample. These samples were without heat treatment in the first stage, but samples were stabilized in advance at 900 ^{o}C in the second and other stages.

Spectral dependence of normal emissivity was measured at 500, 800 and 1,100 ^{o}C in the first phase. Block (resistive) furnace was used in that phase. Spectral dependence was measured against pre-measured spectral dependence of reference coating (ZYP Coating, USA).

Spectral dependence of normal emissivity was measured in the range of 400 - 1,200 ^{o}C (each 100 ^{o}C) in the second and other phases. Heating with laser beam was used in these phases. Values were measured either in comparison with the black body in range of 400 – 980 °C or in comparison with the reference coating in range of 1,000 – 1,200 °C [20, 21].

2.3.1.2 Composition of measurement apparatus

The detection system of the apparatus was compised of spectrometer FTIR Nicolet 6700. The laboratory black body Omega BB-4A served as a reference radiation source. The sample was heated either by block (resistive) furnace in the first phase or by fibrous laser Jenoptik JenLas Fiber CW (400 W) in the other ones. Laser beam was oscilated on the back side of the sample by scan head Scanlab Hurryscan.

Surface temperature of the front side of the sample was measured in two ways: contactlessly by infrared camera FLIR A320 and contactly by thermocouple system Adam 4018 [20, 21].

2.3.2 Testing of thermal resistance using powerful laser

New method of high temperature lifetime testing of coatings using HPDD laser was designed, tested and realized at the New Technologies Research Centre of the University of West Bohemia in Pilsen [23].

Both original and new formulations were prepared, three pieces of each sample, all without heat treatment.

The measuring and testing system used powerful laser for heating of samples. Combination of both contactless heating and moving laser beam enabled us to perform lifetime tests of coatings.

Laser beam was generated by powerful direct diode laser Coherent ISL-4000L with maximum power 4 kW.

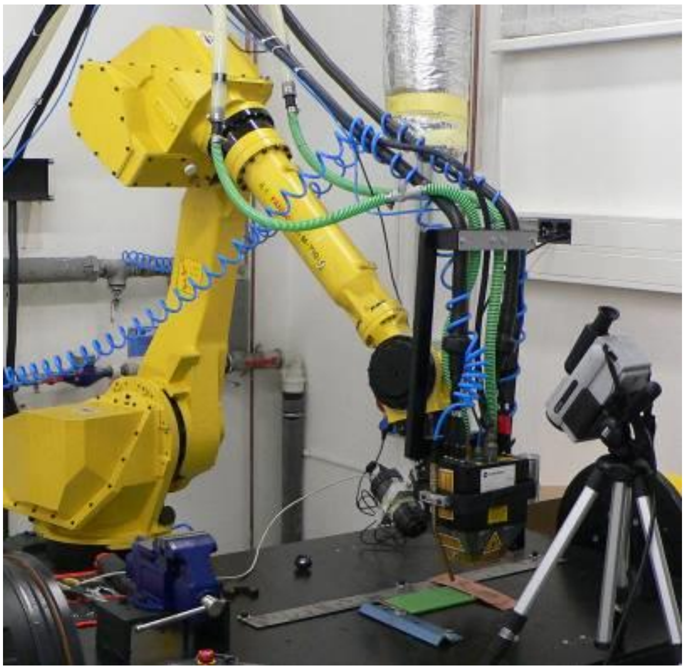

Fig. 1 Laser Coherent ISL-4000L (source [22])

Values of surface temperature depending on the distance in the x-axis were the result of the measurement. The values were plotted in graphs and were compared to each other. Concurrently the surface of a trajectory track beam was examined by an optical microscope.

2.3.3 Thermal conductivity

The measurement was performed at the Institute of plasma physics of the Academy of Science in Prague.

Both original and new formulations were prepared, three pieces of monolithic blocks for each, all without heat treatment.

Xenon-flash method was used for measurement of thermal conductivity by the Anter FL3000 device. The measurement was realized in protective atmosphere of nitrogen and at 500, 600, 700, 800 and 900 ^{o}C. The measurement consisted of three consecutive flashes.

Values of the coefficient of thermal conductivity depending on temperature were the result of the measurement. The values were plotted in graphs and were compared to each other.

2.3.4 Determination of adhesion

Both original and new formulations were prepared, two pieces of samples for each, the first one without heat treatment, the second one was burned at 1,000 ^{o}C.

Determination of adhesion was realized according to the standard ČSN EN 24624. The Elcometer 106 device (see Fig. 2) was used as the tearing equipment. Values of adhesion were measured by the device and surface fracture was explored (see Fig. 4b).

Fig. 2 Elcometer 106

Fig. 3 Glued test specimen

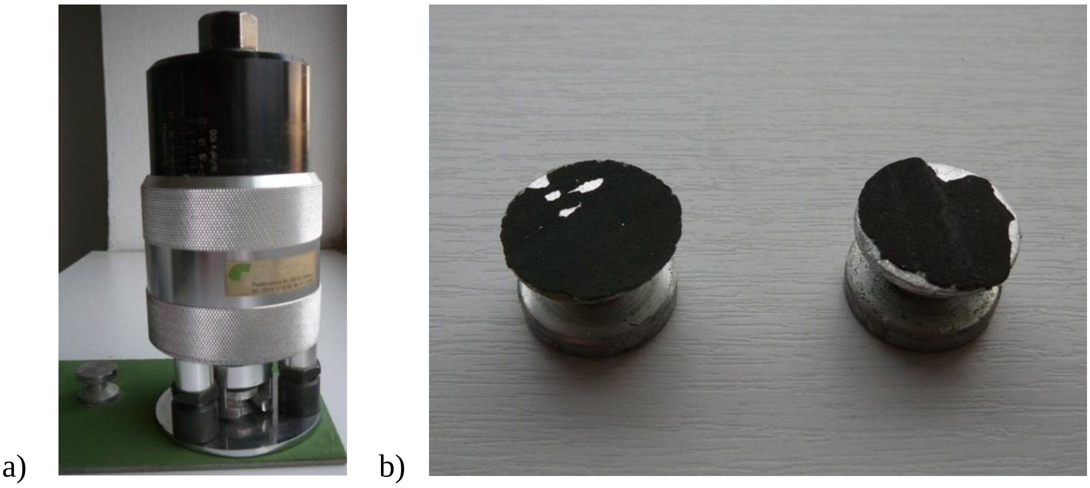

Fig. 4 Tearing device (a), surface fracture (b)

Values of adhesion depending on filler composition (different active ingredients) were the result of the measurement. The values were plotted in graphs and were compared to each other.

