DEPC-MN 025

THE DETERMINATION OF RESIDUAL STRESSES IN COATINGS BY COUPON BENDING

The use of the coupon bending method for the measurement of residual stresses in coatings was explored. The Stoney formula was found to be inadequate for thick thermally sprayed WC/Co, but an extension of this analysis by Freund was found to give consistent results. Both a measurement microscope and a laser triangulation system were used to make measurements, but the laser triangulation system could only be used on samples with a smooth surface. By using the furnace incorporated into the laser triangulation system to heat a FeCrAlY coated sample an estimate of the thermal expansion coefficient of the coating was achieved.

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Introduction

Residual stresses are often generated in coatings during processing because of thermal stresses generated due to the mismatch in thermal expansion coefficients that often exists between the coating and the substrate.

Knowledge and control of residual stress in the coating is important to the coating processor and the user of coated parts as it can affect the adhesion of the coating, and can cause unwanted deformation of the parts.

The bending of a coupon from induced thermal stresses processed at the same time as the components being coated offers a simple way of assessing the magnitude of the residual stresses that are generated [1,2]. The curvature of the coupon is measured by a variety of possible techniques including optical microscopy and laser interferometry, and the residual stress in the coating is calculated from this curvature. This Measurement Note reports on some experiments that were undertaken to evaluate the applicability of the coupon bending technique to coatings.

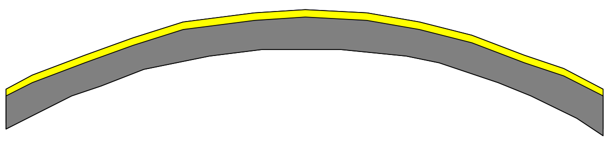

Figure 1 Schematic diagram showing a coated sample (coating is yellow) with thermal mismatch compressive stress in the coating

Experimental Details

Materials

A WC/Co coating 225 µm thick was thermally sprayed onto EN3B steel substrates using a JP5000 HVOF thermal spraying system. The samples were 150 mm in length with a width of 10 mm. The central 60 mm of the samples was coated. Three thicknesses of substrate were used at 1.2, 2 and 3 mm thick. The substrates were grit blasted either by a coarse or a fine abrasive before coating to increase the strength of adhesion of the coating.

As part of an interlaboratory exercise within CEN TC 184 WG5 Ceramic Coatings a 0.5 mm thick sample of tool steel that had been coated with a 2 µm FeCrAlY coating was also examined.

Knowledge of the elastic properties of the materials is necessary for the calculation of residual stress. The Youngs modulus of the coarse grit blasted WC/Co coating was found to be 185 GPa and for the fine grit blasted WC/Co coating 220 GPa by beam bending and impact excitation experiments [3]. The Poisson's ratio of the coating was taken to be 0.25. A Young's modulus of 210GPa was assumed for the EN3B steel substrate.

Test Equipment

Most measurements were made with a Nikon measurement microscope. This microscope was fitted with displacement transducers that gave a measurement accuracy in the X and Y lateral directions of 2 µm and a similar accuracy in the Z height direction when a suitably high magnification objective lens was used. Although this instrument was used, in principle any reasonably high magnification optical microscope can be used once the movement in the Z or focus direction has been calibrated. Usually the focus control of these microscopes is marked so that the Z movement can be recorded.

A Flexus 2960 residual stress measurement system was also used for the FeCrAlY coated sample. This instrument uses laser triangulation to determine the height of the sample in a profile made along the direction of curvature, and is fitted with a furnace that enables the user to explore the variation of residual stress with temperature. This allows estimates of the thermal expansion mismatch between the coating and the substrate to be made. In the event, although measurements were attempted on the thermally sprayed WC/Co samples, the results that were obtained were not meaningful and did not show the curved profile that was clearly evident on the samples. This was felt to be due to the poor finish on these as-coated samples.

Results

Figure 2 shows the results of height measurements made along the coated area of the WC/Co coated samples. It can be seen that substantial bending of the test coupons had occurred (with a convex surface on the coating side of the sample). This bending is an immediate indication that compressive residual stresses have been generated in the samples. The bending shows a progressive increase as the thickness of the substrate is decreased, and the bending of the fine grit blasted samples is smaller than the bending for the coarse grit blasted samples.

