A Method for Measuring the Depth Profile of Strain through a Coating of Intermediate (0.05 mm to 0.2 mm) Thickness

J. Thornton,[1] D. J. Cookson,[2] S. L. Slater[1]

[1] *Airframes and Engines Division, DSTO, Port Melbourne, Victoria, Australia*

[2] *Australian Synchrotron Research Program, Advanced Photon Source, Argonne, IL, U.S.A.*

Introduction

The use of diffraction techniques to measure residual strains is an established procedure,[1] and both neutrons or x-rays can be used. The penetrating power of neutrons for most materials means that they can be used to map the 3-D distribution of strains in a polycrystalline object. However, the practical depth resolution for neutrons is 1 mm. On the other hand, conventional x-ray techniques are confined to measuring the strain in the top 0.02 mm of most materials due to the high absorption of the low-energy x-rays used. This depth can be extended by polishing, but this destroys part of the sample and can affect the strain distribution. We describe a method of nondestructively examining the depth profile of strains to depths of 0.2 mm using synchrotron radiation and report on the first trials of the method.

Methods and Materials

The principle of the method is shown in Fig. 1. The incident parallel beam of x-rays is collimated by a narrow vertical slit (not shown). The angle between the sample and incident beam is set to

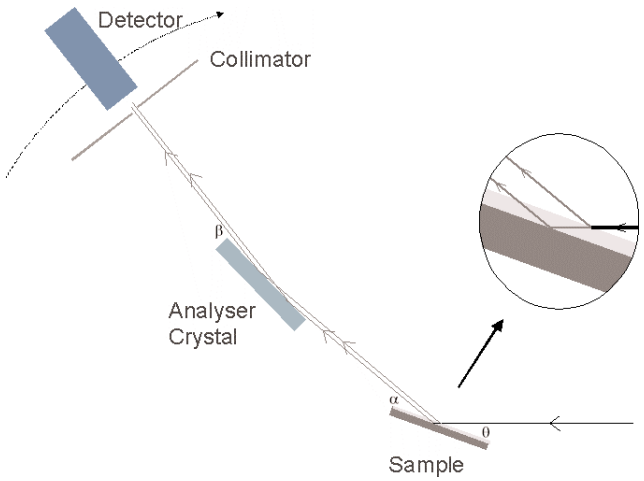

FIG. 1. The experimental apparatus. The light grey surface layer on the enlargement (circle) of the sample represents the coating. The beam leaving the sample is broader than the collimated incident beam.

select the d-spacing for a strong reflection. The incident beam penetrates the entire coating and produces a broad diffracted beam. The intensity variation of the diffracted beam with depth will be dependent on any variation in d-spacing, and hence strain, with depth through the coating. The predictable intensity reduction due to absorption will also be superimposed.

There are two conditions to be met before it is reasonable to relate the nonabsorption component of intensity variation to the variation of strain with depth.

1. The d-spacing variation with depth is not due to composition changes.

2. There are enough crystallites included in the beam to provide a well-averaged signal at all depths.

Many materials, including the coatings in this study, have broad rocking curves. This will also broaden the beam; however, this contribution can be removed by employing a Si(111) analyzing crystal as an angular filter, as shown in Fig. 1.

By rotating the sample relative to the incident beam (θ angle,) the reflection from the coating is selected. For good sensitivity to changes in strain, the highest possible angle that gives a practical signal is chosen. By rotating the analyzer crystal and detector in unison (α angle), the strain value is selected. Rotating only the detector (β angle) is equivalent to scanning through the depth of the coating.

The x-ray energy was chosen to be just below the absorption edge for zirconium to provide good penetration (the more penetrating energies of 30 keV and above were not used because of the associated reduction in sensitivity to strain variations). All the intensities measured by the detector were normalized by the incident flux as measured by an ionization chamber before the sample.

The samples used for this preliminary investigation were produced by plasma spray.[2] In particular, the results shown refer to a coating of 0.3 mm of zirconia on a 2-mm-thick nickel superalloy substrate.

Results

Figure 2 shows the result of scanning though the depth of a plasma-sprayed zirconia coating at three different d-spacings. One d-spacing at the peak of the zirconia rocking curve (strongest), one at a larger d-spacing (compression), and one at a smaller d-spacing (relaxed). The larger d-spacing corresponds to greater compression in the plane of the coating for these atomic

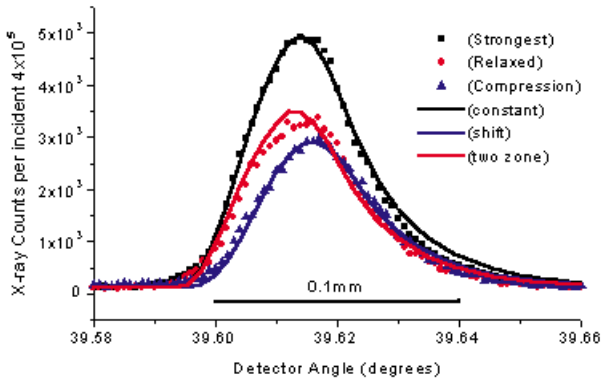

FIG. 2. The data points show the results of three detector scans with α set at 39.56° (compression), 39.615° (strongest), and 39.645° (relaxed). The detector and incident beam slits were both nominally 0.05 mm wide. The curves show the expected profile of x-ray counts versus detector angle given the coating density of 5.1 g/cc.

layers orientated parallel to the surface. Figure 3 shows models of the d-spacing distributions need to give good fits to this data—the curves in Fig. 2.

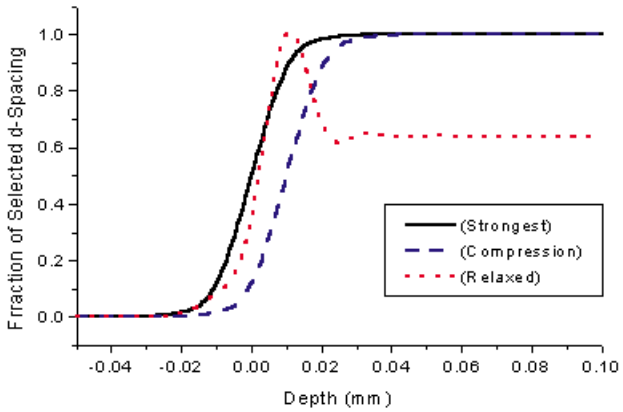

FIG. 3. Model distributions of the variation of the selected d-spacings with depth. Note that they are convoluted with absorption and geometry functions before being used to produce the curves in Fig. 2.

Discussion

The model distributions of Fig. 3 show a relaxed surface with more compressed material further in. This is to be expected with this plasma-sprayed coating. The heat treatment it received would have caused the bulk of the ceramic coating to be placed in compression by the metallic substrate. However, as the coating surface (0.03 mm) is rough and therefore contains gaps, full compression of the surface region would not have occurred. It was clear from these preliminary experiments that the method was limited by the low count rate at the detector. This limited the study to shallow depths (0.1 mm) and also limited the depth resolution by requiring large slit widths (>20 µm). There is scope to improve the count rate without compromising the depth resolution by using an analyzer with a wider acceptance angle. Depth resolution can be improved by using an asymmetrically cut analyzer crystal as a beam expander.

References

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