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RESIDUAL STRESS MEASUREMENTS OF A PLASMA SPRAYED  
COATING SYSTEM USING SYN. (U) NORTHWESTERN UNIV  
EVANSTON IL DEPT OF MATERIALS SCIENCE  
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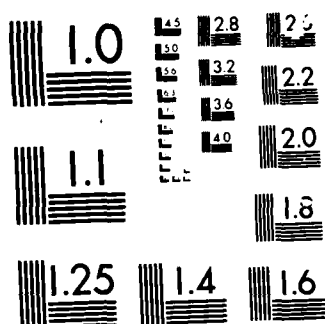
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# NORTHWESTERN UNIVERSITY

## DEPARTMENT OF MATERIALS SCIENCE

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### RESIDUAL STRESS MEASUREMENTS OF A PLASMA SPRAYED COATING SYSTEM USING SYNCHROTRON X-RAY RADIATION

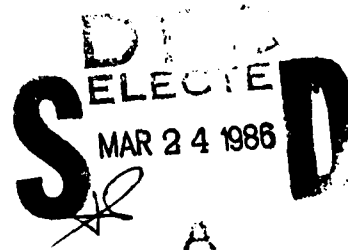
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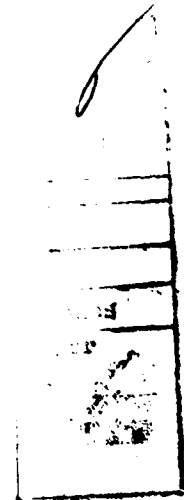
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Plasma spraying is used to form a protective coating on a substrate; for example, an oxide such as  $\text{Al}_2\text{O}_3$  on steel. This highly industrialized coating deposition method involves the production of a high velocity thermal plasma flame into which is inserted a carefully metered flow of powder of the material-to-be-deposited. The powder particles, of the order of 40 micrometers in diameter, melt rapidly and are accelerated at the order of sonic velocity to impinge on a substrate where solidification occurs at rates similar to that for rapid solidification processing (e.g.,  $10^6$  C/sec) [1].

Ceramic oxides are commonly plasma sprayed onto metallic substrates to form coatings for electrical insulation, for wear resistance or for thermal protection. Due to the great difference in modulus and thermal expansion coefficient between the ceramic and the substrate, it is expected that residual stresses form which can have deleterious effects on coating system performance. In fact, during plasma spraying, the substrate normally reaches elevated temperatures due to both preheating cycles and, at the very substrate surface, through the release of heat from the solidifying particles. Following the coating process, when the flame is removed, the temperature of the coating-substrate system drops rapidly. Since refractory oxides have significantly lower thermal expansivities than do metals (e.g.,  $14 \times 10^{-6} \text{K}^{-1}$  vs.  $8 \times 10^{-6} \text{K}^{-1}$  for steel and  $\text{Al}_2\text{O}_3$ , respectively), a ceramic coating will undergo less shrinkage than will a metal substrate. It is thus anticipated that a tensile residual stress will be established at the substrate surface and a compressive residual stress will be established in the coating at the interfacial region adjacent to the substrate. In the extreme, these stresses can be of such a magnitude that interfacial failure can arise leading to coating delamination.

Few studies have been carried out on the residual stresses associated with plasma sprayed coatings. There have been examinations of the residual-

stress-induced curvature of thin metal substrates which have been plasma sprayed, but these studies give average macroscopic strains and do not unambiguously lead to an understanding of the stress distribution within the coating system [2].

X-ray stress measurement techniques, while very well established for determining stresses at the surfaces of bulk specimens are limited in intensity to penetrate the coating and evaluate the stress state associated with the outer surface of the substrate. For the samples used in this study, the commonly employed 211 peaks in the substrate could not be detected with a normal diffractometer with  $\text{CrK}\alpha$ . Synchrotron radiation offers substantially increased intensity and resolution and thus enhanced measurement accuracy. We here report the first residual stress measurements at the substrate surface, beneath a plasma sprayed  $\text{Al}_2\text{O}_3$  coating.

#### Experimental

The x-ray diffraction measurements were carried out at the National Synchrotron Light Source, Brookhaven National Laboratory, beam line X-18A. A fixed exit, sagittally focused Si (111) monochromator was employed, producing a  $1.5 \times 2 \text{ mm}^2$  size beam with an on-sample flux of approximately  $2 \times 10^{10}$  photons/s at 100 mA stored electron beam. The diffraction apparatus consists of a Huber four-circle diffractometer mounted on a leveling table and equipped with ion chamber and scintillation counter incident beam monitors and a solid state detector with associated electronics. The diffractometer assembly, as well as the monochromator, are interfaced to a DEC PDP11 minicomputer.

