## A STUDY OF RESIDUAL STRESSES IN PLASMA SPRAYED

#### ALUMINA COATING

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## ABSTRACT

The present paper shows the results of experimental determination of residual stresses in alumina plasma sprayed coatings. The determination was carried out by the use of X-ray method. An analysis of the influences of several operational factors like: coating thickness, subcoat material, substrate material on the stresses values was also outlined.

## 1. INTRODUCTION

The plasma sprayed coatings exhibit very often significant residual stresses. These stresses are due to the following reasons:

1°) Solidification of sprayed grains on a cool substrate during first film formation. The stresses existing in just solidified grain could be expressed as:

$$\sigma_{\mathsf{T}} = \alpha_{\mathsf{1}} \left( \mathsf{T}_{\mathsf{m}} - \mathsf{T}_{\mathsf{s}} \right) \cdot \mathsf{E}_{\mathsf{1}} \tag{1}$$

with :  $\sigma_r$  tensile stresses,

ai dilatation coefficient of sprayed material,

melting point of sprayed material,

T" substrate surface temperature,

E1 Young modulus of sprayed material.

These stresses could reach for  $\rm Al_2O_3$  grain sprayed into 150°C substrate the value of 500 kG/mm². However, the sprayed particles are never cooled down to  $\rm I_s$  value. They are heated by the plasma heat flux and by the heat flux of solidified and cooled following grains ;

- 2°) The final coating thickness is reached with several passages of plasma torch over the substrate. Therefore, in the coating arise the different temperature gradients with the maximum value localized near the spraying spot. The equalization of the substrate - coating temperatures could lead to stresses generation.
- $3^{\circ}$ ) The stresses could arise in the time of the post-spraying annealing at the temperatures higher than the phase transformation point. This kind of stresses could be espressed as :

$$\sigma = \frac{E_1}{3(2.\nu_1 - 1)} \left(1 - \frac{\delta'}{\delta''}\right) \tag{2}$$

where :  $\delta'$  ,  $\delta''$  are the density of the material before and after phase transformation,

ν<sub>1</sub> is the coating material Poisson modulus.

A very typical example is the transformation of  $\gamma$ -Al<sub>2</sub>0<sub>3</sub> to  $\alpha$ -Al<sub>2</sub>0<sub>3</sub> at  $\sim$  1100°C with the range of density from 3.67 g/cm<sup>3</sup> to 3.69 g/cm<sup>3</sup>.

4°) The last and the hest known cause of stresses generation is the cooling of sprayed sample after processing. This case will be discussed in the next paragraph.

The residual stresses have special importance when spraying ceramics. These materials fail in a brittle manner and in the most cases without plastic deformation. As the ceramics are much weaker in tension than under compression it is important to avoid tension in coating. The fracture as a result of the stress is propagated in the easiest manner at the surface of a specimen /1/. That is why we will discuss the stresses near the coating surface.

The previous papers on the similar subject /2, 3, 4, 5, 6/ concerned calculation and measurements of stresses, in the most cases, in plastic material coatings. Only Hasui and Kitahara /6/ had determined the stresses by the use of mechanical method (bending of sprayed plate) in Al<sub>2</sub>0<sub>3</sub> coating. They found the value of compressive stress  $\sigma \sim 1 \text{ kG/mm}^2$  nearly independent upon the coating thickness.However they did not show the influence neither of the spraying parameters nor of the application of plastic material subcoat on the value of stresses.

There were not the papers applied the X-ray method to the determination of the stresses. This method gives the possibility of stresses determination in multiphases coating and is relatively simple.

## 2. THEORY

The thermal stresses generated when coating is the thin plate specimen (Fig. 1) could be expressed  $\ensuremath{/7/}$ :

$$\sigma(y) = \frac{E(y)}{1 - v(y)} \left[ (\alpha(y)) \left[ T(y) - T_{a} \right] - \frac{R_{e}}{R_{0}} - (\frac{R_{1}}{R_{0}} - y), \frac{1}{R} \right]$$
 (3)

with T ambiant temperature,

$$\frac{1}{R} = \frac{R_{e_1} \cdot R_0 - R_{e} \cdot R_1}{(R_1)^2 - R_0 R_2},$$

$$R_0 = \int_0^a \frac{E_*w_*dy}{1-v}$$
,  $R_1 = \int_0^a \frac{E_*w_*dy}{1-v}$ ,  $R_2 = \int_0^a \frac{E_*w_*y^2}{1-v}$ 

$$R_{e} = \int_{0}^{a} \frac{E \cdot \alpha \left[T(y) - T_{a}\right]w \cdot dy}{1 - \nu}, \quad R_{e1} = \int_{0}^{a} \frac{E \cdot \alpha \left[T(y) - T_{a}\right]w \cdot y \cdot dy}{1 - \nu}$$

