A new approach for quantifying thermal barrier effect. Case study: RE_20 enamel

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DOI: 10.13111/2066-8201.2020.12.4.17

Received: 15 October 2020/ Accepted: 20 November 2020/ Published: December 2020

International Conference of Aerospace Sciences "AEROSPATIAL 2020", Virtual Conference
15-16 October 2020, Bucharest, Romania,
Section 4 - Materials and Structures

Abstract: *Thermal barrier coatings (TBC) are designed to reduce the temperatures at the surface of metallic hot working pieces. A new refractory enamel, denoted RE_20, was developed at INCAS SA. The RE_20 is designed to protect pieces made of EI 468 superalloys. Thermal barrier effect (TBE) is frequently used in the literature to refer to the temperature decreasing at the heat exposed surface, but it is not defined as a quantitative measurand. The paper aims to mitigate this shortcoming by introducing two measurands for a proper assessment of the TBE i.e. relative temperature decreasing (RTD) and relative heat flow decreasing (RHD). The TBE depends on the working temperature, therefore it has to be estimated at elevate temperatures in the 900-1100°C range. This is possible only through thermal diffusivity flash method (ThDM) which facilitates measurements up to 1000°C. Two mathematical models were derived for RTD and RHD. The models were applied to the RE_20/ EI868 systems for two cases: as obtained and as cyclic thermal shock tested at 900°C. The paper addresses* *the following novelties: two new measurands (RTD, RHD), the TBE behavior depending on the working temperature and the TBE dependance on upper temperature of the thermal shock.*

Key Words: *thermal barrier coatings, thermal barrier effect, thermal diffusivity flash method, refractory enamel, turbo reactor engine, thermal shock test*

1. INTRODUCTION

The metallic surfaces of the turbo reactor pieces which come in contact with the hot burned gasses are subjected to high temperature corrosion (900 - 1200ºC) and to erosion due to high speed (~ 1 Mach) solid particle contained in the liquid fuel i.e. pyrolytic carbon, etc. [1-5]. The refractory steels or superalloys developed for aircraft engines so far, have a reduced lifetime in the above mentioned work conditions due to a very severe corrosion action of S, Na, V, P, Ca, Fe, Mg etc. at temperatures over 900ºC in spite of the fact that their concentrations in the burned gas are of the ppm orders (1 ÷15ppm) [1-6].

Thus, the coating of these surfaces becomes a must and there are only a few practical solutions in this case: metallization, plasma - spray coating and enameling. The last solution seems to be the best because it has the highest efficiency/ cost ratio. The enamel coatings can increase the working time of the hot working pieces at least two times because they protect the surfaces against the majority damage factors such as: erosion, hot corrosion- being impenetrable by hot gas, - and thermal shocks. Another very important role played by enamel coatings is the thermal barrier i.e. the decrease in temperature at the surface of the metallic parts that have been exposed to high temperature. The thermal barrier effect (TBE) is usually interpreted as the resistance against heat flow through a body or a wall. In case of a TBC it must be understood as a matter of decreasing the temperature of the metal inner surface due to the applied coat. Nevertheless, there is no well-defined measurand assigned to the TBE.

Consequently, this paper presents some considerations on the quantitative estimation of TBE through two parameters:

1. Relative Temperature Dropping caused by enamel, (RTD);
2. Relative Heat flux Decreasing through engine walls, denoted as RHD.

To our present knowledge, there is no direct method to measure the TBE and the RHD, therefore they must be estimated from indirect measurements. In this regard, we address the thermal diffusivity measurement method (ThDM). The paper addresses two main novelties i.e.
i) the concept of complex TBE consisting in RTD and RHD for quantification of TBE;
ii) the RTD and RHD quantifications based on ThDM.

2. THEORY: THE NEW CONCEPT OF COMPLEX THERMAL BARRIER EFFECT (CTBE)

The simplest model of describing the RTD is shown in Fig. 1 a, b. Hence, if a piece of metallic sheet (combustion chamber, nozzle, volet etc., is unprotected and exposed to hot flue gases, it will get to a T_1 temperature in a very short amount of time i.e. it will suffer a thermal shock which consists in a rapid heating from ambient temperature to a higher T_1. (Figure 1. a).