2.3.5 Scanning electron microscopy

Measurements were performed at the Regional Centre of Advanced Technologies and Materials of the Faculty of Science of Palacký University in Olomouc.

Both original and new formulations were prepared, four pieces of samples for each, two pieces were without heat treatment, another two pieces were burned on 1,000 ^{o}C.

Scanning electron microscopy was used for the study of coating appearance. High resolution microscope Hitachi SU6600 was used. Gold plating was performed on the SCD 050 device.

Pictures with different magnification depending on the filler composition (different active ingredients) were the result of the measurement. The pictures were presented and commented.

3. Results and discusion

3.1. Measurement of spectral normal emissivity (1st phase)

Spectral normal emissivity of reference coating (ZYP Coating), steel surface (sample S01) and prepared samples (S02 to S17) were measured in the first phase. Only block (resistive) furnace was available for heating in this phase of research.

Samples with the highest values of spectral normal emissivity are displayed in the graph in Fig. 5 (at 800 °C). The sample of steel substrate (S01) is displayed for comparison. There is an interesting finding that the steel surface has emissivity is comparable to the emissivity of the best samples for higher wave number (or shorter wavelength), but the emissivity markedly declines with declining wave number (or growing wavelength). Samples S02 (Cr_2O_3), S04 (C-Fe), S06 (C-Fe+Cr_2O_3), S16 (Carbides) and S17 (Nitrides) are compared in the graph.

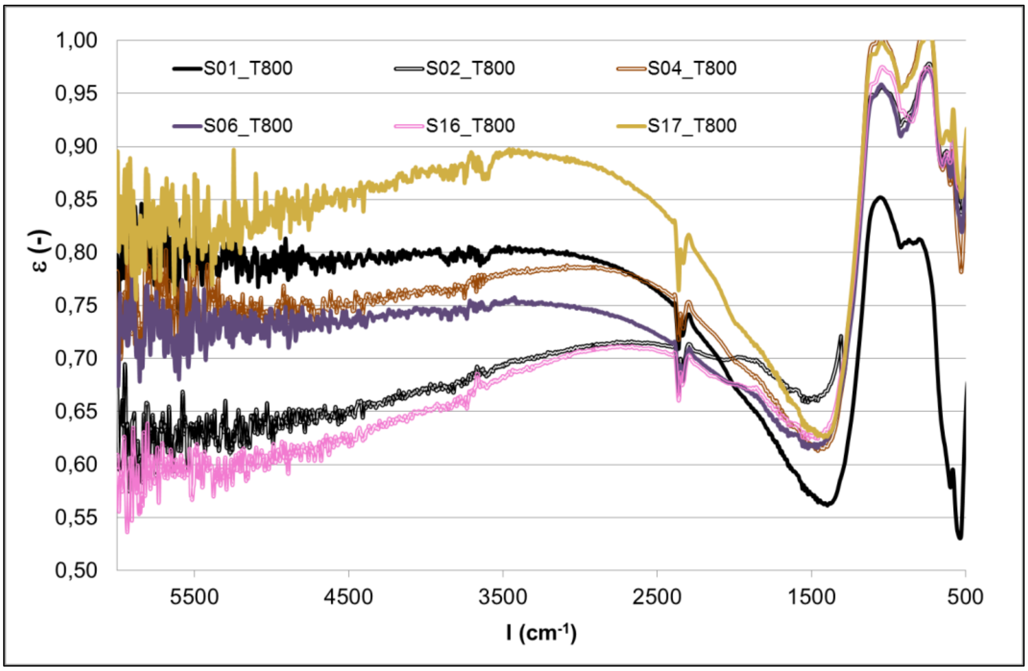

Fig. 5 Absolute spectral dependence of normal emissivity of samples with the highest values (at temperature 800 °C)

The influence of different composition (different active ingredient) is evident especially for the wave number over 1,500 cm^{-1} (wavelength under 6.7 μm). The values oscillate between 0.58 to 0.90 in monitored spectrum range. Samples S04 (C-Fe) and S17 (Nitrides) show the highest spectral normal emissivity from the series, sample S16 (Carbides) shows the lowest emissivity. It shows that there is not a very significant diference between the samples for wave number under 1,400 cm^{-1} (wavelength over 7.1 μm), only the steel substrate (S01) is different.

The samples showed analogical results also for higher and lower temperatures.

Heating of samples was performed in block furnace in the first phase. Samples of Carbonyl Iron and Nitrides had the highest values of spectral normal emissivity.

3.2. Measurement of spectral normal emissivity (2nd phase)

Sample S18 (ZrO_2) was supplied into the set of samples in the second phase, some samples from the first phase were left out (S10 (nano-Fe), S11 (Barite), S12 (CeO_2) and S13 (Boron Carbide)). Spectral normal emissivity was measured for remaining samples again. Samples were heated by laser beam in this and other phases.

Samples with the highest values of spectral normal emissivity are displayed in the graph in Fig. 6 (at 980 ^{o}C). Samples S04 (C-Fe), S16 (Carbides) and S17 (Nitrides) are compared in the graph. Sample of steel substrate (S01) is displayed for comparison. An interesting finding from the first phase was confirmed again. Steel surface (sample S01) shows emissivity comparable to emissivity of the best samples for higher wave number (or shorter wavelength), but the emissivity markedly declines under 700 cm^{-1} (over 14 μm).