The calculations of the stresses on the coating surface were made for the  ${\rm Al}_2{\rm O}_3$  coating sprayed onto NiCr (80/20) subcoat with different thicknesses. This calculations were repeated for the different temperature of the coating surface just after processing ( ${\rm I}_{\vec k}$ ). The material properties use in all the paper are listed in the table 1.

It was assumed that  ${\rm Al}_2{\rm O}_3$  is sprayed into stainless steel and temperature gradient within the coatings is equal to 50 deg/mm. This last value was taken after /8/.

The value of E/(1+v) for  $Al_20_3$  was taken after /9/ where the both coefficients were determined ultrasonically. In these investigations E is proportional to the density. The samples analysed in /9/ were very porous with the density of about 3 g/cm $^3$  but the paper /9/ is the only one, known to the authors, about the mechanical properties of sprayed alumina. It should be also underlined that the dependence of E and v upon the temperature is unknown. Here are assumed the values for the ambiant one.

The calculated stresses depend—upon the subcoat thickness very strongly up to coating thickness d % 120  $\mu m$  (Fig. 2). In this range, the increase of subcoat thickness results in decrease of stresses of 20 - 30 %. For d > 120  $\mu m$  the stresses are rather independent upon either coating or subcoat thickness.

The compressive stresses are nearly proportional to the value of temperature T $_{\rm k}$  (Fig. 3). The minimum from the proceeding figure is also visible for d\*\* 120 - 200  $\mu m$  and is shifted to higher d - values with increase the T $_{\rm k}$  values.

## 3. EXPERIMENTAL

The alumina coatings were sprayed on the substrates of copper and stainless steel having the dimensions 30 x 20 x 2 mm. The kovar ones have had the dimensions 30 x 20 x 1.2 mm. The substrates were fixed to a vacuum suction cup. The temperature of back front of the substrate have been measured using Ni-NiCr thermocouple. The used powder characteristics are shown in table 2.

The spraying process was carried out with the use fo PN $\pm$ 200 plasma torch which working parameters listed in table 3.

The substrates were grit blasted prior to spraying. The coating thickness measurements and metallographical observations were carried out using optical microscope. The stresses were determined using DRON-1,X-ray apparatus with Cu-K $\alpha$  radiation. The sketch of these investigations is showed on Fig. 4.

The values of residual stresses has been found according to following relation /10/

$$\sigma = \frac{E_1 \cdot \operatorname{ctg} \theta_1}{2(1+\nu)\sin^2 \psi} \cdot (2\theta_n - 2\theta_1) = K_1 \cdot (\Delta 2\theta)$$
 (4)

In this investigation it was applied the peak of corundum  $d_{124}$  = 1.404 Å ( $\psi$  = 30°). When the surface near stresses were determined— it was applied the peak of curundum  $d_{024}$  = 1.740 ( $\psi$  = 20°). The precision of  $\Delta 2\theta$  determination was of about 0.01°.

#### 4. RESULTS AND DISCUSSION

## 4.1. Influence of coating thickness on the stresses

Fig. 5 shows the influence of sprayed alumina coatings thickness on the values of stresses. Suprisingly, the stresses decrease when the thickness increases. But the metallographical analysis shows cracks, parpedicular and parallel to the surface with their dimension rising with the coating thickness.

The coating were sprayed with the use of the same parameters but the spraying time was different with the thickness of the spraying layer. Therefore, the temperature of the coating (see Table 3) rose from 400 to 900°C. The residual compressive stresses resulting of cooling after process were, for the thicker coatings, higher than compressive strength of ceramics what induced a cracking. The cracking caused the relaxation of stresses observed in Fig. 5.