T_1 value is lower than the temperature of the burned gases, as the combustion chamber outer walls or of the other hot parts are air-cooled.

The dynamic equilibrium of heat flow through this wall will assume a T_1 temperature on the inner surface and a T_2 on the outer surface.

If the part is protected (Figure 1.b) then the temperature of the internal surface of the substrate will be T_i, obviously with $T_i < T_1$.

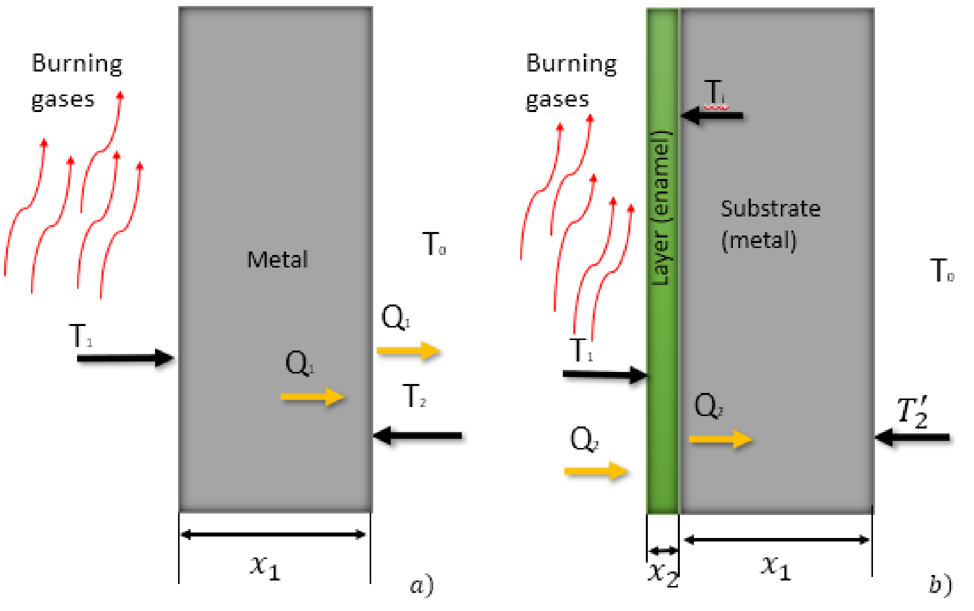

Fig. 1 – Schematic representation of the heat transfer through a wall of a combustion chamber: a) without enamel layer; b) with enamel layer

In this frame, RTD can be defined as:

$$RTD = \frac{T_1 - T_i}{T_1 - T_2} X100\% \tag{1}$$

where T_1 is the temperature of the inner surface of the enamel, T_i is the temperature at enamel-substrate interface and T_2 is the temperature of the outer cooled surface of the metallic wall (Fig. 1.b).

According to the Fourier law and to the geometry in Fig. 1.a, the unit heat flow through the combustion chamber wall ($\dot{q}_o$), if the substrate is uncoated, is derived as [7]:

$$\dot{q}_o = \frac{(T_g - T_o)}{\frac{1}{\gamma_1} + \frac{x_s}{\lambda_s} + \frac{1}{\gamma_2}} \tag{2}$$

where T_g is the temperature of hot gasses; To is the temperature of the coolant air jet; λ_s is the heat conductivity of the substrate, x_s is the substrate thickness; γ_1 is the heat transfer coefficient from hot gasses to the wall; γ_2 is the heat transfer coefficient from the rear part of the wall to the coolant air.

In case of the inner part coated with an enamel layer, the expression of the unit flux heat will become:

$$\dot{q}_1 = \frac{T_g - T_o}{\frac{1}{\gamma_1} + \frac{x_e}{\lambda_e} + \frac{x_s}{\lambda_s} + \frac{1}{\gamma_2}} \tag{3}$$

where x_e is the enamel thickness; λ_e is the thermal conductivity of the enamel.

