

INFLUENCE OF CYCLIC HEAT STRESS ON THE PROPERTIES OF PLASMA-SPRAYED CERAMIC COATINGS

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The paper analyzes the contemporary knowledge by the blasting as the optimal technology for creation of the substrate surface. At the same time, the study support the evaluation of the character of the blasted surface related to the adhesion of the post-applied coatings. Moreover, the study deals with the creation and the characteristics of the ceramic oxide coatings especially used in the conditions of the thermo cyclic loading. In the end of the study, there is analysis of structural changes and crack propagation in the process of the thermal loading at the coatings based on Al_2O_3 and $ZrSiO_4$, which indicates the convenience of investigated coatings for concrete loading conditions.

Key words: Ceramic coatings, plasma spraying, thermal load, structure, adhesion

Utjecaj cikličkog toplinskog naprezanja na svojstva plazmom prskanih keramičkih premaza. Rad analizira današnje znanje o rasprskavanju kao optimalnoj tehnologiji za stvaranje površine supstrata. Istovremeno, istraživanje podupire procjenu osobine prskane površine s obzirom na adheziju naknadnih premaza. Štoviše, istraživanje se bavi stvaranjem i svojstvima keramičkih oksidnih premaza naročito korištenih u uvjetima termocikličkog opterećenja. U kompjuterskim grafovima elaborata dana je analiza strukturalnih promjena i širenja pukotina u procesu termalnog opterećenja na premaze koji se baziraju na Al_2O_3 i $ZrSiO_4$ što pokazuje prikladnost ispitanih premaza za uvjete stvarnog opterećenja.

Ključne riječi: Keramički premazi, prskanje plazmom, termalno opterećenje, struktura, adhezija

INTRODUCTION

For the plasma-sprayed coatings the layered (sandwich-like) structure is typical, formed as a result of a strong deformation and rapid crystallization of sprayed particles. The layered structure gives the coating a certain degree of elasticity and increases its thermal stability during thermal cycling. It should be noted that the properties of the sprayed coating differ from those of the same material in the initial state. [1,2,3]

Particle temperature and their velocity during spraying depend on the type of plasma gas and on the technological parameters of spraying and, of course, on the material and the granulometric composition of the powder being sprayed.

The surface of the substrate just is pre-treated. One of the most widely used and progressive methods of surface pre-treatment under plasma sprayed coatings is blasting. This type of pre-treatment serves two purposes, i.e. cleaning of the substrate surface (this obtaining the so-called juvenile surface) [4,5] and its roughening leading to better „anchoring” of the coating into the substrate.

For blasting under plasma sprayed coatings sharp-edged blasting material is suitable – corundum, steel or cast-iron grit, but also some non-conventional materials, like slag (by-product in Fe, Cu production). [6,7] Surface blasted by a sharp edged blasting material shows non-oriented character with sharp peaks. The heat transfer from melted particles impacting on the peaks of such a rough surface to the substrate is relatively slow and at certain combinations of substrate-coating materials conditions are created for formation of chemical bonds which results in higher values of adhesion. The material most widely used for plasma spraying is oxide ceramics. [8,9] This material consists entirely or predominantly of one refractory oxide. Some basic materials are present in nature in the form of oxides, other are prepared chemically or thermal decomposition. Properties of the coatings substantially differ from those of the basic materials. This is caused by a number of factors entering into action during the flight of the sprayed particle through the plasma jet and during the coating formation.

The obtain refractory properties it is necessary that the coatings have high density and good thermal conductivity. Surrounding atmosphere should not penetrate into the coating. On the contrary, to obtain good ther-

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mal-insulation properties it is necessary to form porous coatings with low thermal conductivity

The surrounding atmosphere can penetrate through some coating of the above-mentioned type down to the surface of the basic material. This result is an oxidation of the substrate and subsequently in the breakdown of the coating-substrate bond. Therefore, to obtain refractory and thermal – insulating coatings an interlayer is deposited, using materials forming a barrier between the coating and the substrate. For the above-mentioned coatings it is necessary to use refractory materials which are stable at high temperatures. For applications characterized by frequent temperature changes the porous coatings are suitable. When, in addition, such a coating is subjected to the influence of a high velocity flow, it is necessary to increase its density. [10,11] For some applications thicker coatings may be needed, but the adhesion of the coating to the substrate has always to be considered. In addition a thick coating may separate due to thermal deformations occurring during heating as a result of different thermal expansions of the substrate and the coating.