For these experiments, a wavelength of  $1.1163(2) \text{ \AA}$  was chosen, yielding the 422 reflection at  $145^\circ 2\theta$ , near the upper limit of the diffractometer motion. This reflection was chosen for uniformity with traditional laboratory stress measurements on steels, which usually involve the 211 reflection and  $\text{CrK}\alpha$ . The specimens were mounted on the diffractometer axis with a positional

accuracy of  $\pm 0.1$  mm (the diffractometer radius was 46 cm). Scattering angles were accurate to within 0.02 degrees, whereas the psi settings were accurate to 0.05 degrees.

Measurements were made on three steel specimens: 25 X 50 X 3 mm. One was on a grit blasted surface (standard treatment in preparation for plasma spraying), one was after plasma spraying with 96 micrometer of  $Al_2O_3$  and the third was the grit blasted back side of the sprayed specimen, in order to evaluate the effects of heating during the spraying operation. The plasma spraying was carried out using a Plasma-Technics system with robotic gun manipulation and the parameters listed in Table 1. For each specimen, the 422 reflection was recorded at  $2\theta$  intervals of 0.05 degrees over a range of 1 degree for six psi tilts between 0 and 45 degrees (tilts away from the normal focussing position around an axis in the specimen surface). The peak profiles were least squares fitted with Gaussian or Lorentzian curves. In each case, four parameters were estimated: Peak position, peak width, peak intensity and background. Even though peak-to-background ratios were different for Gaussian and Lorentzian fits, the peak positions agreed to within 0.001 degrees. The error estimate in absolute peak positions was better than 0.01 degrees for all cases.

Fig. 1 shows the results of our experiments as plots of d spacing vs.  $\sin^2\psi$ . From the slopes of these lines, the stresses calculated in the Reuss limit [3] are given in Table 2.

Table 1  
Plasma Spray Parameters

|                | Gases                     | Pressure | Flow Rate   |
|----------------|---------------------------|----------|-------------|
| Amperage 500   | Ar/Primary                | 1.8 Bar  | 52.5 l/Min. |
| Voltage 72 ± 4 | H <sub>2</sub> /Secondary | 1.5 Bar  | 12.5 l/Min. |

The powder carrier gas was Ar at 1.5 Bar and a flow rate of 5.5 l/Min.

The powder flow rate was 55 gm/Min. The spray distance was 100 mm.

Table 2  
Stress Estimates for Various Surface Conditions

| Surface      | Stress (MPa) | Standard Deviation (MPa) |
|--------------|--------------|--------------------------|
| Grit blasted | -181         | 12                       |
| Sprayed      | -79          | 11                       |
| Back side    | -94          | 6                        |

Compressive residual stresses are noted for the grit blasted steel surface ( $-181 \text{ MPa} \pm 12 \text{ MPa}$ ). When the steel specimen was plasma sprayed, sufficient annealing occurred, resulting in a significant reduction in the residual stress on the unsprayed side of the substrate specimen. Of course, the annealing was the result of heating from the plasma flame, and the backside was likely at a lower temperature than the coated side. Nonetheless, the residual stress of the uncoated side decreased by about 77 MPa to a value of  $-94 \text{ MPa} \pm 6 \text{ MPa}$ .

The steel surface under the plasma sprayed  $\text{Al}_2\text{O}_3$  coating registered a stress of  $-79 \text{ MPa} \pm 11 \text{ MPa}$ , which amounts to a decrease of 102 MPa; an approximately 25 MPa greater drop in compressive residual stress than that experienced for the back, uncoated side. A simple explanation for this difference in residual stress between the coated and uncoated backside is that the coating introduced to the former a tensile surface stress of about 25 MPa.

It is shown here that synchrotron x-ray radiation is sufficiently penetrating through a 96 micrometer thick  $\text{Al}_2\text{O}_3$  coating to enable a relatively accurate assessment of the residual stresses associated with a steel substrate. This technique can lend itself to wide ranging applications for the evaluation of stress states associated with thick films and coatings.