Based on the assumption that $\dot{q}_1$ has a steady state flow and on Eq.(3), the temperature drop across the enamel was derived as:

$$T_1 - T_i = \left(T_g - T_o\right) * \frac{\frac{x_e}{\lambda_e}}{\frac{1}{\gamma_1} + \frac{x_e}{\lambda_e} + \frac{x_s}{\lambda_s} + \frac{1}{\gamma_2}} \tag{4}$$

The temperature drop across the covered wall was derived as:

$$T_1 - T_2 = \left(T_g - T_o\right) * \frac{\frac{x_e}{\lambda_e} + \frac{x_s}{\lambda_s}}{\frac{1}{\gamma_1} + \frac{x_e}{\lambda_e} + \frac{x_s}{\lambda_s} + \frac{1}{\gamma_2}} \tag{5}$$

According to Eq. (1), the mathematical expression of the TBE is:

$$TBE = \frac{T_1 - T_i}{T_1 - T_2} = \frac{\frac{x_e}{\lambda_e}}{\frac{x_e}{\lambda_e} + \frac{x_s}{\lambda_s}} = \frac{\frac{\lambda_s}{\lambda_e} * x_e}{x_s + \frac{\lambda_s}{\lambda_e} * x_e} \tag{6}$$

The Eq. (6) gives the theoretical base for RTD estimation provided that the values of λ_e, λ_s, x_e and x_s are known.

Unfortunately, λ_e cannot be solely measured, but only in a mixed manner i.e. as a sandwich as is depicted in Fig. 1.b. Besides, the values of λ_e and λ_s depend on *T* and, generally, they decrease as *T* increases [6]. Because the enamel is designed to work at higher temperature, the values of λ_e and λ_s must also be measured at elevate temperatures (900^{o}C-1200^{o}C). The only method fitted to thermal characteristics measurement at higher temperatures is ThDM [6, 8-11].

In this case, the ThDM provides an effective thermal diffusivity coefficient, denoted hereafter as α_E. The thermal diffusivity coefficient (α) is defined as:

$$\alpha = \frac{\lambda}{\rho c_P} \tag{7}$$

where ρ is the mass density and c_P is the specific heat capacity of the material at constant pressure.

Note: For the sake of simplicity, the symbol c will be use instead of c_P, hereafter.

In the case where $\dot{q}_1$ has a steady flow without temperature jumps at interface then the following relationships can be written:

$$\dot{q}_1 = \frac{\lambda_E(T_1 - T_2)}{x_s + x_e} = \frac{\lambda_e(T_1 - T_i)}{x_e} = \frac{\lambda_s(T_i - T_2)}{x_s} \tag{8}$$

which leads to the expression of λ_E:

$$\lambda_E = \frac{x_s + x_e}{\frac{x_e}{\lambda_e} + \frac{x_s}{\lambda_s}} \tag{9}$$

As λ_E and λ_s are accessible by measurements it is convenient to express the ratio λ_s / λ_e as a function on λ_E and x_s and x_e as follows:

$$\frac{\lambda_s}{\lambda_e} = \frac{\lambda_s}{\lambda_E}\left(\frac{x_s}{x_e} + 1\right) - \frac{x_s}{x_e} = \frac{\alpha_s \cdot \rho_s \cdot c_s}{\alpha_E \cdot \rho_E \cdot c_E}\left(\frac{x_s}{x_e} + 1\right) - \frac{x_s}{x_e} \tag{10}$$

where α_E, ρ_E and c_E are the effective values of the parameters assigned to the coated wall.

The expressions of the ρ_E and c_E were derived as:

$$\rho_E = \frac{\rho_s x_s + \rho_e x_e}{x_s + x_e} \tag{11}$$

$$c_E = \frac{c_s \rho_s x_s + c_e \rho_e x_e}{\rho_s x_s + \rho_e x_e} \tag{12}$$

The expression of RTD obtained by substituting the Eqs.(8-10) in Eq.(4) is:

$$RTD = \left[1 - \frac{\alpha_E \cdot (c_s \rho_s x_s + c_e \rho_e x_{e)} . x_s}{\alpha_s \cdot \rho_s \cdot c_s * (x_s + x_e)}\right] X\ 100\% \tag{13}$$

Eq.(13) links the RTD concept with thermal diffusivity flash measurements in an engineering manner i.e. making use of common sense hypotheses and of accessible math.

