ESRF	**Experiment title:** Residual Stresses in Thermal Barrier Coated Systems	**Experiment number:** HS652
Beamline: ID15a	**Date of experiment:** from: 27-1-99 to: 1-2-99	**Date of report:** 7-7-99 *Received atESRF:* 13 JUIL. 1999
Shifts: 15	**Local contact(s):** Agnes Royer	

Names and affiliations of applicants (* indicates experimentalists):

Prof P.J. Withers[1]
Dr A. Fox[2]
Dr R. C. Reed[2]
M.R. Pinkerton[1]
[1] Manchester Materials Science Centre, Grosvenor St., Manchester M1 7HS.
[2] Department of Materials Science, University of Cambridge, Pembroke St., Cambridge CB2 3QZ.

Report:

Experimental Aims

1.) To measure the in-plane residual stresses in a range of thermal barrier coated systems in order to afford a comparison with theoretical predictions and neutron data.

2.) To investigate the viability of applying a sine squared technique to stress measurement in layered systems.

Samples and Experimental Set-up

A white beam was used having an energy spectrum of 0-175keV, with the detector positioned at a diffracting angle of $2\theta=7.5^\circ$. 100μm slits were used to produce a gauge of length 1.6mm in transmission geometry, and 0.1mm in reflection. A schematic of a typical sample and the two set-up geometries is shown in Fig 1. Unfortunately, experimental problems associated with faults in the translation/rotation stage meant that it was difficult to rotate the sample with the required concentricity.

A range of samples were investigated having topcoats of zirconia, of thickness 180-250μm; bondcoats of NiCrAlY or CoNiCrAlY, of thickness 80-150μm; and substrates of steel or nimonic, of thickness 120-200μm. In our analysis it was assumed that for thin layers the out of plane stress is zero throughout the system ($\sigma_3=0$). It is also assumed that the in-plane stress is isotropic ($\sigma_1=\sigma_2$).

1.)Stress Analysis in Transmission

In-plane strains through the sample thickness were deduced from observed peak shifts in the energy spectra using the Bragg equation. Diffraction profiles taken at 0.2mm increments through a sample are shown in Fig 2, with the bond coat and substrate layers clearly visible. Stress was evaluated using the appropriate continuum elasticity equations, given our assumptions about the stress state. Reference stress free lattice spacings (d_o values) were measured in debonded samples for the top and bond coats, and calculated for the substrate layers by application of the principle of stress balance. Resulting stress profiles are shown in Fig 3 and 4, along with theoretical predictions and available neutron data.

Discussion

The stress profiles qualitatively support the theoretical predictions and neutron data, but deviate in magnitude. The disparities observed may be caused by a geometrical surface effect (Webster et al, J. of Neutron Research, Vol. 3 223-240). Such an effect over the scale of mms could account for the positive tilt with respect to the theoretical predictions seen in several of the layers (bond coat Fig 3, bond coat Fig 4), as well as the smaller than predicted stress ranges in the substrate layers. The magnitude of the surface effects will be quantified in future experiments.

2.)Sin$^2\psi$ technique

This technique is based on the assumption that $\sigma_3 = 0$. It allows σ_1 to be extrapolated from a range of d-spacing measurements taken at various ψ angles, by application of Eqn 1. This method usefully dispenses with the need for a d_o measurement.

$$d_1 - d_3 = \frac{\sigma_1 d_o (1+\upsilon)(\sin\psi)^2}{E} \approx \frac{\sigma_1 d_3 (1+\upsilon)(\sin\psi)^2}{E} \qquad \textbf{(1)}$$

Good data could only be achieved in reflection geometry to a depth of ~0.5mm, due to the long path lengths within the sample. A stress profile derived by this method is shown in Fig 4.

Discussion

The stress profile is qualitatively consistant with the theoretical predictions, but of a different magnitude to both the theoretical predictions and the transmission results. The disparities observed may be due to experimental errors caused by the faulty stage, as well as to a geometrical surface effect. In reflection the variation in path lengths, and hence absorption, across the gauge volume may also produce significant shifts in peak position. However, the technique shows promise, and with refinement should prove to be a useful tool for the investigation of stress in layered systems.

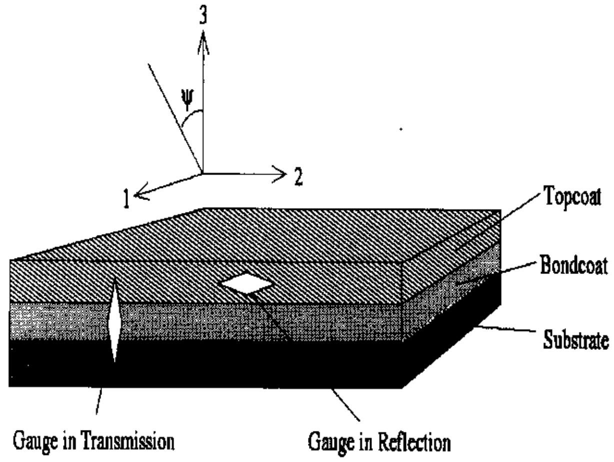

Fig.1 Schematic of layered system illustrating gauge geometries.