A routine was written in Labview™ to calculate the curvature of the samples, and to calculate the residual stress in the coating using the Stoney formula (see analysis section later). This routine calculated the slope of the data points, and then plotted these slopes against the distance along the sample (Figure 3). A regression analysis gave the gradient m that is related to the sample curvature R as $R=1/m$.

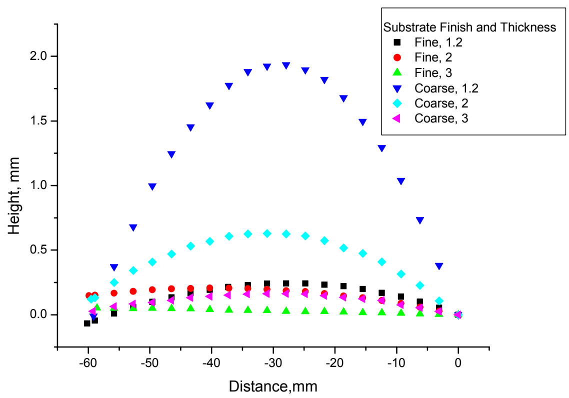

Figure 2 Results of measurements on thermally sprayed WC/Co samples. The figure in the legend denote substrate thickness.

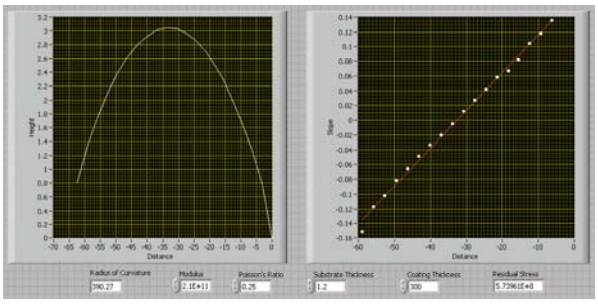

Figure 3 Front panel of Labview™ routine to evaluate curvature and residual stress in coatings. Left hand panel shows fit though data, right hand panel shows regression fit of slopes to derive curvature.

The preparation of the samples for coating might also have introduced thermal stresses into the sample. This was checked by measuring the profiles of some uncoated substrates (Figure 4) that had been grit blasted. It was found that there was bending characteristic of induced thermal stress, but that the size of the bending was very small.

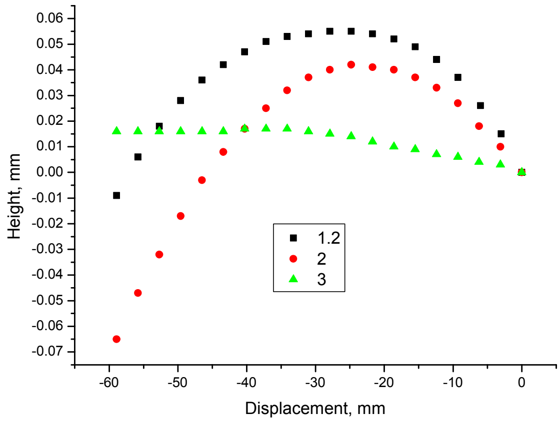

Figure 4 Profiles of coarse grit blasted substrates. Numbers in legend refer to thickness of substrate.

The radius of curvature results are plotted in Figure 5. The different points for the same conditions are measurements on different samples. The radius of curvature for the coarse grit blast samples are larger than those for the fine grit blast samples. There is also a large scatter in the results.

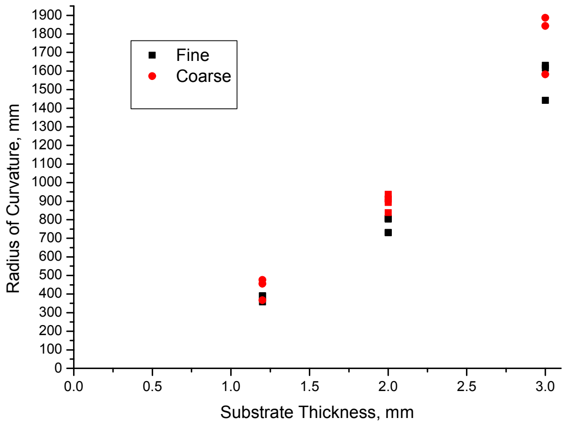

Figure 5 Radius of curvature results.