If the wall is not coated with enamel on the inside part, the unit heat flux through it is:

$$\dot{q}_o = \frac{T_g - T_o}{\frac{1}{\varepsilon_1} + \frac{x_s}{\lambda_s} + \frac{1}{\varepsilon_2}} \tag{14}$$

The relative percentage decrease in heat loss through the wall, if it is coated, is defined as RHD, i.e:

$$RHD = \frac{\dot{q_0} - \dot{q_1}}{\dot{q_0}} = \frac{x_e * \frac{\lambda_s}{\lambda_e}}{\frac{\lambda_s}{\gamma_1} + \frac{\lambda_s}{\gamma_2} + x_s + x_e * \frac{\lambda_s}{\lambda_e}} X\ 100\% \tag{15}$$

RHD is linked to the flash method through Eq.(15) as follows:

$$HBE = \frac{\frac{\alpha_s \cdot \rho_s \cdot c_s}{\alpha_E \cdot \rho_E \cdot c_E}(x_s + x_e) - x_s}{\frac{\lambda_s}{\gamma_1} + \frac{\lambda_s}{\gamma_2} + \frac{\alpha_s \cdot \rho_s \cdot c_s}{\alpha_E \cdot \rho_E \cdot c_E}(x_s + x_e)} = 1 - \frac{\frac{\lambda_s}{\gamma_1} + \frac{\lambda_s}{\gamma_2} + x_s}{\frac{\lambda_s}{\gamma_1} + \frac{\lambda_s}{\gamma_2} + \frac{\alpha_s \cdot \left(1 + \frac{x_e}{x_s}\right)^2}{\alpha_E \cdot \left(1 + \frac{\rho_e c_e x_e}{\rho_s c_s x_s}\right)} * x_s} X\ 100\% \tag{16}$$

Eq.(16) can be written more compact and in a more meaningful form as:

$$RHD = \frac{(\xi - 1)}{\xi + \omega}\ X\ 100\% \tag{17}$$

where ξ and ω are non-dimensional parameters:

$$\xi = \frac{\alpha_s \cdot \left(1 + \frac{x_e}{x_s}\right)^2}{\alpha_E \cdot (1 + \frac{\rho_e c_e x_e}{\rho_s c_s x_s})} \tag{18}$$

$$\omega = \frac{\lambda_s}{x_s}\left(\frac{1}{\gamma_2} + \frac{1}{\gamma_2}\right) \tag{19}$$

The ξ depends on α_s, α_E, ρ_s, ρ_E, c_s, c_e but critically on x_e/x_s, which is a small quantity of the 0.02 order.

As the value of ξ can be calculated based on available data such as ρ_s, ρ_E, c_s, c_e and on data obtained by measurement (α_s, α_E), the RHD can be quantitatively assessed, which provides a powerful instrument for ranking TBCs, and also for quantitative assessment of the progress in the TBC research field.

3. MATERIALS AND METHODS

3.1 Basic tests on EI_ 868 superalloy and enamel

The ThD measurements were carried on micro-composite enamel, denoted RE_20, that coated pieces of EI_868 super alloy sheet.

The elemental composition of the substrate, measured with a SpectromaxX SDAR-OES spectrometer is shown in Table 1.

The expanded uncertainty having 95% confidence level, U (95%), assigned to the measurement data is presented in Table 1. The prescribed composition of EI_ 868 superalloy is given in the first row of Table 1 [12].

Table 1 – Elemental composition of the substrate [%] mass

Element	C	Si	Mn	Cr	Ni	Mo	Fe	W	Al	Ti	S	P
EI_868 [12]	≤ 0.10	≤ 0.80	≤ 0.50	23,50 -26,50	25.0 -30.0	≤ 1.50	≤ 4.0	13.00 -16.00	≤ 1.50	≤ 4.0	≤ 0.013	≤ 0.013
c	0.12	0.39	0.33	23.53	25.52	1.10	2.41	14.00	0.30	0.66	0.004	0.007
U(95%)	0.04	0.08	0.06	0.04	0.04	0.08	0,10	0,6	0,08	0,12	0,002	0,006

The comparative analysis of the data in Table 1 shows that the EI_868 alloy is not compliant regarding the concentration of C.