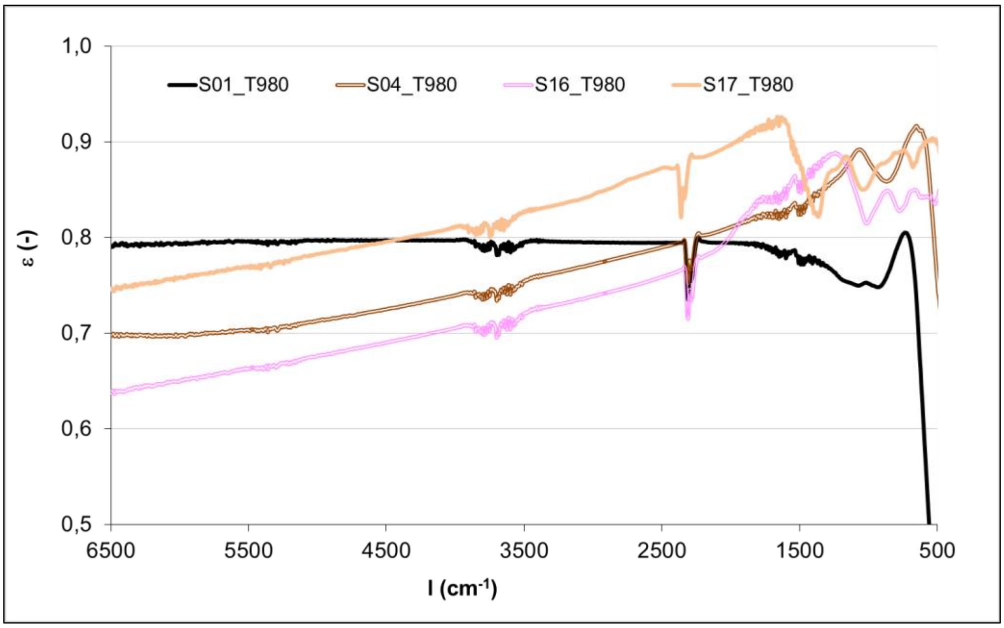

Fig. 6 Absolute spectral dependence of normal emissivity of samples with the highest values (at temperature 980 ^{o}C)

The influence of different composition (different active ingredient) is evident especially for the wave number over 1,500 cm^{-1} (wavelength under 6.7 μm). Sample S17 (Nitrides) shows the highest spectral normal emissivity from the series, its values oscillate between 0.75 to 0.93 in monitored spectrum range. The values are higher approx. by 12 % in comparison with sample S04 (Carbonyl Iron). It was discovered that no significant difference between the samples exists for wave number under 1,150 cm^{-1} (wavelength over 8.7 μm), only steel substrate (S01) is different, it has markedly worse spectrum here.

The samples showed analogical results also for higher and lower temperatures.

Heating of samples in the second phase was performed by laser beam. The observed results were following:

- results from the first phase were confirmed, laser form of heating mainly saves time,
- these active ingredients (Carbonyl Iron, Carbides and Nitrides) showed high values of spectral normal emissivity (between 0.70 and 0.93 in monitored spectrum range),
- only two active ingredients with the highest spectral normal emissivity were chosen for next phases: Carbonyl Iron and Nitrides, while Nitrides had higher values approx. by 12 % for the wave number over 1,500 cm^{-1} (wavelength under 6.7 μm).

3.3. The influence of the content of active ingredient on spectral normal emissivity (3rd phase)

Active ingredient content was incresed from 25 vol. % to 50 and 75% vol. %.

The increase of content of Carbonyl Iron led to a slight increase of spectral normal emissivity, approx. by 15 % in monitored spectrum range. The increase will be unprofitable for practical use (the price-performance ratio will multiply).

The increase of content of Nitrides had no (or rather negative) effect on spectral normal emissivity in monitored spectrum range.

3.4. The influence of combination of active ingredients on spectral normal emissivity (4th phase)

The best two active ingredients were combined in ratio 1:1 (vol.) and samples with increased content were also prepared and measured.

Combination of the ingredients had no effect on spectral normal emissivity, emissivity value actually rapidly decreased with the increase of content at 980 °C.

3.5. Evaluation of spectrometric measurements

Composition of sample S17 (Nitrides, with content 25 vol. %) was chosen for comparative tests with the original formulation. The comparison between material with original formulation (S02) and material with improved spectral normal emissivity (S17) is shown in next graph (see Fig. 7).