EXPERIMENTAL METHODICS

The aim of the experimental work was to study substrate pre-treatment under plasma sprayed coating different blasting materials, as well as the structural and adhesion changes of plasma-sprayed ceramic coatings on the Al_2O_3 and $ZrSiO_4$ basis resulting from cyclic thermal stressing. The obtain results enable to recommend particular material for a given type of stress.

Pre-treatment of surface substrate:

In order to optimally the pre-treatment of the surface beneath the ceramic coatings, we used the following blasting means:

- steel grit, $d_z = 0,9mm$,
- corundum grit, $d_z = 0,9mm$,
- demetallized steel-plant slag, $d_z = 1,2mm$,
- round steel shot, $d_z = 0,7mm$.

The blasting was realised on the laboratory blasting equipment with blasting wheels of the Di-2 type with grain velocity $80m.s^{-1}$. The blasting was realised with the required quantity of the blasting mean necessary for ideal deck of surface after impact of blasting means.

Selection of parameters related to the quality evaluation of blasted surface considered the need for obtaining the new surface with the character of sharp peaks and valleys. The mentioned character of the surface is necessary for mechanical anchorage of plasma-sprayed coatings in general. For purpose to characterize the surface in the vertical direction (in general, it is customary to use the arithmetic-mean deviation of the assessed profile Ra and the maximum height of the profile Rz), it is necessary to use the parameters classifying the surface also in terms of the broken surface and its character. The difference between the surface blasted with shot (rounded blasting material) and the surface blasted with grit (angular blasting material) makes it possible to quantify only its correlative aspect. In this evaluation, a very im-

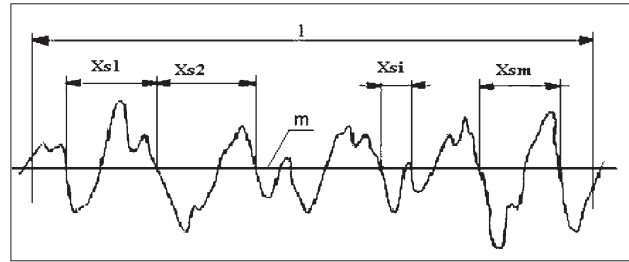


Figure 1. Mean width of the profile elements

portant role is played by the material ratio of the profile $Rmr(c)$, the relative material ratio Rmr , and the material-ratio curve of the profile (Abbot-Firestone curve).

The blasted surface (very important from the viewpoint of fixing of the coatings on the blasted surface of the base material) can be identified only by using the parameters describing the surface in terms of the frequency of peaks in the profile and its pitch, i.e., the mean width of elements of the profile RSm determined from the following correlation:

$$RSm = \frac{1}{m} \sum_{i=1}^m XSi, \quad (1)$$

Figure 1 reflects the importance of the indicated parameters (l – sampling length).

Properties experiments of ceramic coatings:

Experiments were performed with Al_2O_3 and $ZrSiO_4$ coating materials in powder form, applied with plasma-spraying equipment with water stabilization, made by Aquacentrum, with torch PAL 160, on the S355J0 substrate. Samples had the form of stepped disks $\varnothing 30/\varnothing 50$. Coating was applied on both faces of the sample. To obtain the required adhesion of the coating to the substrate, the substrate surface was pre-treated by blasting, using the so far obtained know-how on the mechanical pre-treatment by blasting.

Adhesion.

To obtain exact values of adhesion we used a mechanical tear-off destructive method, in which the adhesion is determined as a stress perpendicular to the surface of basic material, needed to tear the coating off the substrate.