However, if the expanded measurement uncertainties are considered, it can be stated that the substrate composition corresponds to the superalloy predicted category.

Samples of_ EI 868 sheets (50×25×1.2 mm) were coated with RE_20 on a single side using wet spray technique.

The barbotine coated specimens were fired at 1320ºC for 3.5 mins in an electrical furnace.

The oxidic composition of the enamel coatings measured with Xepos XRF spectrometer is given in Table 2.

Table 2 – The oxidic composition of the RE_20 enamel

SiO_2	BaO	Cr_2O_3	B_2O_3	Al_2O_3	CaO	MgO	ZnO	Mo_2O_3	NaO
27.6	20.7	27.6	2.3	2.1	2.8	1.7	3.1	2.1	10.0

The ThDM were carried on 3 types of specimen i.e. uncoated EI_868 sheet, considered as reference specimen, enamel coated specimen (Fig. 2.i.a) and cycled thermal shocked specimen at 900ºC (Fig. 2.i.b) [13].

A thermal shock test consists of a sharp transition of the specimen into a furnace chamber from the room temperature to 900ºC, then maintained for 5 mins at this temperature, followed by a transition from the furnace into a cool air jet, which cools down the specimen at room temperature in 3 minutes.

This process was repeated automatically 200 times. The enamel has a thickness of about 50 μm and o micro-composite structure (Fig. 2.ii, iii).

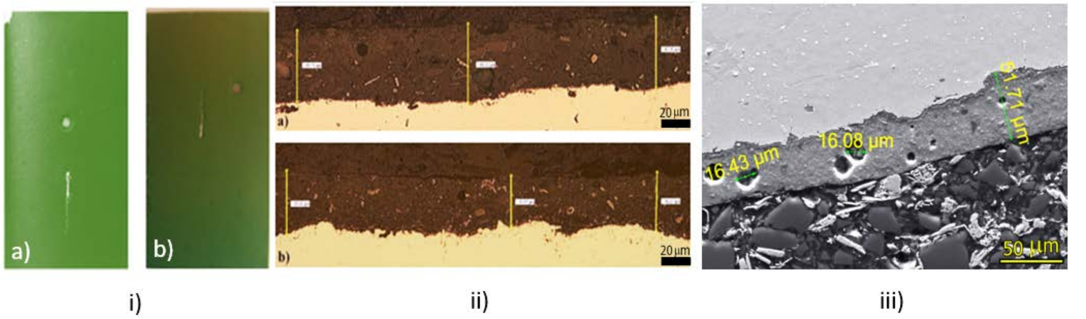

Fig. 2 – Images of the specimens that undergone ThDM: i): a) macrostructure of the uncycled specimen; i): b) macrostructure of the thermal shocked specimen; ii): a) microstructure of the uncycled specimen; ii): b) microstructure of the thermal shocked specimen; iii) SEM images of a cross-section through the thermal shocked specimen at 900°C

The thermal shock tests do not modify in a significant manner the structure of the enamel coatings as is depicted in Fig. 2.

3.2 Thermal Diffusivity Flash Method

As was pointed out above, the CTBE can be properly estimated based only on thermal diffusivity measurement.

Accordingly, the main features of the ThDM are presented below. Thus, in a vertical setup (Fig. 3), a light source (flash lamp) heats the sample from the bottom side and a detector on top detects the time-dependent temperature rise.