The bending of the FeCrAlY sample was measured both by the measurement microscope and by the Flexus instrument (Figure 6). It can be seen that the agreement between the two instruments is very good. However, because the Flexus is an automatic instrument a much higher density of points can be measured (1000 points in Figure 6).

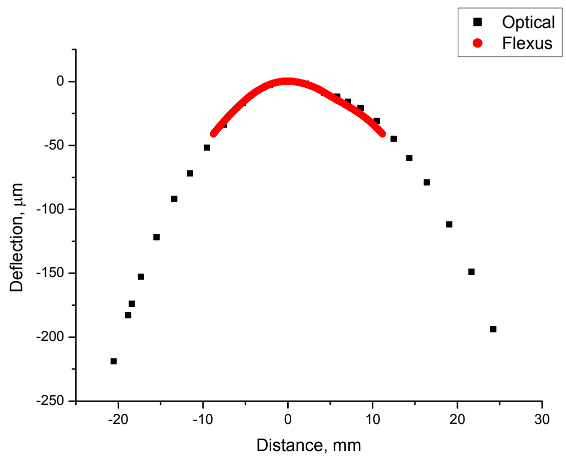

Figure 6 Comparison of measurements of bend of FeCrAlY sample with optical and flexus instruments.

Figure 7 shows a second Labview™ routine that was used to fit the data. This routine calculated both a best fit parabola and a best fit circle to the data. The curvature is then obtained as either the radius of the circle, or as the inverse of half of the second derivative of the parabola. Analytically the parabola fitting is far easier, and gives virtually identical results. It should be noted that the fits deviate from the results due to a kink on one side of the sample. The large number of data points that can be acquired with the Flexus enables a stepwise fitting of the data along the sample such that the variation of curvature along the sample can be measured accurately. For simplicity, an average value of the curvature of the whole sample was used in this analysis.

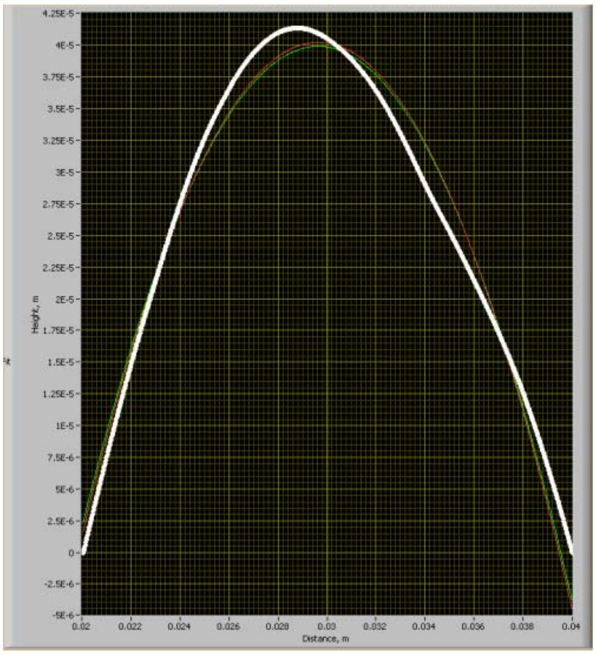

Figure 7 Front panel of Labview™ routine used to fit circles and parabolae to coupon bending data

The Flexus instrument allows the temperature of the sample to be varied. Figure 8 shows the difference in height for two measurements made on the FeCrAlY sample at temperatures of 21 ^{o}C and 78 ^{o}C. This graph shows the change in bending caused by the change in temperature. This change can be used to calculate the difference in thermal expansion coefficient between the substrate and the coating.