## 4.2. Influence of application the metallic subcoats

The application of frequently used NiAl and NiCr subcoats caused the lowering of residual stresses (Fig. 6). In the case of coatings sprayed into copper substrate only the application of three-coating structure (with the mechanical mixed cermet 01 + NA (50/50)) gives the real lowering of stresses of about 30 %. For the Al<sub>2</sub>O<sub>3</sub> coating sprayed onto stainless steel substrates such lowering is obtained already by the application of single subcoat NiCr. It should be also underlined that such low values of stresses, in comparison with those discussed in previous paragraph, could be caused by application of compressive air cooling during spraying procers.

## 4.3. Influence of different substrate materials

This analysis was carried out for the  ${\rm Al}_2{\rm O}_3$  (O1) sprayed onto copper and st. steal substrates which have high dilatation coefficient (see Table 1) in comparison with kovar substrate which has this one relatively low.

It is obvious, after Table 4, that the stresses are lower for the  ${\rm Al}_2{\rm O}_3$  coatings sprayed into material with the low dilatation coefficient.

# 4.4. The comparison of stresses observed with the use of different depth of X-ray penetration

The penetration depth of  $\chi\text{-ray}$  is proportional to the sine of angle 0 according to expression /10/ :

$$x = \frac{K_{X} \cdot \sin \theta}{2 \mu} \tag{5}$$

where: µ - linear absorption coefficient (1/cm),

 $K_x$  - constant,  $K_x$  = 4.6 when assuming that 99 % of  $\chi$ -ray is diffracted from the depth less or equal to x.

The calculation of x-values for sprayed alumina are shown in Table 5. These are only roughly estimated because the  ${\rm Al}_2{\rm O}_3$  materials coefficients were taken as for sintered alumina.

The stresses observed near the coating surface are one order of magnitude higher than those averaged from the greater depth. It is important to discuss the absolute values of stresses. In paper /6/ the value of compressive stresses within  $\mathrm{Al}_2\mathrm{O}_3$  coating plasma sprayed into mild steel substrates was about 1 kG/mm $^2$ . In this paper these values are even higher than 100 kG/mm $^2$ . The authors of paper /6/ used as a substrates the mild steel band of 130 mm length and of 1.5 mm thickness. The bending of this band after spraying was the base for stress calculation. But using so long substrates the stresses could be easier relaxed. The relaxation ability is, roughly estimating, proportional to the ratio of substrate length to thickness. This ratio was in the paper /6/ of about 87 in this paper - 15. The second source of divergence could be the temperature  $I_{\rm p}$ , which has a strong influence on the stresses values (see Fig. 3). At last, from authors opinion, the basical origin of divergence are the unknown values of materials coefficients i.e.  $\boldsymbol{E}$  and  $\boldsymbol{\nu}$ . It seems to be that it is necessary to carry out the stresses determination together with the investigation of E and v coefficients.

#### 5. CONCLUSIONS

The application of x-ray method to the residual stresses determination leads to demonstration that the use of metallic subcoats causes the lowering of the stresses. The same effect gives the application of substrate material with nearly the same thermal dilatation coefficient. In the paper, the analysed stresses, are the compressive ones and have their origin in sprayed sample cooling after the process. It is clear that the additional attention is need to the inves-

tigations of stresses caused by phase transformation. The accurate knowledge of the absolute values of stresses needs the determination of mechanical moduluses of Young and Poisson of the sprayed coatings and also the determination of temperature gradient within coating-during and after process.

#### ACKNOWLEDGEMENTS

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#### FIGURES CAPTIONS

- Fig. 1 The geometry of analysed thin plate.
- Fig. 2 Compressive stresses on the  ${\rm Al}_2{\rm O}_3$  coating surface depending upon thickness of coating (a) and subcoat (b). Theoretical calculations.
- Fig. 3 Compressive stresses on the surface of  $\Lambda l_2 \theta_3$  coating depending upon coating thickness (d) and substrate temperature just after spraying process  $(T_k)$ . Theoretical calculations.
- Fig. 4 The sketch of  $\chi$ -ray stresses determination 1 direction of  $\chi$ -ray radiation.
- Fig. 5 The compressive residual stresses within  $\Lambda l_2 0_3$  (OD) layer sprayed onto copper substrate. The dependence upon the coating thickness.
- Fig. 6 The compressive residual stresses in  $\Lambda l_2 \theta_3$  (01) coatings sprayed onto different substrates and subcoats.