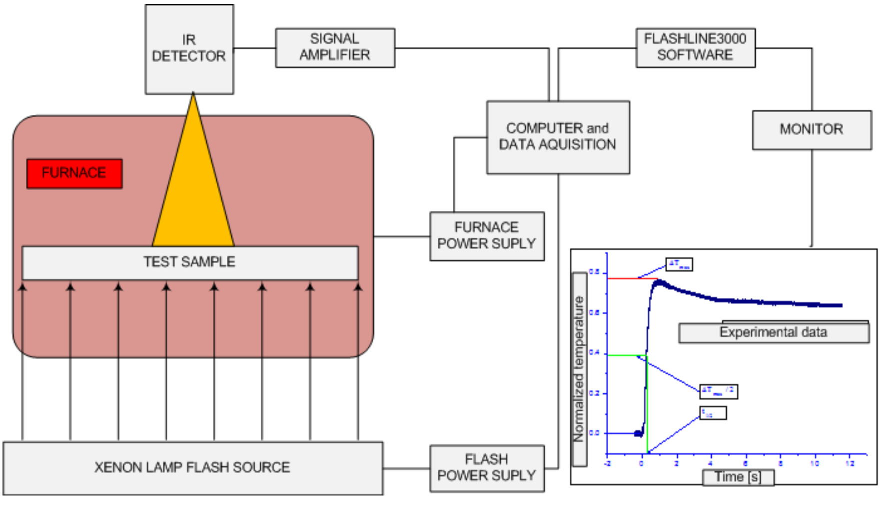

Fig. 3 – Schematic representation of the ThDM [11]

The heat pulse is uniformly distributed over the specimen surface, and it is absorbed by a layer of material which is very thin in comparison to the thickness of the sample. The thermal diffusivity is determined from the time required to reach one-half of the peak temperature.

The heat pulse duration is negligible compared to the thermal response of the specimen. The parameters that contribute to the calculation of the thermal diffusivity value (α) are: the thickness of the sample (*L*), the time required for the temperature of the upper part of the sample to increase until reaching a percentage of 50% of its maximum value, denoted $t_{1/2}$. The thermal diffusivity coefficient (α) is calculated as [8, 11]:

$$\alpha = 1{,}338 t_{1/2}/L^2 \tag{20}$$

Thermal diffusivity measurements were carried with a FlashLine 3000 thermal system equipment for measuring diffusivity using the “flash” method according to ASTM E1461-13 standard.

The tests were performed at the Thermophysical Properties Research Laboratory at Materials Science Faculty, The “Politehnica” University of Bucharest, Romania.

The FlashLine 3000 is equipped with an electrical furnace which facilitates the measuring of the α at different temperatures in the [25, 750] ºC range. Thus, the α was measured for the following temperatures: 125, 230, 341, 447, 555, 659 and 746. 851, 947°C.

The procedure consists in exposing the sample with dimensions of 0.7X0.7 mm for 1h at a prescribed temperature followed by triggering a pulse of radiant energy of high intensity and of short duration emitted by a Xenon lamp with light power of 1W.

At each temperature, five tests were performed in repetitive conditions,/. Each repeated test was carried out with a time gap of 30 minutes as to ensure that the specimen has reached the thermal equilibrium with the furnace chamber.

The average value of the 5 repeated tests at each temperature is reported with the standard deviation calculated based on these results.

4. RESULTS AND DISCUSSIONS

The thermograms provided by a FlashLine 3000 for the 3 specimens under study have a profile similar to that shown in Fig 4.