Structural stability.

We investigated structural stability of booth coating, i.e. Al_2O_3 and $ZrSiO_4$. These coatings were subjected to cyclic thermal stressing with following parameters:

- heating to $800^\circ C$ / holding time 20 min. /cooling into water, cooling rate $25 - 30^\circ C.min^{-1}$.

Qualitative X – ray analyses with a Mikrometa 2 apparatus, using CuK_α radiation with $\lambda = 1,5404\text{\AA}$, was performed on following samples:

- Al_2O_3 and $ZrSiO_4$ powder before spraying,
- coatings in as-sprayed state,
- coatings after 1., 2., 3., 4. thermal cycle.

RESULTS AND DISCUSSION

In Figure 2, we present the profiles of the surfaces blasted with various blasting materials, steel grit, corun-

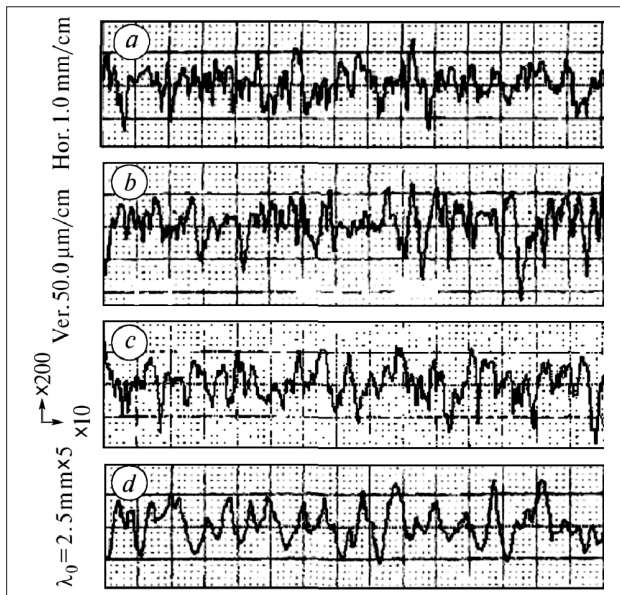


Figure 2. Profiles of the surfaces blasted with various blasting materials: (a) steel grit, (b) corundum grit, (c) demetalized steel-plant slag, (d) steel shot.

dum grit, demetalized steel-plant slag and steel shot. Their granularity was selected to obtain surfaces with the corresponding values of R_a (of about $10,5-12 \mu\text{m}$). The value of R_z varied within the range $65-80 \mu\text{m}$. In visual records, different characters of the obtained profiles are well visible. Actually, these profiles contain the following numbers of peaks:

- the surface blasted with steel grit has 33 peaks per 10 mm of the surface;
- the surface blasted with corundum grit has 28 peaks per 10 mm of the surface;
- the surface blasted with demetalized steel-plant slag has 26 peaks per 10 mm of the surface;
- the surface blasted with steel shot has 19 peaks per 10 mm of the surface.

By analyzing the surfaces subjected to blasting treatment, we conclude that the mean width of elements of the profile RSm for the surfaces blasted with corundum grid is:

- about 12% higher than for the surfaces blasted with steel grit,
- about 10% lower than for the surfaces blasted with slag, and
- about 35% lower than for the surfaces blasted with steel shot.

Parallel with the indicated facts, different characters of the individual surface topography are well visible in the visual survey of the records.

The investigation of the influence of the cyclic thermal stressing on the adhesion of coatings, Figure 3, has shown a steep decrease in adhesion for the ZrSiO_4 coating, even when its initial value was 20% higher than that for Al_2O_3 .

Figure 4 represents corundum grain fragment embedded in the boundary between the steel substrate and ceramic coating the boundary between the steel substrate and ceramic coating.