TABLE 1

## PROPERTIES OF ANALISED MATERIALS

Properties	A1203	N1Cr(80/20)	Stainless steel	kovar	соррег
$\frac{E}{1+v} - 10^{-3}$ (kG/mm <sup>2</sup> )	7.4	16	14		- 10
a . 10 <sup>6</sup> 20 - 700°C (1/dag)	7.3	16	20	8	20

## TABLE 2

## THE APPLIED POWDERS

hotations	Powders	X-ray phase	grets size(um)	Manufacturar	Permarks
01	A1203+2 1 T102	corundum	- 70 + 10	Meton (USA)	type 101 SF
อก	A1263 39 %	corundum	- 4u	Inst. Nucl. Res. (Poland)	spherical
NC	nicr (89/20)	-	- 50	Tacha. Univ. Wroclaw (Poland)	spherical
NA.	N1A1 (70/3J)		- 120 + 40	Techn. Univ. Wrncley (Polend)	composite

TABLE 3
PLASMA SPRAY PARADETERS

Powdar	AIS	nc.	00	01	NA + 0105-2/50
Plasma gases i Primary i Ar, (Gdm³/s) Secondary i H <sub>2</sub> (Gklm³/s)	1.2 0.12	1.2 0.12	1.2 0.40	1.2 0.12	1.2 U.12
Feeding gases : Ar, (Ndm <sup>3</sup> /s)	0,03	0.83	0.83	0.83	0,83
Spraying distance (cm)	10	10	10	10	10
Promit (FW)	32	12	49	32	32
Sliding substr. volocity (cm/s)	U.40	0.40	G.40	0,40	3,46
Substrate back front temperatures : Prior to apraying (°C) After spraying (°C)	150 350	150 350	150 400 - 900	150 450	150 400
Cooling by compressed air ?	yes	yes	no	yes	yas

TABLE 4

COMPRESSIVE RESIDUAL STRESSES WITHIN A1203 (01) COATING

SPRAYED ONTO DIFFERENT SUBSTRATES

TABLE 5

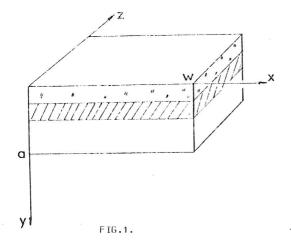
THE RESIDUAL SIRESSES IN THE Algo, COATINGS WITH THE USE OF DIFFERENT X-HAY FEMERATION DEPTH (K  $_{\chi}$  = 4.81

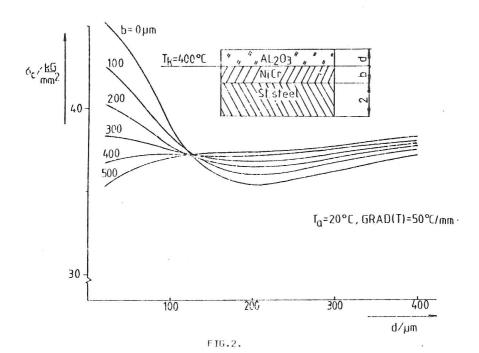
Angle 0	corundum hal	y-ray constration depth	Regidual compressive stresses (kG/mm <sup>2</sup> )		
	COLOUR HXC	(um)	St. Steni/NC/ 01"	Cu/NA/01 **	
33,50	124	100	31	35	
25.25	G24	110	110	120	

<sup>\*</sup> coating thicknesses, NC \* 30 um, 01 \* 35 um

<sup>\*\*</sup> coating thicknesses, NA = 20  $\mu m_{\star}$  01 = 20  $\mu m_{\star}$ 

<sup>\*\*\*</sup> values calculated for sintered elumine





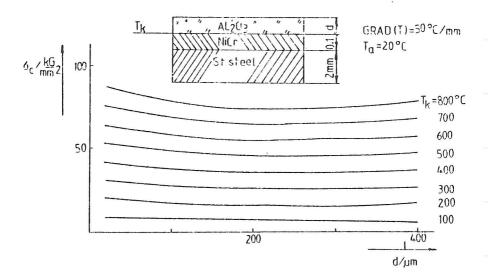


FIG.3.

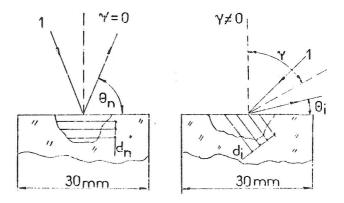


FIG.4.