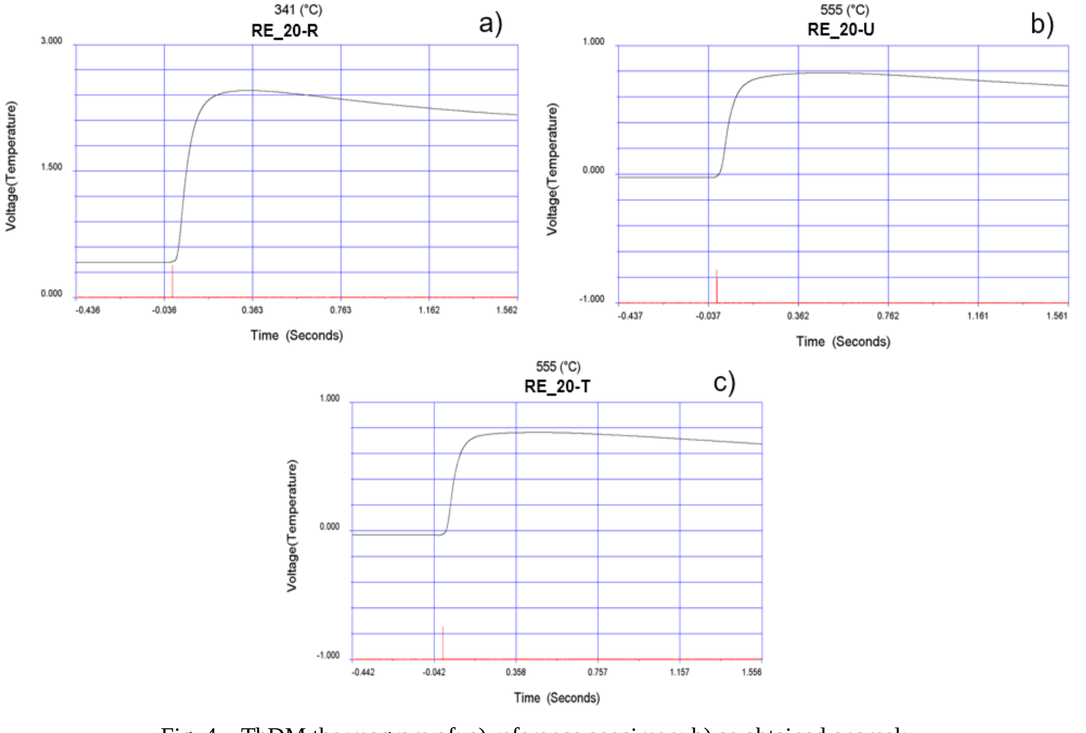

Fig. 4 – ThDM thermogram of: a) reference specimen; b) as obtained enamel; c) thermal shocked enamel at 900°C

Table 3. – The outcomes of the ThDM measurements on reference (R), untested (U) and tested specimens

Specimen	Thickness [mm]	T [ºC]	(α) α_E [cm²/s]	Standard deviation [cm²/s]
RE_20-R	1.19	125	0.0287	0.0003
		230	0.0324	0.0002
		341	0.0365	0.0002
		447	0.0403	0.0002
		555	0.0435	0.0004
		659	0.0459	0.0006
		746	0.0482	0.0005
		851	0.0515	0.0004
		947	0.0547	0.0006
RE_20-U	1.23	125	0.0287	0.0003
		230	0.0324	0.0002
		341	0.0365	0.0002
		447	0.0403	0.0002
		555	0.0435	0.0004
		659	0.0459	0.0006
		746	0.0482	0.0005
		851	0.0515	0.0004
		947	0.0547	0.0006
RE_20-T	1.23	125	0.0287	0.0003
		230	0.0324	0.0002
		341	0.0365	0.0002
		447	0.0403	0.0002
		555	0.0435	0.0004
		659	0.0459	0.0006
		746	0.0482	0.0005
		851	0.0515	0.0004
		947	0.0547	0.0006

Comparative analysis of the graphs in fig. 5 shows that the thermal diffusivity values of the tested specimen are greater than that of the untested specimen, but smaller than that of the reference specimen.

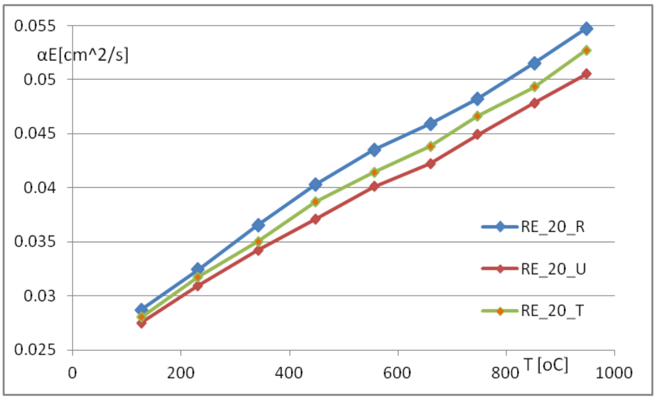

Fig. 5 – The α dependence on temperature of: a) reference specimen, blue line; b) as obtained enamel, red line; c) thermal shocked enamel at 900°C, green line

From the α_E point of view, the thermal shocked specimen behaves better than the substrate, but worse than the unshocked one (Fig. 5).

Based on the achieved data it is foreseen that at about 1000°C all samples have a thermal conductivity at least twice higher compared to the values obtained at room temperature, which is bad news!

The RTD and RHD for different temperature were estimated based on data given in Table 4 and on measurement data α_s and α_E given in Table 3.

