Thermal characteristics of enamels and enamelled metal sheets

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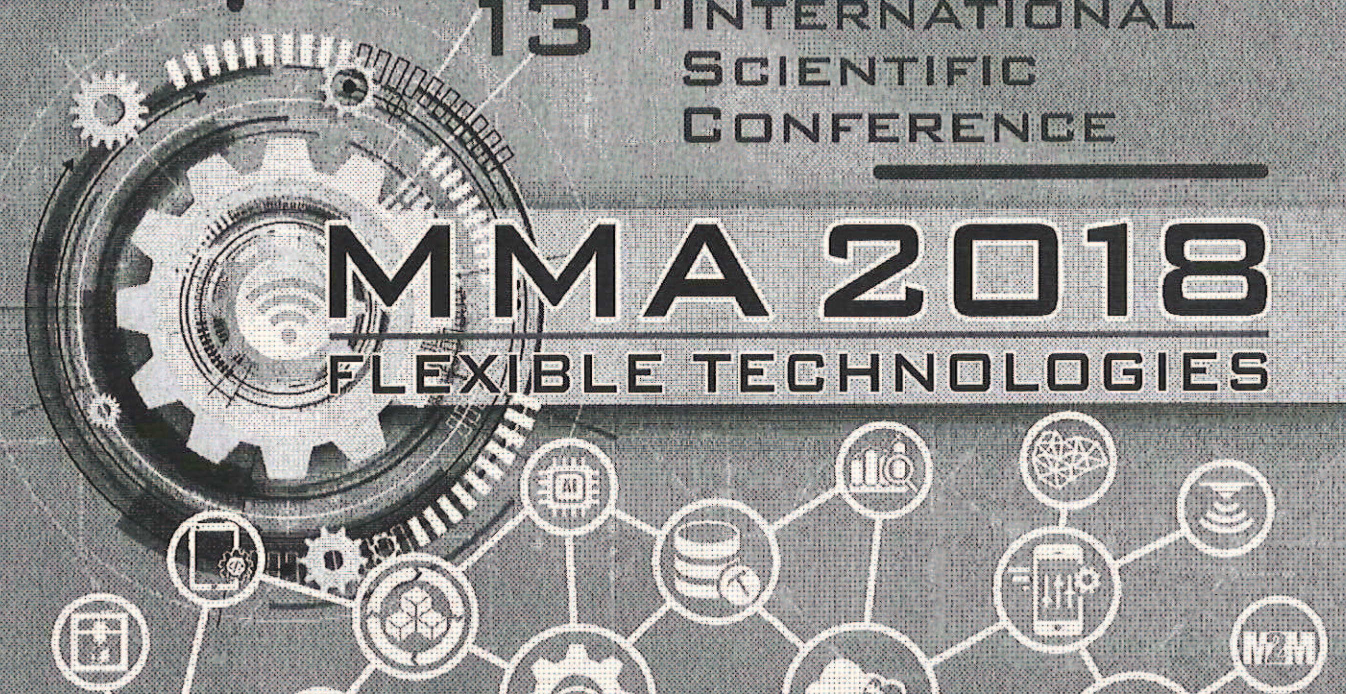

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THERMAL CHARACTERISTICS OF ENAMELS AND ENAMELLED METAL SHEETS

Abstract: *The main task of the research was to find out why sometimes cracking of the enamelled layer occur and to what extent enamel layer affects the heat transfer in the cooking oven. The samples of the enamelled streel sheet were metallographically analysed by scanning electron microscopy (SEM-EDX) and optical microscopy (OM). Measurements of the thermal properties of two types of enamels, which are used for oven interior enamelling was carried out in accordance with the standard ISO 22007-2 by transient planar heat source method (TPS Hot Disk 2200). To ensure proper measurement conditions, the samples of the enamels were prepared in the form of thick layers. The results show that enamel density has significant influence on its thermal properties. An assessment of the influence of the thermal conductivity of the enamel layer on the heat transfer in the oven is also given.*

Key words: *Thermal properties, Enamels, Enamelled steel sheet, TPS method, Measurement*

1. INTRODUCTION

Porcelain enamel is a glassy coating over the metal substrate, intended to improve its corrosion resistance, abrasion resistance, antibacterial characteristic or aesthetic appearance. Enamelling is a technological process in which a fine enamel powder is applied to the metallic substrate and heated to the melting temperature of the enamel (750-850°C), whereby the melt adhesively binds to the metal substrate and a solid, continuous glass coating is formed on the surface of the metal surface.