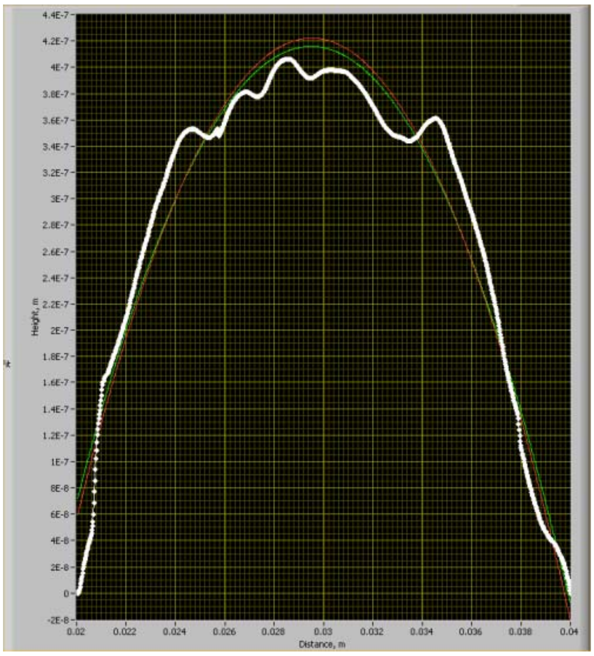

Figure 8 Plot of change in bending for two profiles measured with Flexus instrument at two temperatures of 21°C and 78°C.

Analysis

For coatings that are thin relative to the substrate the simple formula for residual stress σ due to Stoney [1] is appropriate;

$$\sigma = \frac{E h_s^{\ 2}}{(1-\upsilon) 6 R h_c}$$

where s is the residual stress, h_s is the substrate thickness h_c is the coating thickness, E is the modulus of the coating, ν the Poisson ratio for the substrate and R is the radius of curvature of the coupon.

When the coating is thick, this approximate solution is no longer valid, and the formula for σ due to Freund et al [2] can be used

$$\sigma = \frac{E h_s \left(1 + hm(4 + 6h + 4h^2) + h^4 m^2\right)}{6Rhm(1+h)}$$

where σ is the residual stress, $h = h_c/h_s$ with h_c the coating thickness, $m = M_c/M_s$ where M_c and M_s are the biaxial moduli, $M = E/(1-\nu)$, for the coating and the substrate respectively.

It should be noted that both of these formulae are only valid for small deformations of either the coating and the substrate.

The calculated residual stress values can be seen in Figure 9. This shows that the Freund analysis gives a higher value for the residual stress than the Stoney formula, with the ratio between the Freund analysis and the Stoney formula decreasing as the ratio of the thickness of the coating to the thickness of the substrate decreases (Figure 10).

The Freund analysis yields a value for the compressive residual stress in the coating that is reasonably constant with substrate thickness at 903±89 MPa for the coarse grit blasted substrate and 921±65 MPa for the fine grit blasted substrate. Although much of the variation of apparent residual stress calculated with the Stoney formula does seem to have been eliminated by the use of the Freund analysis, the residual stress for the 1.2 mm thick substrate does seem to be a little lower than the values calculated for the other two substrate thicknesses.

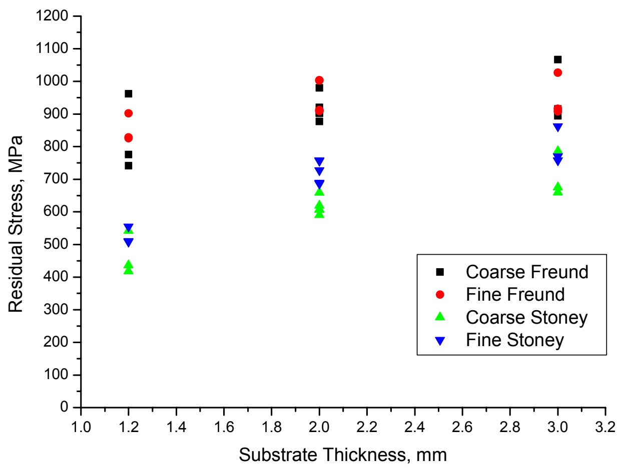

Figure 9 Residual stress in coating calculated by Freund analysis and Stoney formula.