Table 4. – The outcomes of the ThDM measurements on reference (R), untested (U) and tested specimens [6, 7, 11, 12]

ρ_S [kg/m^3]	ρ_e [kg/m^4]	c_S J/kg*K	c_e J/kg*K	λ_s W/mK	x_S µm	x_e µm	γ_1 J/m^{2}s	γ_2 J/m^{2}s
7950	2.7	640	840	21	1200	60	650	650

The RTD values for the 750-1000°C temperature range is quite constant i.e. 4. 76% (Table 5) whilst the RHD is more efficient at 75°C compared to higher temperature. However, the RHD takes smaller values in the 750-1100°C range.

Table 5. – TBE and RHD values corresponding to data given in Table 4 and Table 3

	RE_20-U		RE_20-T	
T[°C]	TBE[%]	RHD[%]	TBE[%]	RHD[%]
750	4.760	0.042	4.650	0.036
850	4.761	0.037	4.662	0.031
900	4.762	0.032	4.673	0.030
950	4.764	0.031	4.686	0.029

Using the data given in Table 4 and the α_s and α_E given in Table 2 for the specimen undergone thermal shocks at 900°C, a simulation was performed for the case of increased enamel thickness from 50 to 200 µm (Fig.6).

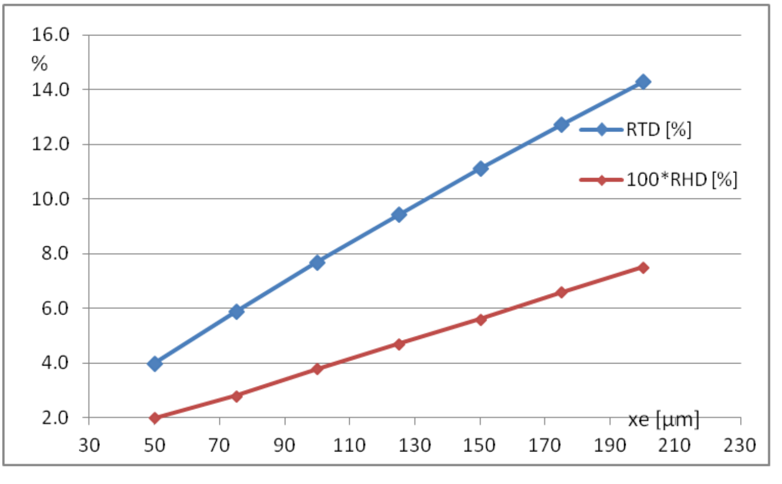

Fig. 6 – The CTBE dependence on enamel thickness of the specimen tested at 1100°C

Fig. 6 shows that RTD increases linearly as enamel thickness increases and RTD is greater than 10% for thickness greater than 125µm. The RHD is less than 0.1% for all the above mentioned thickness range.

Thus, our derived RTD indicates an enamel coating thickness greater than 125μm for a significant RTD.

Due to physical and technological constrains, the enamel thickness must be kept less than 100μm, therefore the researches must be conducted toward decreasing the thermal conductivity of the enamel as to improve its TBE.

Our derivation shows that RHD takes smaller values. Also, the RTD increasing leads to an increasing in RHD.

5. CONCLUSIONS

A new complex measurand is proposed for quantitative estimation of the thermal barrier effects of a TBC i.e. CTBE=[RTD, RHD], especially for quantifying CTBE at elevated temperatures.

The CTBE could be implemented only through thermal diffusivity method designed to operate at higher temperatures (900-1200°C).

Our measurements show that the thermal diffusivity value of the enamel-EI 868 system increases with the working temperature.

The TBE values for an enamel-EI 868 systems with enamel thickness of 60 μm and substrate thickness of 1200μm is approximately 4.7%, while RHD <0.1% for the [750-1000°C] temperature range.

The values of the RTD and RHD increase with the thickness of the enamel. The enamel thickness must be kept less than 100μm due to physical and technological constrains, therefore for improving of CTBE it is necessary to decrease the thermal conductivity of the enamel by changing its composition.

We consider that further researches are necessary to improve the accuracy of the input data used in the CTBE model.

Also, the enamel thermal conductivity must be reduced by changing its elemental composition, but mainly its phase composition.

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