The enamels can be classified in several ways: depending on the substrate on which the enamel is applied, depending on the function they perform or according to the enamelling methods [1].

According to the metal substrate they can be classified as:

- Enamels for steel sheet
- Enamels for cast iron
- Enamels for aluminium
- Enamels for stainless steel and high-temperature resistant alloys
- Enamels for electronic applications
- Enamels for jewellery (precious metals, copper alloys)

According to the function they are designed for:

- Ground enamels that improve adherence between cover coats and substrate
- Cover coat enamels, which are further divided by the colour or by the certain physical and/or chemical property
- Direct enamels, which have the function of both ground and cover enamel

According to the enamelling technology:

- Dry electrostatic enamels
- Wet electrostatic enamels

Recently, the most commonly used industrial enamelling technology employs direct enamels, with one coat and one firing process or for some application ground and cover coat enamels with one unified firing process.

When selecting a steel for enamelling, it is important to consider the following factors: Basic ability for enamelling (enamelability), absence of surface defects, and resistance to sagging at enamelling temperatures, while other characteristic such as formability, weldability, strength, etc. are of primary concern of product manufacturing technology before enamelling or mechanical property requirements of the final product.

By the term enamelability of steel we mean the absence of excessive formation of gas bubbles that cause the boiling of the enamel. Evolution of gas, which can originate from steel and enamel as well, is caused by various reactions at the steel/enamel interface. With spectrographic analysis, it was found out that gases evolving when enamelling steel are carbon monoxide, carbon dioxide, hydrogen, water vapour, and nitrogen. The most detrimental gas evolving reaction is the oxidation of carbon in the steel surface and the formation of carbon monoxide. Therefore, steel with low carbon (usually below 0.08 wt.%) or carbon stabilized (with Ti or Nb) chemical compositions should be primarily selected. If high-carbon steel cannot be avoided, special surface preparation techniques must be applied before enamelling.

2. THERMAL CONDUCTIVITY MEASUREMENT

Transient planar heat source method (TPS, Hot disk AB®) was used for the measurement of the thermal properties of enamels. With this method, the thermal conductivity can be measured with an accuracy of ± 2 % and specific thermal capacity and temperature diffusivity with accuracy of ± 5% in accordance with ISO 22007-2 standard [2]. Thermal conductivity can be measured in the range from 0.01 to 500 $Wm^{-1}K^{-1}$, specific thermal capacity up to 5 $MJm^{-3}K^{-1}$ and temperature diffusivity from 0.1 to 300 $\mathrm{mm^2s^{-1}}$. The device is also equipped with a laboratory muffle furnace and allows the measurement of thermal properties up to 700°C in an inert atmosphere (Figure 1).

Fig 1. TPS 2200 Hot disk AB ® analyser.

The thermal analyser utilises a sensor element in the shape of double spiral (Figure 2), which acts both as heating element for increasing the temperature of the sample and as an electric resistance thermometer. The spiral is made by selective etching from 10 μm thick nickel foil, supported and electrically insulated on both sides with insulating foil. In the temperature range from sub-zero up to 300°C polyimide foil (Kapton® Du Pont) is used as insulating material, while above 300°C mica foil is used as insulating material. Before the measurement the sensor is sandwiched between two halves of the sample (solids) or embedded in the sample (liquids, powders). The main principle of the measurement system is that the sensor is first supplied with a constant electrical power which creates a dynamic temperature field in its surroundings, and then 200 x in succession measures the change in sensor electrical resistance at a selected time interval. Parameters like heating power, measurement time for electrical resistivity measurement and sensor radius are used to optimize the settings for the experiment.

Since we are not equipped with a module for measuring the thermal properties of thin insulating films, enamel samples were prepared in the form of an approximately 1 cm thick layer. To make such samples, first dies (9 x 6 x1.5 cm) from low carbon ferritic steel were made. Dies were filled with enamel powder and gradually heated to 850°C and 900°C for 10 minutes and then slowly cooled in the furnace [3].

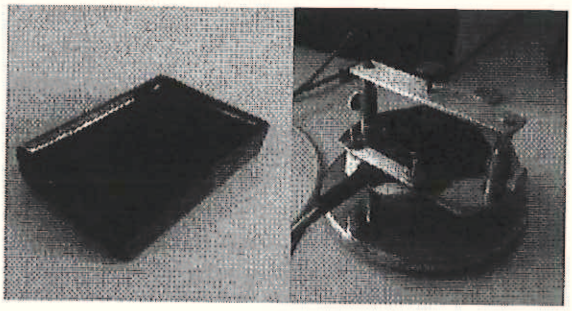

Fig 2. Thick enamel layer (left), sensor position (right).