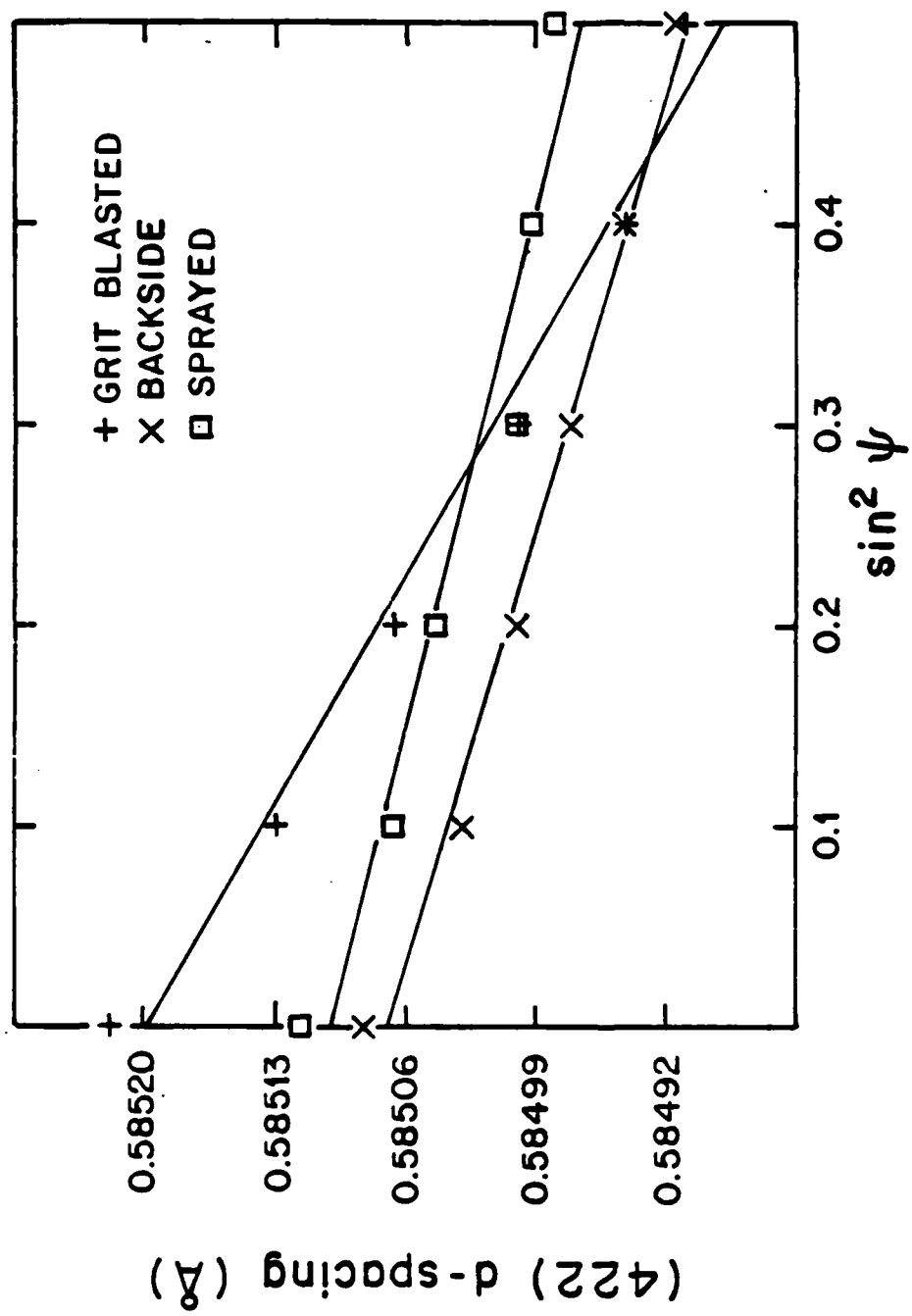
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### References

- [1] S. Safai and H. Herman, In H. Herman (ed.), Ultrarapid Quenching of Liquid Alloys, Vol. 20 of Treatise on Matls. Sci. & Tech., Academic Press, New York, 1981, p. 183.
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Figure 1. (422) d-spacing ( $\text{\AA}$ ) plotted against  $\sin^2 Q$  for three different surfaces.



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| 13. ABSTRACT<br>Residual stresses have been measured for the first time in the substrate below a plasma coating of ceramic. The effect of the heating by the coating during application was detected. <i>experiment</i> |  |                                                                                   |                 |

| 14. KEY WORDS                                    | LINK A |    | LINK B |    | LINK C |    |
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