THE MECHANICS OF INTERFACE FRACTURE: (4) ROOM TEMPERATURE SPALLATION OF α-ALUMINA FILMS GROWN BY OXIDATION

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Abstract: An analytical mechanical model is developed to predict the room temperature spallation behavior, including the separation nucleation, stable and unstable growth, and final spallation and kinking off, of α-alumina films grown by oxidation on Fe-Cr-Al alloy. The predictions from the developed model are compared against experimental results and excellent agreement is observed. The work reveals a completely new failure mechanism of thin layer materials.

1. Introduction

Tolpygo and Clarke [1, 2] presented an excellent experimental study on the room temperature spallation failure of α-alumina films grown by oxidation on Fe-Cr-Al alloy. Their observations are remarkable and thought-provoking. In their work, $\alpha\text{-Al}_2\text{O}_3$ films of different thicknesses were formed on the surface of Fe-Cr-Al heat-resistant alloy substrates of different thicknesses by oxidizing them at 1200°C for different time periods. Then, the film-substrate material systems were cooled to room temperature at different cooling rates. Cooling causes an increase of compressive in-plane residual stress due to thermal expansion mismatch. Their major observations were as follows: No separation or spallation failure occurs during cooling at any rate. For specimens cooled at rates in the range $5°–200°C$ min^{-1}, circular interfacial separations between the film and the substrate nucleate, grow in separation distance and propagate radially, all after reaching room temperature, at a constant compressive residual stress far below the critical buckling stress, and apparently spontaneously. After a period of slow and stable growth, some of these separations then grow abruptly and the oxide spalls off. The present work hypothesizes that pockets of energy concentration (PECs) exist due to dynamic and non-uniform plastic relaxation or creep in the film and Fe-Cr-Al alloy substrate during cooling, and may be the driving energy for room temperature spallation failure. Based on this hypothesis, an analytical mechanical model is developed [3, 4] to predict the spallation behavior, including the separation nucleation, stable and unstable growth, and final spallation and kinking off.

2. Results

The model predictions are compared with experimental results [1, 2] (Figure 1). The solid dots represent a series of measurements of the size of individual separations as a function of time at room temperature. The time of 0 min corresponds to the moment when the specimen was placed under the microscope and its temperature was close to ambient. Data are shown for four different separation bubbles on a single specimen after isothermal oxidation for 25 h at 1200°C and cooling at 20°C min^{-1}. The bubbles were successively monitored using optical microscopy. The whole process includes nucleation, stable growth, unstable growth, and final

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spallation. The nucleation of separation bubbles was not recorded due to the difficulty of making timely observations of nucleating bubbles using this monitoring technique. Stable growth, however, with a radius far smaller than the critical buckling value, was readily observed. At a certain critical radius, shown by the dashed line from the model, which is again far smaller than the critical buckling radius with a clamped edge condition, unstable growth abruptly occurs. It is pertinent that all four separations start unstable growth at approximately the same radius as predicted by the model, and then all eventually spall off also at approximately the same radius as predicted by the model, and shown by the top solid line.
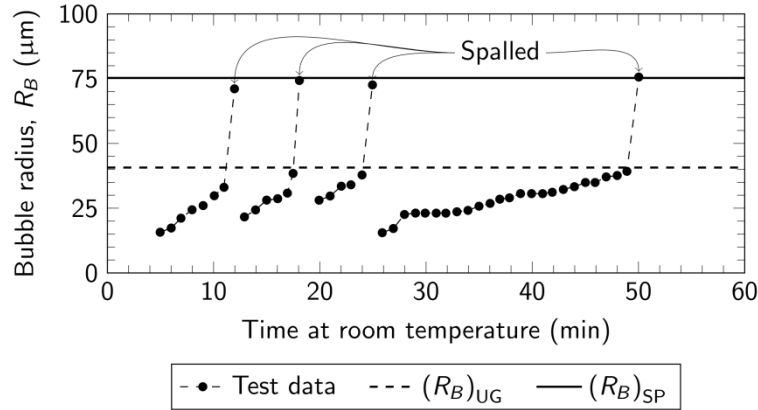

Figure 1. Separation bubble radius versus time at room temperature with film thickness $h = 4.9\ \mu m$ and compressive residual stress in film $\sigma_0 = 4.46\ GPa$

3. Conclusions

The present mechanical model, based on the PEC hypothesis, predicts very well several aspects of the room temperature failure of α-alumina films grown by oxidation, including the initiation of unstable growth, and the size of spallation or kinking off. One third of this energy is used to bend the separation outwards to form a bubble after nucleating the interface separation using two thirds of its energy. Stable growth of the bubble is then driven by the bubble energy. The bubble becomes a buckle at a critical radius at which point unstable growth starts, driven both by the bubble energy and by buckling. The bubble energy reaches its maximum value at a certain radius. The spallation occurs when the PEC energy is big enough to break the film.

References

[1] V.K. Tolpygo, D.R. Clarke. Spalling failure of α-aluminia films grown by oxidation. I. Dependence on cooling rate and metal thickness, *Mater. Sci. Eng.* 2000;A278:142–150.

[2] V.K. Tolpygo, D.R. Clarke. Spalling failure of α-aluminia films grown by oxidation. II. Decohesion nucleation and growth, *Mater. Sci. Eng.* 2000;A278:151–161.

[3] S. Wang, C.M. Harvey and B. Wang. Room temperature spallation of α-aluminia films grown by oxidation. Under review.

[4] C.M. Harvey, B. Wang and S. Wang. Spallation of thin films driven by pockets of energy concentration. Under review.