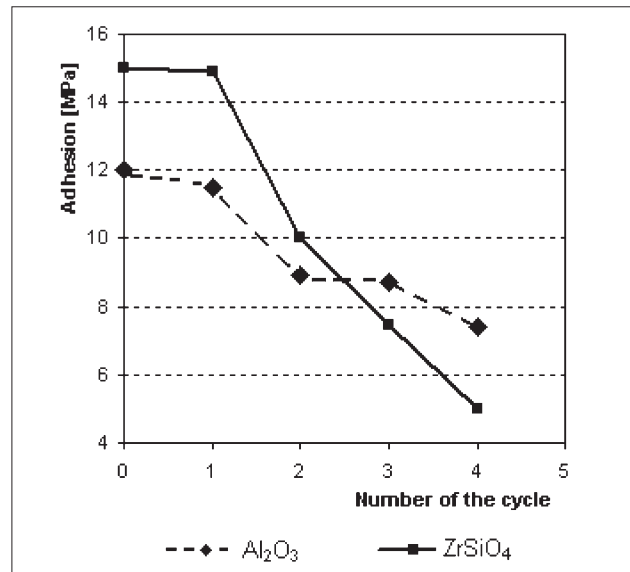


Figure 3. Change in adhesion in dependence on the number of thermal cycles

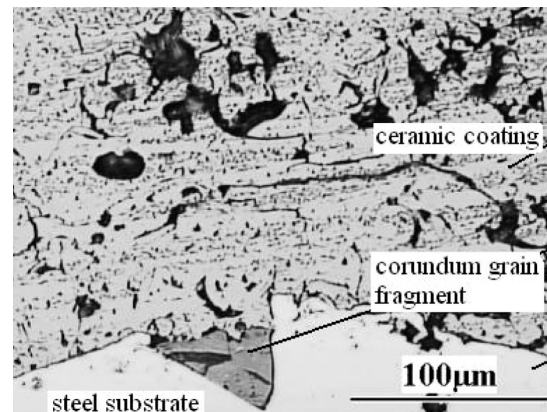


Figure 4. Picture of corundum grain fragment embedded in the boundary between the steel substrate and ceramic coating

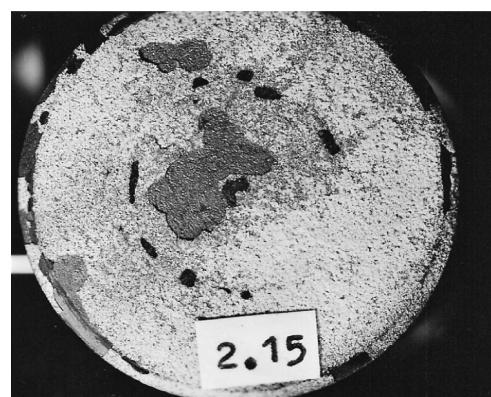


Figure 5. ZrSiO_4 coating after 7 thermal cycles

The reasons of the adhesion decrease after the samples were subjected to the thermal cyclic stressing may be seen in following factors:

- a) Porosity of 15 to 25%, with ZrSiO_4 showing up to twice as high porosity as Al_2O_3 . High porosity of ZrSiO_4 caused rapid corrosion of the basic material, as is evidences in Figure 5 taken after 7 thermal cycles.

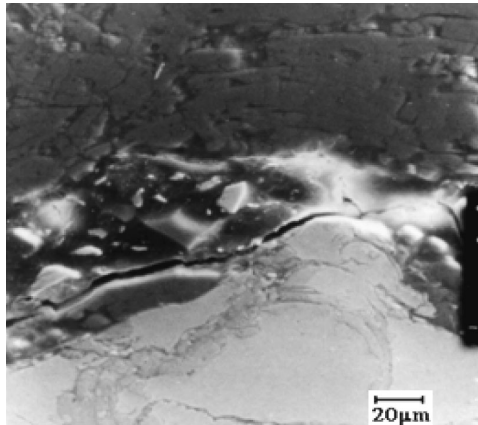


Figure 6. Microcrack on the Al_2O_3 coating – NiCr interlayered boundary found after live thermal cycles