Since enamel melts wet the steel surface well it is very difficult to make two identical samples with parallel free surfaces (non-concave), which would enable the insertion of the sensor between them without an air gap. Therefore, the measurements were carried out using a single-side method, where the sensor is "sandwiched" between the investigating sample and insulation material of known thermal conductivity (Styrofoam). Several measurements were performed on various areas of the individual sample.

Table 1. Thermal properties of enamel 1

Pos.	λ [$\mathrm{Wm^{-1}K^{-1}}$]	a [$\mathrm{mm^2s^{-1}}$]	Cp [$\mathrm{MJm^{-}3K^{-1}}$]
1	0.6821	0.4286	1.591
1	0.6845	0.4335	1.579
1	0.6852	0.4390	1.579
1	0.6840	0.4385	1.560
2	0.6667	0.4239	1.573
2	0.6678	0.4273	1.563
2	0.6683	0.4251	1.572

Table 2. Thermal properties of enamel 2

Pos.	λ [$\mathrm{Wm^{-1}K^{-1}}$]	a [$\mathrm{mm^2s^{-1}}$]	Cp [$\mathrm{MJm^{-}3K^{-1}}$]
1	0.8786	0.4410	1.992
1	0.8879	0.4626	1.919
1	0.8867	0.4588	1.932
1	0.8866	0.4588	1.932
2	0.8641	0.4267	2.025
2	0.8621	0.4311	2.000
2	0.8626	0.4309	2.002

Several samples prepared from the same enamel, but with different densities (due to firing temperature variation) were measured. Results show significant influence of density on the thermal conductivity of enamel. Tables 6 and 7 show results of thermal conductivity of individual enamel as a function of its density.

Table 3. Thermal conductivity as a function of enamel layer density (enamel 1)

ρ [$\mathrm{g/cm^3}$]	λ[$\mathrm{Wm^{-1}K^{-1}}$]
1.88 – 1.89	0.66 – 0.69
1.77 – 1.78	0.65 – 0.67
1.73 – 1.75	0.60 – 0.61

Table 4. Thermal conductivity as a function of enamel layer density (enamel 2)

ρ [$\mathrm{g/cm^3}$]	λ [$\mathrm{Wm^{-1}K^{-1}}$]
2.20 – 2.22	0.94 – 0.95
2.06 – 2.13	0.83 – 0.84

3. MICROSTRUCTURE INVESTIGATION

The microstructure of the enamel is shown in the figure 3. The results of micro-chemical analyses (EDX) of the areas marked in the figure 3 by squares are presented in the table 2.

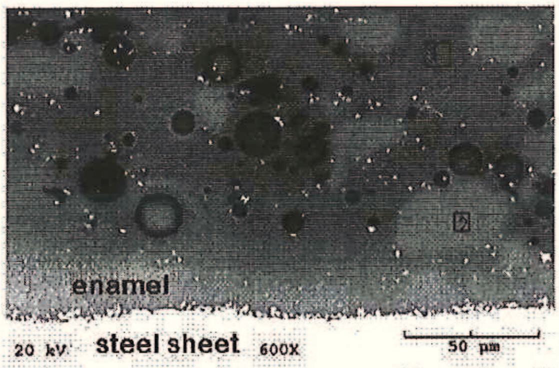

Fig 3. Microstructure of enamel layer (SEM).

Table 3. Microchemical analysis (EDX) of areas marked in Figure 3

Element	M. 1 wt. [%]	M. 2 wt. [%]	M. 3 wt. [%]
O	15.237	13.091	15.020
Na	5.298	4.471	-
Al	0.716	0.880	-
Si	47.904	45.043	84.980
K	5.923	6.181	-
Ca	4.852	2.717	-
Ti	6.638	2.961	-
Mn	3.749	1.328	-
Fe	6.038	8.537	-
Co	1.588	0.923	-
Cu	0.744	0.353	-
Zr	1.313	13.515	-