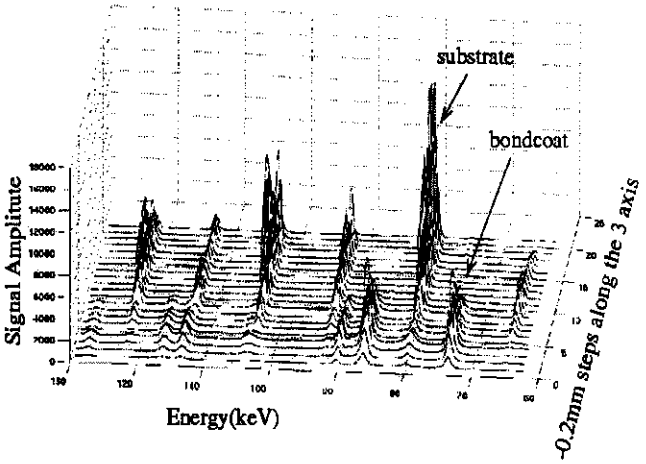

Fig. 2 CoNiCrAlY topcoat transmission spectra (scans 0-10), and steel spectra (scans 7- 20).

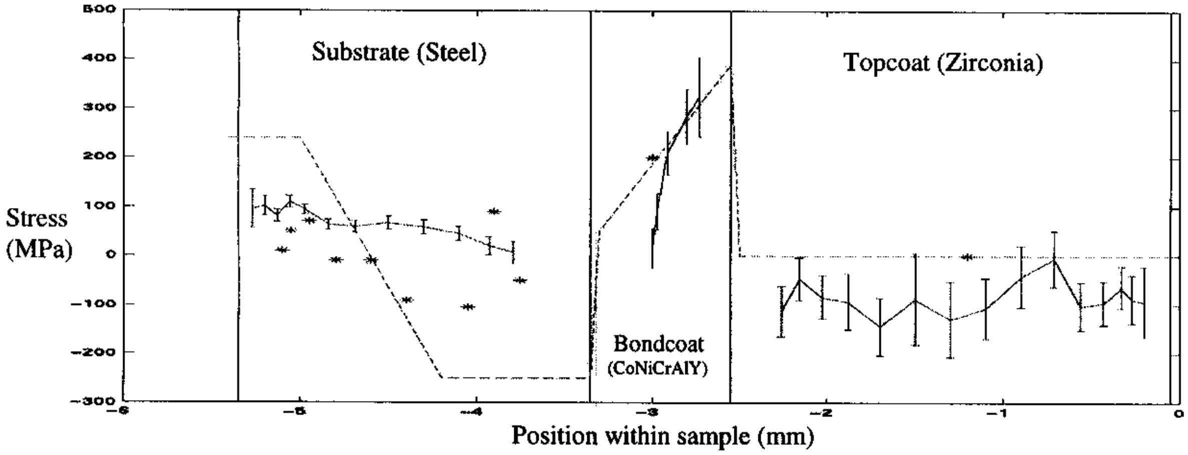

Fig. 3 In-plane calculated stresses (--), transmission measurements (– with error bars). Superimposed are low spatial measured neutron measurements (*) for a similar system but with the topcoat surface -0.4mm.

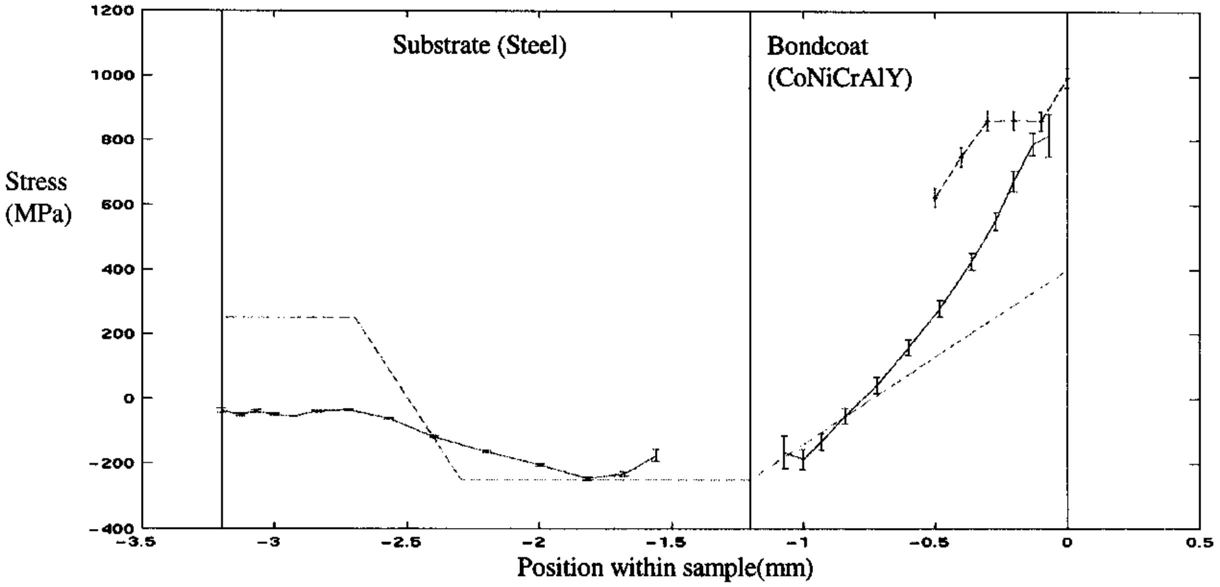

Fig. 4 In-plane calculated stresses (--), transmission measurements (– with error bars), and $\sin^2\psi$ measurements (-- with error bars).