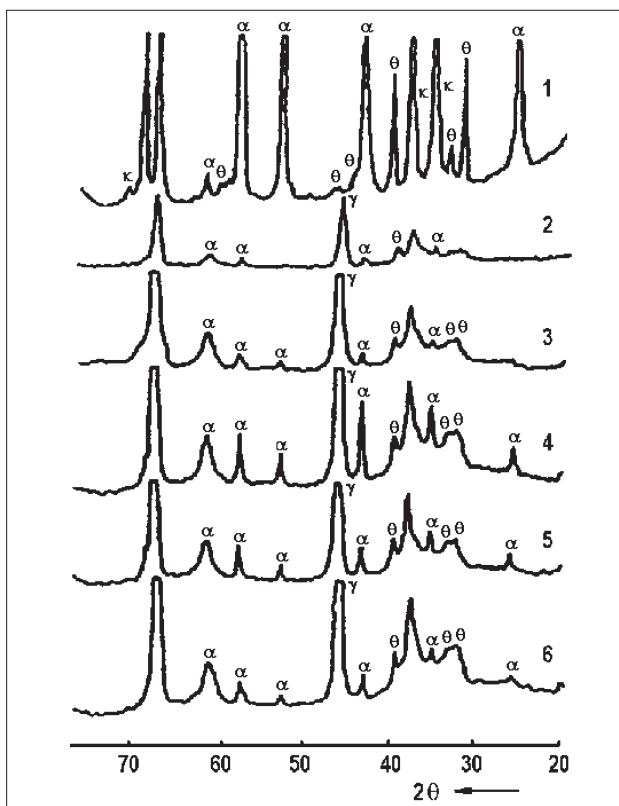


Figure 7. X-ray pictures of Al_2O_3 : 1 - Al_2O_3 powder, 2 - Al_2O_3 coating in as-sprayed state, 3-6 - coating after 1 to 4 thermal cycles; α , θ , κ , γ - phases of Al_2O_3

- b) Different values of the thermal expansion coefficients for substrate and coating (steel $\alpha = 11,7 \cdot 10^{-6} \text{C}^{-1}$, Al_2O_3 $\alpha = 7,6 \cdot 10^{-6} \text{C}^{-1}$, ZrSiO_4 $\alpha = 3,5 \cdot 10^{-6} \text{C}^{-1}$, what is most apparent for zirconium coating on steel. This difference was manifested by the formation of a crack on the substrate-interlayer after cyclic thermal stressing (Figure 6).

It was found during investigation of structural changes that in the ZrSiO_4 coating deposited by plasma spraying no structural changes take place during thermal cycling. However, these changes are apparent in the Al_2O_3 coating, whose structure became stable after two-three cycles (Figure 7).

Considering the changes taking place in the Al_2O_3 and ZrSiO_4 coatings during their thermal stressing, the stable zirconium coating shows better properties. This is valid for thermal cycles up to 800°C .

It may be concluded that, using a suitable interlayer eliminating the corrosion of the substrate due to porosity and compensating different thermal expansion of substrate and coating, the ZrSiO_4 coating seems to be more resistant to cyclic thermal stressing up to 800°C . This conclusion is based on higher initial adhesion and stability of the zirconium coating.

CONCLUSION

Based on the previous outcomes analysis we can state, that in term of variety of blasted surface, sharp blasted means, especially steel grit material and corundum are convenient for pre-treatment of the steel substrate surface under plasma-sprayed coatings. Blasted means - corundum, which has similar material characteristics as the ceramic coating, is convenient for ceramic coatings.

In term of the use of coatings on the base of Al_2O_3 and ZrSiO_4 , in conditions of thermal cyclical loading it is necessary to use inter-layers compensating different thermal expansion of substrate and coating.

Plasma-sprayed coatings on the base of ZrSiO_4 show high porosity compare to the Al_2O_3 coatings. Regarding phase stability, ZrSiO_4 coatings with inter-layer for thermo-cyclical loadings appear as more suitable.

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