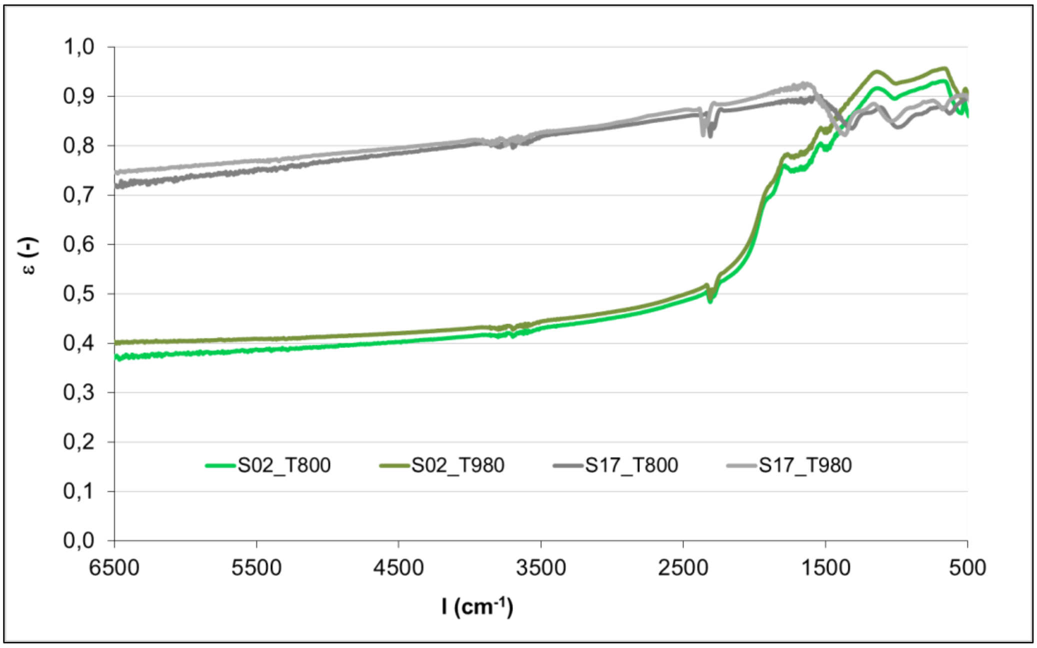

Fig. 7 Absolute spectral dependence of normal emissivity of original material (S02) and the most emissive sample (S17) at temperatures 800 and 980 °C

3.6. High temperature resistance

The experiment result is shown in Fig. 8. Pictures of individual trajectories are complemented by temperature record (see Fig. 9 - 11).