From the figure 3 can be seen that enamel layer is not homogenous, but consist of various crystal phases, pores, and amorphous areas with different chemical compositions. Brighter contrasted areas have much higher Zr content (added as ZrO), while darker contrasted areas contain a higher percentage of Ti and Mn (added as TiO_2 and MnO). Sharp dark phases are SiO_2 crystals, and spheres porosity. In addition, small bright particles located near the steel-enamel interface and in areas rich with titanium and manganese are also visible. Although the quantitative evaluation of the chemical composition with EDX microchemical analysis of such small analytical volumes is not accurate, it nevertheless points out the differences in chemical composition of the bright particles located near the steel/enamel interface and bright particles in the bulk enamel layer. Namely, particles in the enamel contain chromium while particles at the interface contain cobalt. The chromium was not detected in particles at the steel/enamel interface except for location 2 (Figure 4), which shows a different, more sharp morphology, so that the possibility of interaction between the particle already in the enamel and steel surface cannot be excluded. In addition, the particles in the enamel do not contain cobalt, which is present in all particles at the interface. Since the presence of chromium in enamels is not common [4], we analysed the enamel powders before the enamelling process.

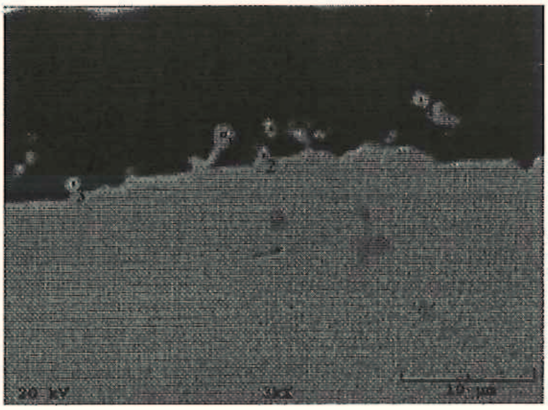

Fig 4. Steel/enamel interface (SEM).

Table 4. Microchemical analysis (EDX) of areas marked in Figure 4

El.	M. 1 wt. [%]	M. 2 wt. [%]	M. 3 wt. [%]	M. 4 wt. [%]	M. 5 wt. [%]
O	4.230	4.921	7.821	12.682	8.779
Na	3.159	2.642	5.300	4.532	4.919
Al	0.534	0.436	0.971	1.095	0.803
Si	15.438	11.630	25.410	35.872	27.692
K	1.079	0.805	1.943	3.215	2.191
Ca	1.278	0.869	2.202	3.409	2.701
Ti	1.346	1.624	2.472	3.847	2.817
Cr	-	4.615	-	-	-
Mn	0.636	0.867	1.328	2.358	1.677
Fe	63.106	65.821	48.264	28.678	43.851
Co	6.716	3.494	2.827	1.620	3.510
Cu	2.048	1.848	0.794	-	-
Zr	0.430	0.427	0.666	2.693	1.060

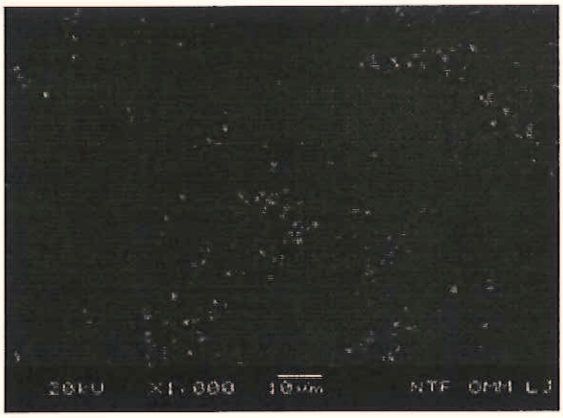

Fig 5. Bright particles on the enamel surface (SEM).

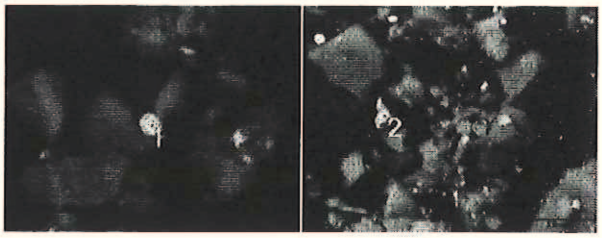

Fig 6. Bright particles in the enamel powder (SEM).

