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#### ON THE MICROSTRUCTURE OF PLASMA-SPRAYED CHROMIUM OXIDE

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<u>Résumé</u> - On a étudié l'effet des variations des paramètres du procédé sur la microstructure des revêtements de  $Cr_2O_3$ . Ces variations, ainsi que les paramètres des poudres affectent curieusement très peu la microstructure. On postule que la décomposition de  $Cr_2O_3$  qui semble inévitable eu égard aux hautes températures du procédé, est le facteur qui contrôle l'évolution de la microstructure.

<u>Abstract</u> - The effect of variations in process parameters on the microstructure of chromium oxide coatings was studied. Changes in process parameters and powder parameters affect the microstructure to a surprisingly small extent. It is postulated that decomposition of  $\operatorname{Cr}_20_3$  - which seems inevitable due to the high temperature process - is the factor that controls the microstructure development.

#### I - INTRODUCTION

Erosion is causing society enormous costs due to material losses. To reduce this spoilage, materials are often covered with erosion resistant layers. These may be applied by using techniques such as chemical vapour deposition and plasma or flame spraying. A complete replacement of metallic materials by polymers or ceramics may be preferable in some situations.

Plasma-spraying is being used for application of several oxide materials on metals. Examples are alumina, titania, zirconia and chromia. The latter oxide is very interesting due to its relatively high hardness and high melting temperature. An important application for plasma-sprayed chromium oxide coatings is in pump components for improved surface fittings.

In order to optimize the process conditions for plasma spraying of chromium oxide layers on stainless steel, a study was undertaken to investigate the relationship between the process parameters selected, the microstructure developed and erosion properties achieved /1/. Surprisingly, very small effects of the process parameters variation on the resulting microstructure were found. Thus considerable changes in spray distance, electrode condition, plasma gas and power as well as powder quality and layer thickness influenced the microstructure to an extremely small extent. Chromium oxide layers from different process conditions are very similar and characterised by populations of large and small cracks and some limited porosity. This paper will discuss in detail the development of this characteristic microstructure of plasma-sprayed chromium oxide.

<sup>(1)</sup> Now with ASEA CERAMA AB, S-915 00 Robertsfors

### II - EXPERIMENTAL

The basic material in this study was chromium oxide powders from METCO Corporation. Major additives in the powders 136 F and 136 CP are silicon dioxide and titanium dioxide. For comparison powder 106 with no additives was also used. Characteristics of these original powders are shown in table 1. In addition to these powders, grain-size classified powders, > 38 µm or < 38 µm were also plasma-sprayed.

Powder	Particle size distribution (w/o)					Additives (w/o)	
	> 90 Jum	mى 63 <	> 45 µm	mىر 38 <	> 20 µm	SiO <sub>2</sub>	TiO <sub>2</sub>
136 F	< 1	. 3	16	40	85	4.4	2.4
136 CP	< 2	28	60	70	94	5.0	2.8
106	< 2	37	49	60	n d	0.2	-

Table 1: Selected properties of chromium oxide powders

The plasma spraying was performed using an equipment METCO 7 MB. The process parameter changes used are summarized in table 2. Pretreatment of the steel substrate included degreasing and grit blasting with alumina.

Process Parameter	Variation	
Spray distance	50 - 100 mm	
Electrode condition	New and old electrodes	
Plasma gas	$N_2/H_2$ and $Ar/H_2$	
Power	38 kW and 61 kW	
Powder quality	Different particle size distributions and additives	
Thickness of layer	mu, 200 – 600 سر, m	

Table 2: Process parameter variations in this study

The evaluation of microstructure and phase development during plasma-spraying was performed using SEM-EDS and X-ray diffractometry.

#### III - RESULTS and DISCUSSION

The main finding in this work is the remarkable independence of process parameters on the resulting microstructure. Plasma sprayed chromium oxide layers produced under different process conditions have all very similar microstructures, characterised by populations of large and small cracks in a matrix of varying grey level. Typical microstructural features are presented in figures 1 and 2. The resultant characteristic microstructure is discussed in terms of electron density variations and phases obtained.