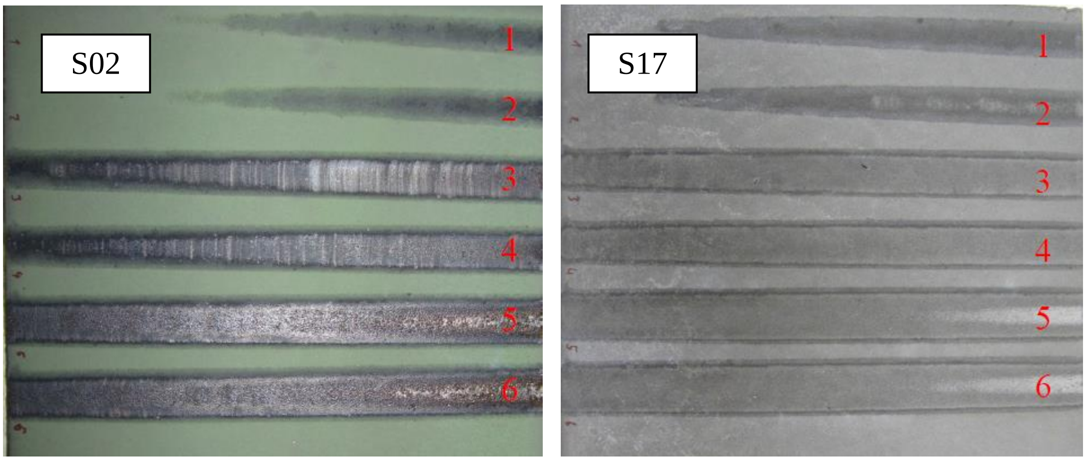

Fig. 8 Effect of laser beam on both materials

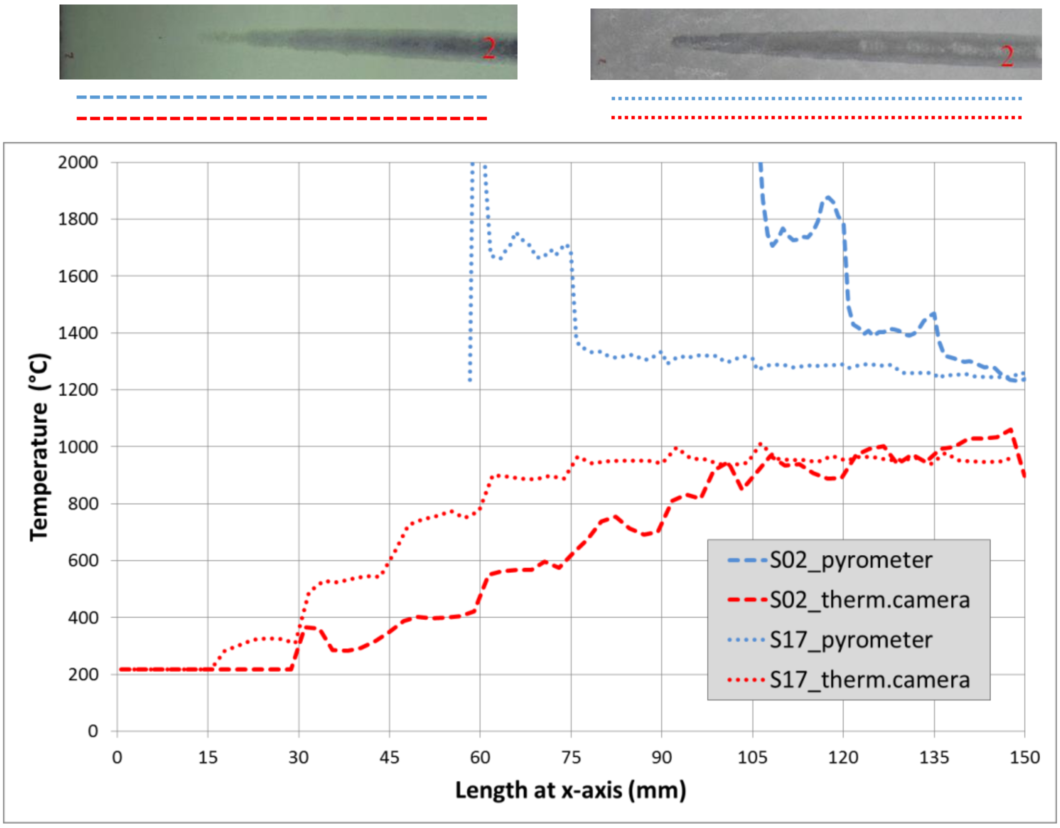

Fig. 9 Temperatures measured on trajectory No. 2

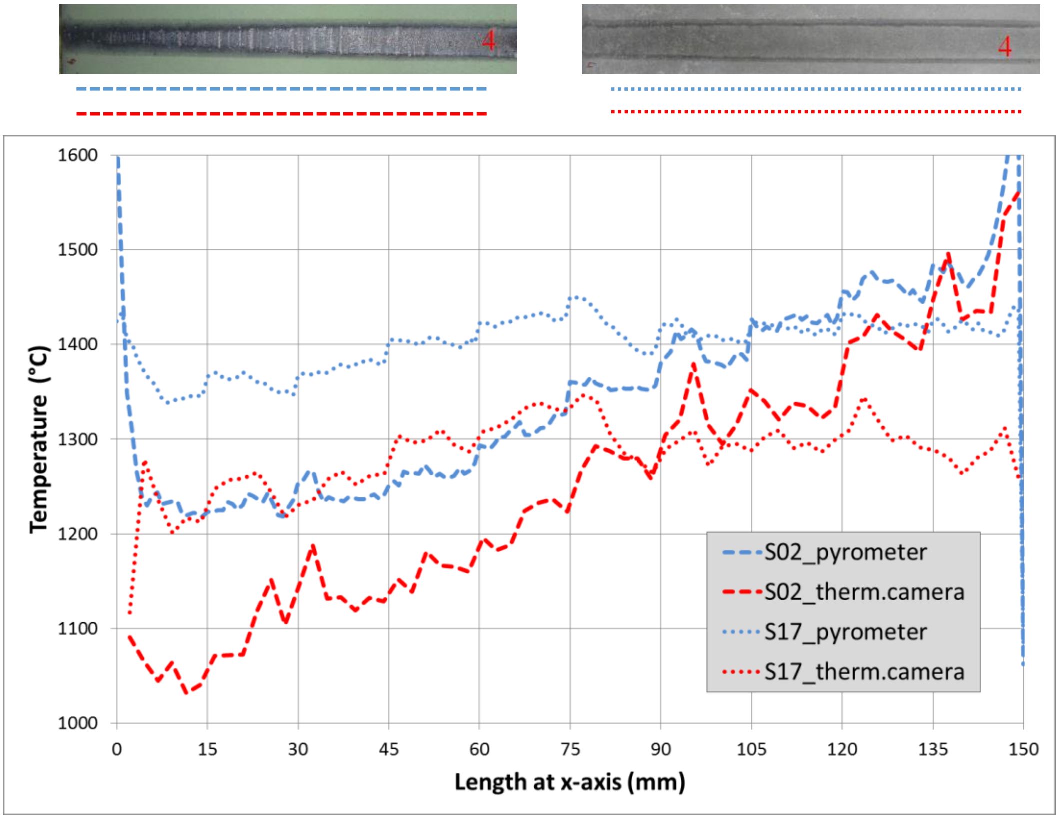

Fig. 10 Temperatures measured on trajectory No. 4

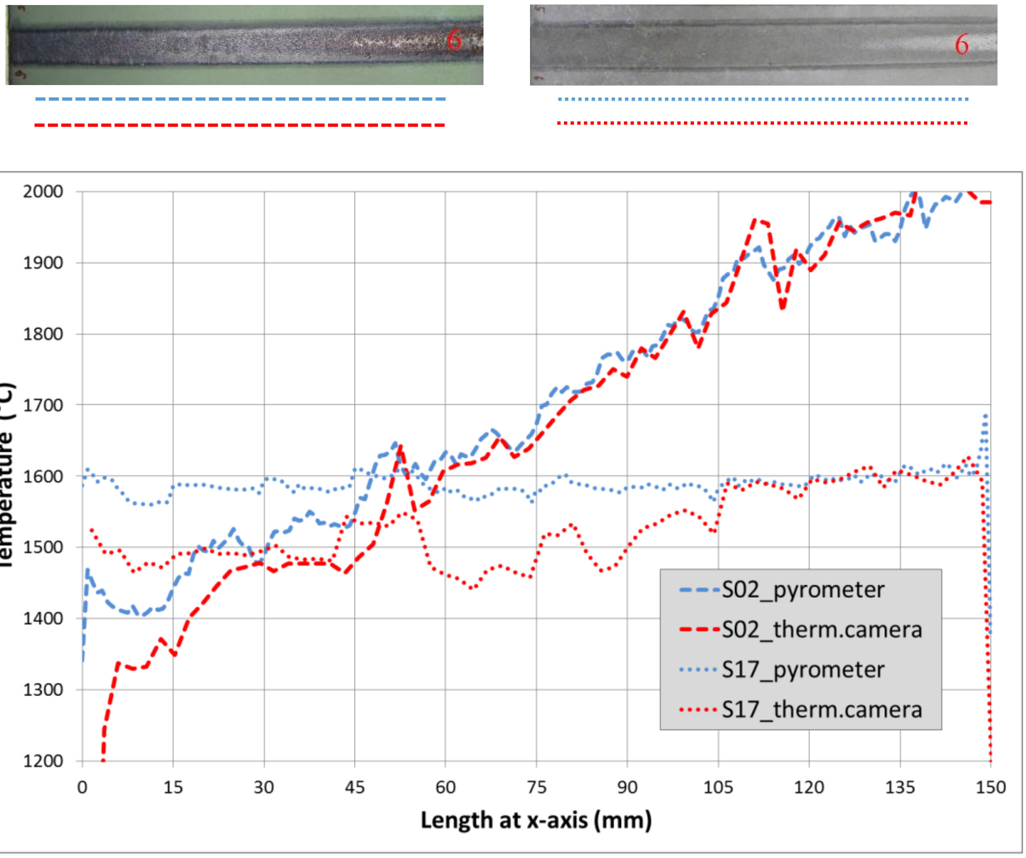

Fig. 11 Temperatures measured on trajectory No. 6

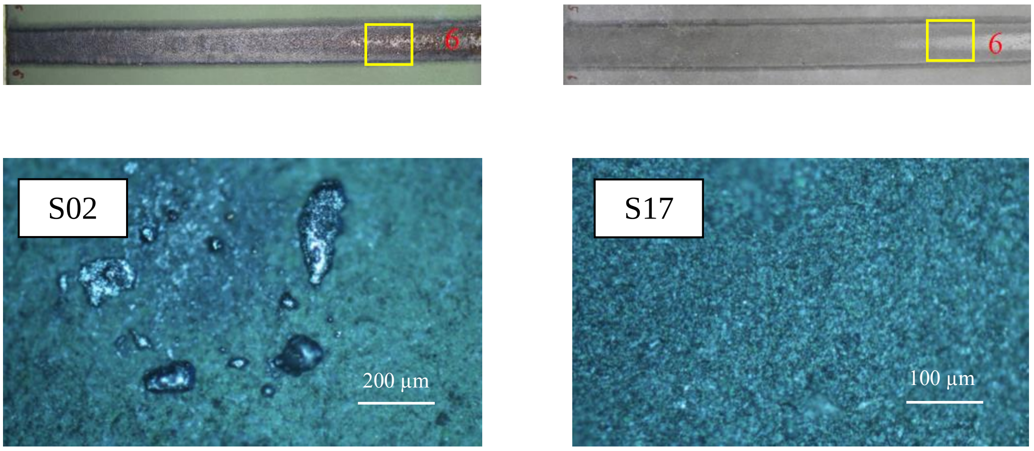

Fig. 12 Microscopic pictures of yellow marked position

The materials behaved differently at first sight. Monitored surface heated up with different speed and to different surface temperature. It was caused by the different values of spectral normal emissivity and thermal conductivity. Temperature of sample S02 increased proportionately with the increase of laser beam power and rose up to 2,000 ^{o}C. On the other hand sample S17 heated up faster, but its temperature did not increase proportionately with the increase of laser beam power, maximum temperature stabilized at 1,600 ^{o}C.