Table 5. Microchemical analysis of bright particles in enamel layer after firing (Figure 5) and in enamel powder before firing (Figure 6)

Element	M. 1 Fig. 5 wt. [%]	M. 2 Fig. 5 wt. [%]	M. 1 Fig. 6 wt. [%]	M. 2 Fig. 6 wt. [%]
O	6.025	5.319	0.484	11.61
Na	2.232	1.307	0.455	1.49
Al	1.163	0.253	0.45	0.997
Si	17.889	8.226	3.81	7.603
K	1.509	0.659	0.71	0.314
Ca	0.805	0.571	1.247	0.259
Ti	1.104	2.416	0.614	0.228
Cr	27.364	19.629	26.18	14.521
Cu	0.469	1.507	1.285	0.702
Fe	39.536	59.735	64.604	61.823
Zr	1.904	0.376	0.145	0.45

Figure 6 shows the enamel powder after final grinding, ready for application to the metal substrate. As we can see, in the powder is a noticeable number of shiny particles of similar size (most of which less than 1 μm) as detected in the enamel layer after firing. By microchemical analysis (EDX) was determined that these particles also contain higher concentrations of chromium and iron (Table 5). This is a proof that the particles found in the enamel layer after firing originate from the previous preparation of enamel powder and are not the result of chemical reactions during firing of the enamel.

4. HEAT TRANSFER THROUGH ENAMELLED STEEL SHEET

Heat transfer rate through enamelled steel sheet per unit of area can be calculated by equation:

$$U = \frac{\Delta T}{\frac{1}{\alpha_{out}} + \frac{\delta_{steel}}{\lambda_{steel}} + \frac{\delta_{en}}{\lambda_{en}} + \frac{1}{\alpha_{in}}} \; [\frac{W}{m^2}]$$

Where δ_{steel} and δ_{en} represents thickness of steel sheet and enamel layer, λ_{steel} and λ_{en} thermal conductivities of the steel sheet and enamel, α_{out} in α_{in} heat transfer coefficients (radiation + convection) on both sides of enamelled steel sheet, and ΔT temperature difference. For heat transfer coefficients in kitchen ovens values between 15 - 40$\mathrm{Wm^{-2}K^{-1}}$ can be found in the literature [5]. In the case of one side enamelled steel sheet (200 μm thick enamel layer + 1 mm thick steel sheet), individual thermal resistances can be evaluated.

Thermal resistances:

$$\frac{\delta_{steel}}{\lambda_{steel}} = \frac{0{,}001}{54} = 1{,}85 \cdot 10^{-5}\ \mathrm{m^2K/W}$$

$$\frac{\delta_{en}}{\lambda_{en}} = \frac{200 \cdot 10^{-6}}{0{,}3 \div 1{,}3} = 6{,}6 \cdot 10^{-4} \div 1{,}5 \cdot 10^{-4}\ \mathrm{m^2K/W}$$

$$\frac{1}{\alpha_{in/out}} = \frac{1}{15 \div 40} = 6{,}6 \cdot 10^{-2} \div 2{,}5 \cdot 10^{-2} \mathrm{m^2K/W}$$

We can quickly figure out that conduction thermal resistance of enamelled steel sheet is negligible factor compared to convective resistance if heat transfer coefficient at only one side is below 100 $\mathrm{Wm^{-2}K^{-1}}$. Even if we consider extreme values of heat transfer coefficients (500 $\mathrm{Wm^{-2}K^{-1}}$) on both sides of enamelled steel sheet and calculate the heat transfer rate with limited values of enamel thermal conductivity (0,3 or 1,3 $\mathrm{Wm^{-1}K^{-1}}$) the difference in heat transfer rate will be less than 13%. However, in the case of kitchen ovens, such values of heat transfer coefficients will never be achieved.

5. CONCLUSIONS

The thermal properties of the enamel are mostly affected by the density of the enamel. The greater the density, the higher will be thermal conductivity and the specific volume heat capacity, which is in accordance with the theory. Namely, gases exhibit at least an order of magnitude lower thermal conductivity than solids. The density of the enameled layer depends on the chemical composition of the enamel and the process parameters of the enameling which influence the amount of porosity.

Enamel layer has a multiphase structure composed of amorphous areas with different chemical composition, crystal phases (SiO_2, metal particles) and pores. Certain crystalline phases (metal inclusions) found in the enamel layer after firing originate from the process of preparing the powder (grinding, sieving) or are intentionally added. In all analyzed enamel powders, the metallic inclusions were detected, which have a similar chemical composition and size as metal inclusions located in the enamel layer after firing. If these particles agglomerate during firing they will cause cracking of the enamel layer.

Microstructural observations enable precise monitoring of the changes in the material at any technological stage and allow faster detection of the defect origin.

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