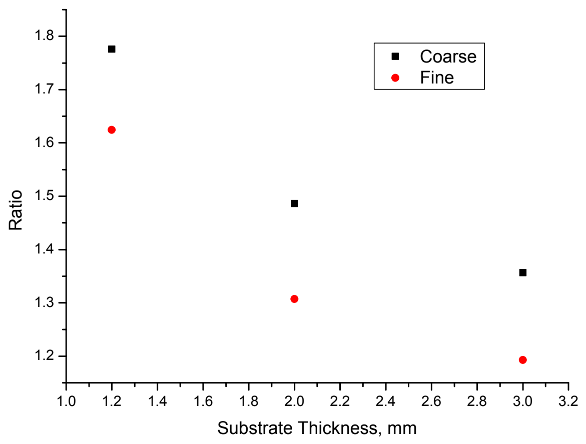

Figure 10 Ratio of value of residual stress calculated from Freund analysis to value calculated form Stoney formula.

Application of the Stoney formula to the FeCrAlY coated sample yields a value of residual stress in the coating of 3.65 GPa.

When profiles are measured at two different temperatures, the difference in bending for small changes in temperature is due to the additional stress generated by thermal mismatch as

$$\frac{d\sigma}{dT} = \left(\frac{E}{1-\upsilon}\right)(\alpha_s - \alpha_c)$$

where α_s is the thermal expansion coefficient of the substrate and α_c the thermal expansion coefficient of the coating.

In this respect a small temperature is a change sufficiently small enough so that no significant variation in physical or structural properties of the coating or substrate occur.

The application of this analysis to the two profiles measured for the FeCrAlY coating yields a value of α_s-α_c of 2.7 x 10^{-6} $^{o}C^{-1}$

Discussion

The experiments and results described here support the use of coupon bending to measure residual stress in coated systems. It is clear that for thick coatings the use of the Freund analysis gives more consistent results and should be used. The difficulty with this is that this requires the measurement of the elastic properties of the coating. This can be difficult to achieve, although techniques are emerging that enable this to be carried out more simply [3].

The use of the Stoney formula should be restricted to thin coatings where it is expected that good results can be achieved.

This technique is attractive because all that is necessary is a reasonably high resolution optical microscope. The measurements can be carried out quite quickly and the calculations can be performed easily with a spreadsheet. The more sophisticated Flexus measurement instrument only worked on the FeCrAlY sample with its relatively smooth surface. The as sprayed surface of the WC/Co sample was too rough to produce meaningful results. Attempts were made to smooth the surface of the WC/Co samples with SiC paper but this was found to be very time consuming. Proper diamond grinding would be necessary to give the required finish and this would make the whole procedure too complex and time consuming.

This technique does require the use of small test coupons rather than components themselves. There may be differences in the coatings that are achieved and hence the residual stresses on coupons rather than components, but these can be minimised by exposing the coupons to exactly the same coating conditions as the components.

The use of the Flexus with the FeCrAlY shows how the change in coupon bending with temperature can yield measurements of the difference in thermal expansion properties between the coating and substrate and hence the coating itself.

Conclusions

The use of coupon bending has been shown to be a simple and effective method for

measurement of residual stress in a thick thermally sprayed WC/Co coating, and in a thin FeCrAlY coating.

For the thick coating and the dimensions of substrate used, the use of the Stoney formula underestimated the residual stress by as much as 40 %. However, the use of the Freund analysis gave consistent results but required the knowledge of the elastic properties of the coating for the analysis. The residual stress in the WC/Co coating was 900-920 MPa.

The Flexus laser triangulation instrument fitted with a furnace enabled the thermal expansion mismatch between coating and substrate to be measured, but this instrument was shown to require smooth surfaces for operation. For the FeCrAlY coating both a measurement microscope and the Flexus machine gave similar values for residual stress in the coating of about 3.5 GPa.

Acknowledgements

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References

1) G G Stoney, Proc Roy Soc, 82A(1909)172
2) L B Freund, J A Floro, E Chason, Extensions of the Stoney Formula for Substrate Curvature to Configurations with Thin Substrates or Large Deformations, Applied Phys Let, 74(1999)1987-1989
3) Private communication, John Nunn, National Physical Laboratory

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