Materials responded differently to the effect of the laser beam. Material S17 was almost unchanged regardless the power of the laser beam, while material S02 was changed (melted) due to higher power of the laser beam. From this point of view the material S17 was more heat resistant.

3.7. Determination of the coefficient of thermal conductivity

Measured results are shown in next graph (see Fig. 13).

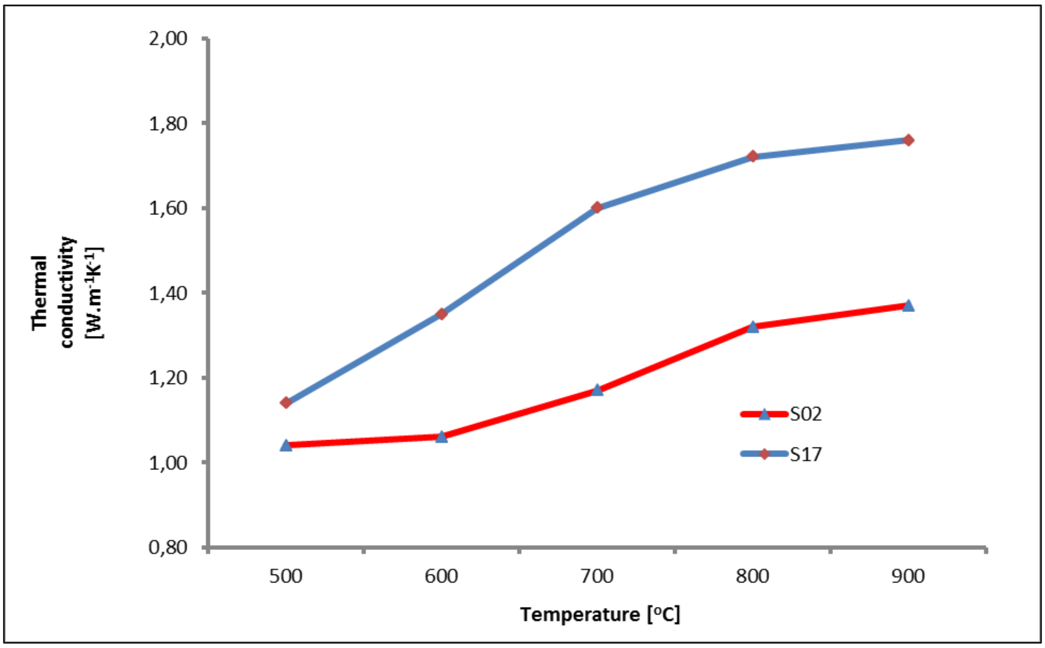

Fig. 13 Temperature dependence of the coefficient of thermal conductivity of the original material (S02) and the most emissive sample (S17)

The results showed thermal conductivity within the value range typical for ceramic material [24]. Slight increase with temperature was observed in both samples. Material S17 will be more conductive due to higher coefficient of thermal conductivity, between 10 and 37 % in monitored temperature range.

3.8. Determination of adhesion

Average adhesion values of individual samples are compared in next graph (see Fig. 14).