### Electron Density

The crack pattern developed in the chromium oxide layer isstrongly related to the grey level of the matrix, i.e. to the electron density. This is clearly seen in the

backscattered electron image micrograph presented in figure 2 above. Variation in electron density is normally explained in terms of different chemical composition. In plasma-sprayed chromium oxide coatings the presence of discrete impurities (mainly Zr) explains to some extent this appearance. However detailed elemental line analysis does not confirm this to be the main reason. Variations in electron density may be caused by the additives, since these are unevenly distributed in the original powder batch. This is especially pronounced for SiO<sub>2</sub>. See figure 3.

However, the same crack pattern and the similar difference in grey level were later observed in the study when chromium oxide quality 106 with no extra additives was plasma sprayed. This means that the main contribution to this grey level variation has to be found elsewhere.

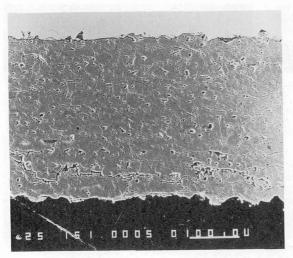
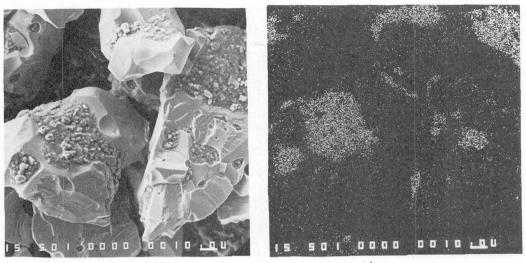


Fig 1. Dverview of the microstructure of plasma sprayed chromium oxide layer. (bar = 100 µm)



Fig 2. BEI-micrograph showing variation in grey level related to the crack pattern. (bar = 10 µm)



a)

b)

Fig 3. Distribution of SiO<sub>2</sub> in the original chromium oxide powder 136 CP a) SEM-micrograph<sup>2</sup> b) X-ray mapping of silicon

## Phases obtained

The totally dominant phase in all the plasma sprayed layers is  $\alpha$ -Cr<sub>2</sub>O<sub>3</sub>. All the peaks in the diffractograms correspond to this phase. X-ray diffractograms taken from different depths in the plasmasprayed layers were identical. No solid solutions of Cr<sub>2</sub>O<sub>3</sub> exist. However detailed evaluation reveals that the intensity of one peak (d = 2.04 Å) is too high. Moreover the peak at 1.18 Å is somewhat distorted. These two peaks correspond also to the main peaks of elemental chromium, hkl-planes (110) and (211). In the case of powder 106, the presence of chromium (II) oxide was also detected. See figure 4. Decomposition of Cr<sub>2</sub>O<sub>3</sub> is discussed by Boch et al /2/ who in addition to  $\alpha$ -Cr<sub>2</sub>O<sub>3</sub> propose Cr<sub>3</sub>O<sub>4</sub>.

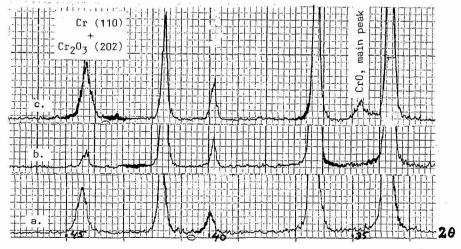


Fig 4. Parts of X-ray diffractograms indicating presence of elemental chromium as well as chromium (II) and chromium (III) oxides. (Cu-K<sub>st</sub> radiation)
a) Plasma-sprayed 136 F
b)∞-Cr<sub>2</sub>0<sub>3</sub>
c) Plasma-sprayed 106

#### IV - CONCLUSION

It has been shown that the grey level variation in plasma-sprayed chromium oxide coatings, easily seen in BEI-micrographs, arises from different oxidation states of chromium. In addition to the main phase  $\propto$ -Cr<sub>2</sub>O<sub>3</sub> elemental chromium and chromium (II) oxide are also present. The mixture of different phases will probably enhance crack formation during cooling of the plasma-sprayed layer. The electron density variation strongly related to the crack pattern supports this. It is thus postulated that decomposition of  $\propto$ -Cr<sub>2</sub>O<sub>3</sub> - which seems inevitable due to the high temperature process - is the factor that controls the microstructure developed. Decomposition of SiO<sub>2</sub> was also observed during the plasma spraying. The SiO<sub>2</sub>- contents in the coatings were approximately 2.0 w/o compared to 4-5 w/o in the original powder.

#### ACKNOWLEDGEMENT

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