ESRF	**Experiment title:** **Investigation of near-interface radial strain profiles in a tool steel structure coated with plasma-sprayed, stabilised zirconia.**	**Experiment number**: ME29
Beamline: BM16	**Date of experiment**: from: 07/06/2000 to: 09/06/2000	**Date of report**: 24/08/2000
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Report:

This experiment takes place in a study which has the objective of developing the use of a thermal barrier coating for an industrial foundry application. Cylindrical tool steel specimens coated with plasma-sprayed, stabilized zirconia were tested in thermal fatigue by repeated immersion in liquid aluminum. Stress measurements performed by neutron diffraction technique put into evidence an evolution of residual stress from a compressive to a tensile state in a 0.4 mm-thick near-interface region of the substrate, due to thermal fatigue effects. Nevertheless, for technical reasons such as probe resolution and material's diffraction properties, the residual stress states in the coating and in the immediate vicinity of the interface (i.e. at less than 0.2 mm from the interface) could not be investigated accurately enough [1, 2]. The aim of this experiment was to investigate the radial strain profiles in the coating and in the substrate around the interface using synchrotron radiation technique, in order to complete the results given by neutron diffraction experiments.

The investigated samples are cylindrical thermal fatigue specimens (diameter 25 mm) coated with plasma-sprayed, stabilised zirconia. The substrate is a tool steel (typical composition 0.38%C, 5.30%Cr, 1.30%Mo, 0.40%V) and the coating is a zirconium oxide ($Zr0_2$) stabilised with 25 wt% of cerium oxide (CeO_2) and 3 wt% of yttrium oxide (Y_2O_3), and is constituted with a metastable, tetragonal solid solution after deposition [1, 3].

The measurements were conduced on the beamline BM16 using a monochromatic beam (wavelenght 0.3 Angström) and beam size of 60x60x4000 microns. The measured reticular plans were (101) for the zirconia coating ($d \approx 2.990$ Å, $2\theta \approx 5.750°$) and (110) for the steel substrate ($d \approx 2.029$ Å, $2\theta \approx 8.480°$). Two samples were investigated:

- Specimen 1: initial state (non tested in thermal fatigue); coating thickness: 1.7 mm;
- Specimen 2: submitted to 1000 thermal cycles; coating thickness: 0.7 mm.

Reference interreticular distance d_0 were measured on powders ground from substrate and coating of the both specimens in order to calculate strains using the formula $\varepsilon = (d - d_0)/d_0$.

Despite the energy used for this experiment was the highest available on the instrument, we could not reach the interface due to a high absorption of the beam by the material. The measurements could only be performed up to a maximum depth of 250 microns.
Two profiles were measured at different locations in both specimens; in the sample 2 on which the coating was partially delaminated, one measurement were performed in the remaining coating and one in the substrate were the coating had came off during the thermal fatigue experiment. The figures 1 and 2 above show the variation of interreticular distances vs depth.

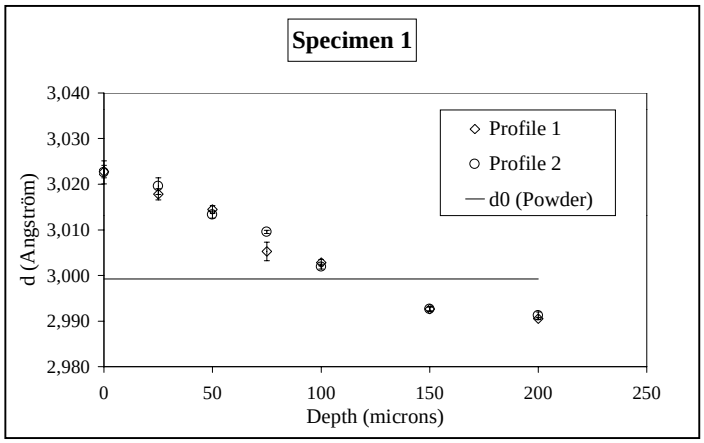

Fig. 1: Profiles of interreticular distance d(101) of the coating measured in specimen 1 (non tested in thermal fatigue).

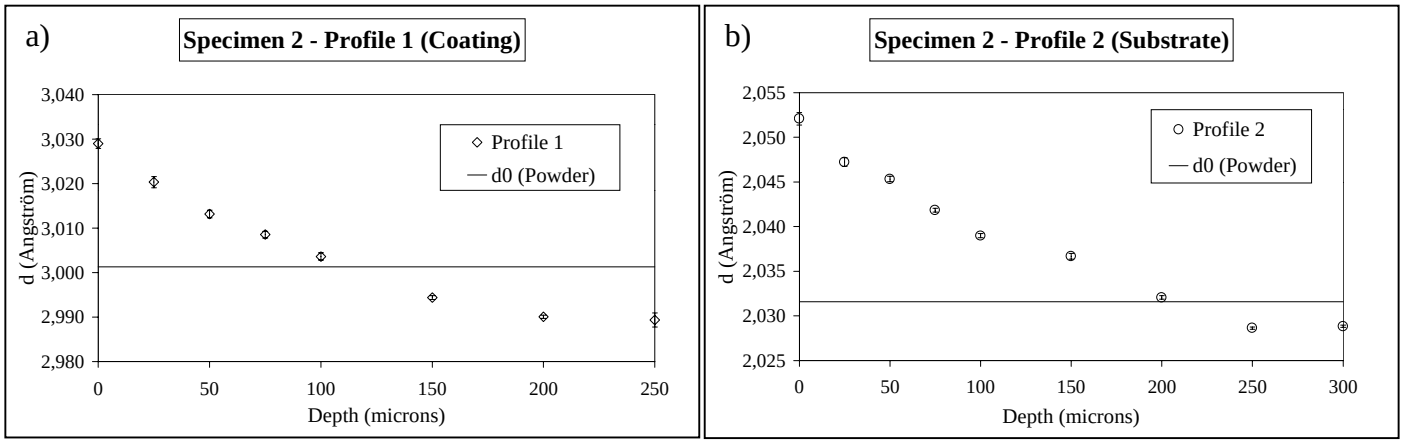

Fig. 2: Profiles of interreticular distances measured in the specimen 2 (tested at 1000 thermal cycles):
a) d(101) of the zirconia coating in the zone non affected by coating failure;
b) d(110) of the steel substrate in the zone of coating delamination.

From the above figures we can see that the variations of interreticular distances are too large to be due to mechanical residual stresses. Further investigation will be needed to confirm a possible gradient of composition in the surface layers of the both materials. A continuation proposal will be presented to the next round in order to investigate the near-interface region using a higher beam energy on the beamline ID15A.

References

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