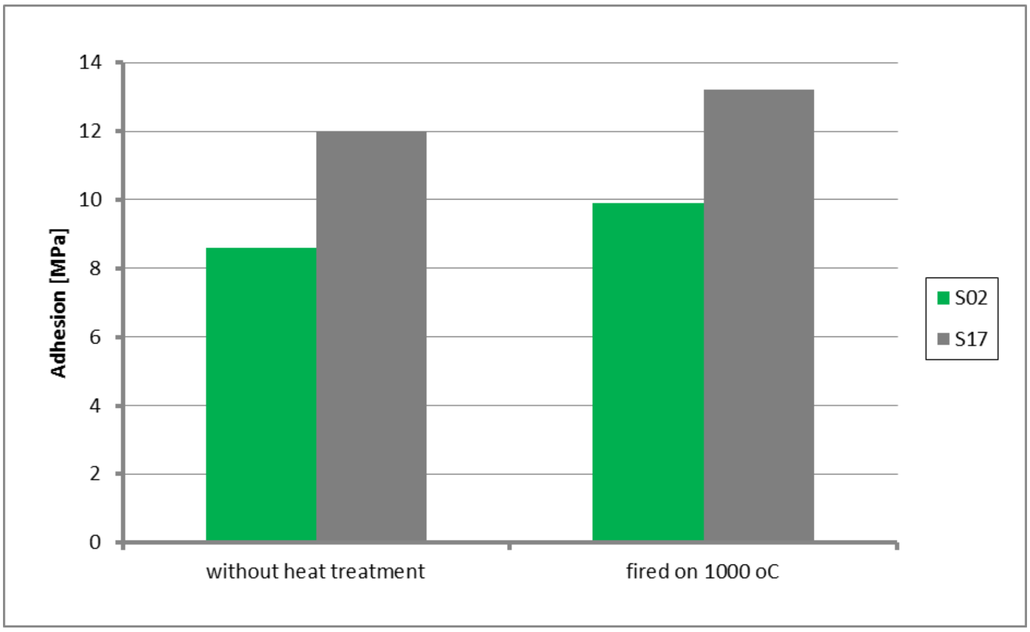

Fig. 14 Adhesion of individual samples

The first comparison was performed for burned samples. Burning increased adhesion of the original material (S02) by 15 %, for sample S17 by 10 %. The second comparison was performed for different active ingredients. Sample S17 had higher adhesion by 40 % for unburned sample and by 33 % for burned sample.

Material S17 was more adhesive to steel surface than original material S02.

3.9. SEM analysis

3.9.1 SEM analysis of sample S02

3.9.1.1 SEM pictures, without heat treatment

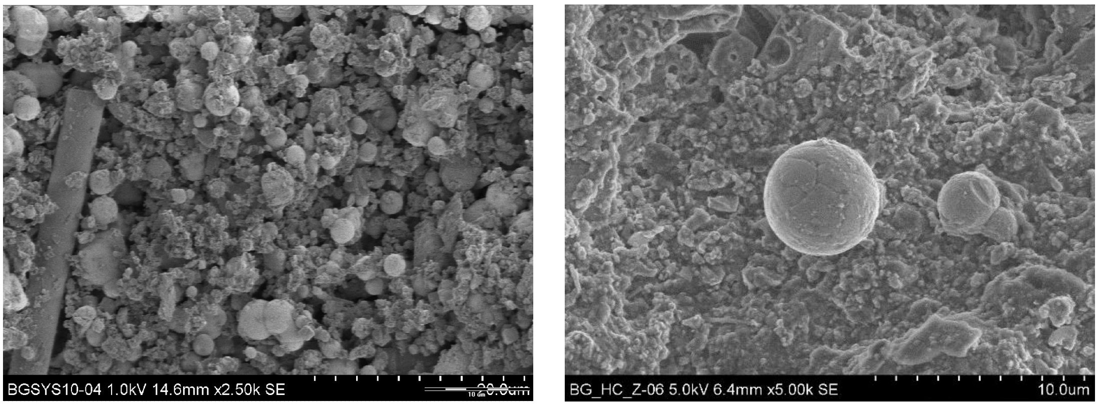

Fig. 15 Texture of sample S02, without heat treatment, magnification 2,500 and 5,000 times

3.9.1.2 SEM pictures, burned on 1,000 ^{o}C

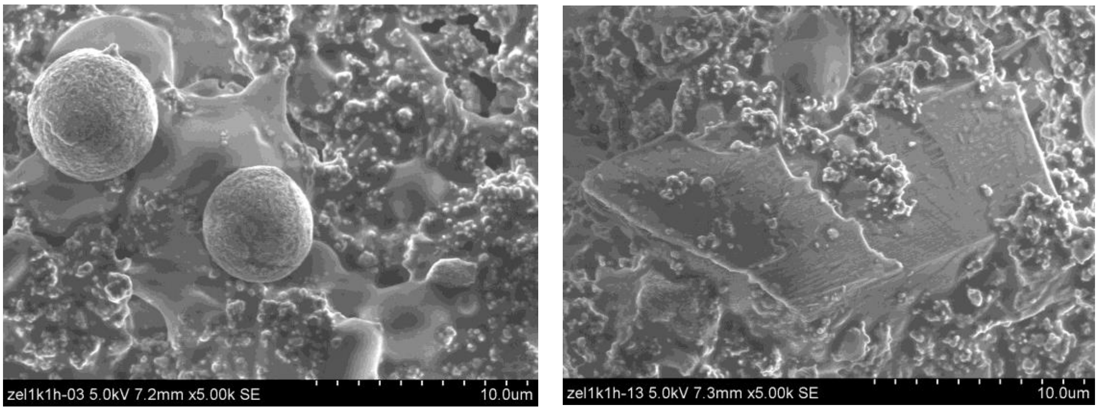

Fig. 16 Texture of sample S02, burned on 1,000 ^{o}C, magnification 5,000 times

3.9.2 SEM analysis of sample S17

3.9.2.1 SEM pictures, without heat treatment

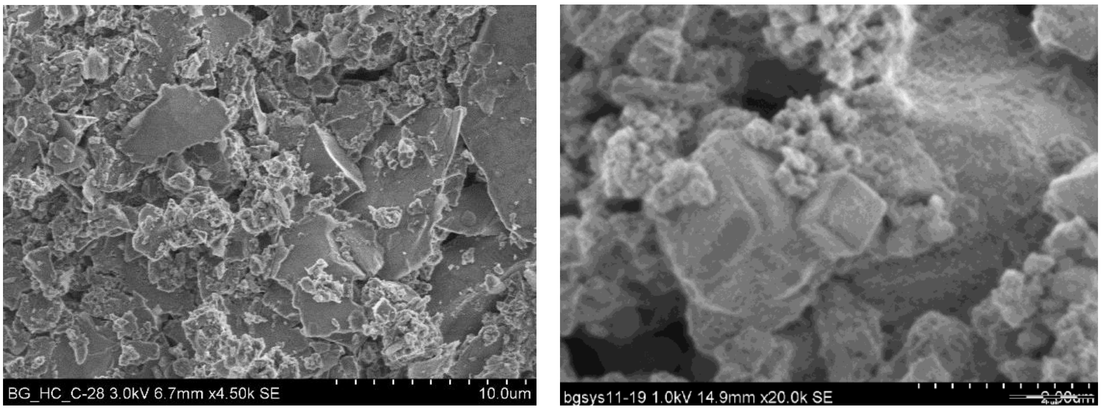

Fig. 17 Texture of sample S17, without heat treatment, magnification 4,500 and 20,000 times

3.9.2.2 SEM pictures, burned on 1,000 ^{o}C

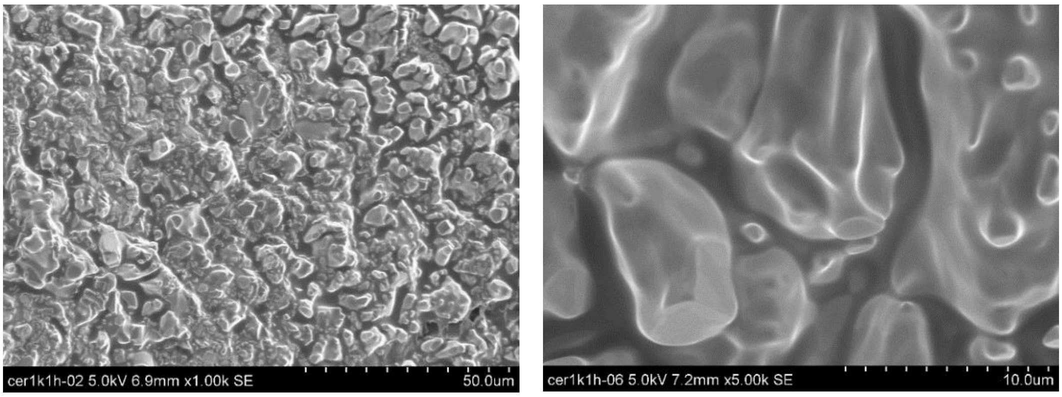

Fig. 18 Texture of sample S17, burned on 1,000 ^{o}C, magnification 1,000 and 20,000 times

3.9.3 Evaluation of SEM pictures

Both fibrous (wollastonite) and sphere particles (including chromium oxide) are visible in the pictures of the original material (S02) without heat treatment. The influence of high temperature caused more sintered surface morphology, only greater particles stood out above surface.

Both sphere and mainly plate-like particles of active ingredient are visible in the pictures of the new material (S17) without heat treatment. The influence of high temperature caused more sintered surface morphology, only greater particles stood out above surface.

Both materials show similar surface morphology after the heat treatment and from this point of view are comparable.

4. Conclusion

The objective of this research was to find a suitable active ingredient. It should have a positive effect on spectral normal emissivity of the resulting material especially in the range of short and medium wavelength of IR spectrum. At the same time other properties should be comparable to the original material.

In the begining active ingredients with known high value of emissivity (> 0.75) and simultaneously with high melting temperature (> 1,500 ^{o}C) had to be found (Iron, Silicon Carbide). Ingredients with unknown emissivity, but with high melting temperature, were also used (Chromium Oxide, Carbonyl Iron, Cerium Oxide, Magnesite, Magnetite, Barite, Kaolin, Aluminium Nitride, Boron Nitride, Zirkonium Oxide, Boron Carbide).

These results were found during spectrometric measurement:

- Carbonyl Iron and Nitrides showed the highest values of spectral normal emissivity, their values oscillated between 0.70 and 0.93 in monitored spectrum range, Nitrides had higher values approx. by 12 % in spectrum range over 1,500 cm^{-1} (under 6 μm),
- neither increase of content of active ingredient in filler, nor combination of the two active ingredients led to better emissive properties.

Active ingredient Nitrides (sample S17, in basic concentration 25 vol. %) was chosen as the ingredient with the best spectral normal emissivity. Its comparison with the original material (S02, Chromium Oxide) was executed through comparative tests. The tests were chosen with regard to practical implementation of the results in industry.

The new material (S17) was more resistant to high temperature. High power laser beam (4 kW) caused neither visible nor microscopic vestiges on the surface of the material.

The new material (S17) was more thermal conductive, between 10 and 37 % within the temperature range between 500 and 900 ^{o}C.

The new material (S17) was more adhesive to steel surface by 40 % for unburned coating and by 33 % for coating burned on 1,000 ^{o}C.

After burning on 1,000 ^{o}C the new material (S17) showed similar (sintered) surface morfology as the original material.

In conlusion it can be state that the reasearch accomplished all objectives. The effect of filler composition on the emissive properties in short and medium wavelenght range was demonstrated. Optimal active ingredient in the filler of the inorganic composite (BG HitCoat®, commercially used in industry) was determined. The active ingredient is a mixture of Aluminium Nitride and Boron Nitride (termed “Nitrides” in the paper) in volume ratio 1:1. The new product was subjected to comparative laboratory tests, in which it was fully comparable to the original material.

5. References

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6. Publications

Mauer, Kalenda, Honner, Vacíková
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