doi:10.1017/S1431927613011173
Microsc. Microanal. 19 (Suppl 2), 2013

Fracture Properties of Diffusion Aluminide Bond Coats: An *In Situ* SEM Study of Microbeam Bending

Nagamani Jaya B[1], Sanjit Bhowmick[2], S. A. Syed Asif[2], Oden L. Warren[2], Vikram Jayaram[1], Sanjay K. Biswas[3]

[1]Materials Engineering, Indian Institute of Science, Bangalore, Karnataka 560012, India
[2]Hysitron, Inc., Minneapolis, Minnesota 55344, USA
[3]Mechanical Engineering, Indian Institute of Science, Bangalore, Karnataka 560012, India

PtNiAl bond coats are diffusion aluminides deposited on superalloy substrates of turbine blades by initially electrodepositing a layer of Pt on a Ni-based superalloy substrate and then pack aluminizing the substrates to obtain a β-phase matrix [1]. Aluminide coatings are heterogeneous composite-like structures consisting of a number of precipitates of varying morphology, size, composition, and distribution, and are rich in refractory elements. The coatings being Al-rich provide oxidation resistance to the superalloy blades by forming a stable protective α-Al_2O_3 layer on the surface. Nevertheless, bond coats are subject to thermomechanical stress and impact as the components are exposed to severe operating conditions causing failure by cracking and delamination [2]. Hence, it becomes important to optimize the bond coat composition not only for good oxidation resistance, but also to achieve the best possible fracture properties across the coating thickness.

The compositional and microstructural gradients that develop in diffusion aluminide bond coats require adoption of small-scale testing methods to determine fracture properties at specific locations of the coating. In this study, an *in situ* SEM-based bending technique was applied to micro-scale beams that were machined at different compositional zones of the coating. Three coating compositions with varying initial Pt thickness and changing Ni:Al ratios across the thickness were chosen. The microbeams were machined in a doubly clamped configuration with nominal dimensions of 100 μm in length, and 10 μm x 8 μm cross section using focused ion beam [3]. Pre-notches at the tensile edges of the beams were made by low current milling. The beam dimensions were chosen to conform to plane strain loading condition. The in situ bending experiments were conducted using a PI 85 SEM PicoIndenter (Hysitron, Inc.). TriboScan software (Hysitron, Inc.) was used to monitor, capture and analyze the load-displacement data. The load-displacement data and the real-time video of crack initiation and propagation were synchronized and captured during the experiment using a frame grabber in communication with the TriboScan software, which aided the post-experimental analysis. The pop-in loads corresponding to crack initiation were used to calculate the K_{IC} (Fig. 1a). Extended finite element method with the assumption of linear elastic fracture mechanics was applied to determine stress distribution and stress intensity factors at the crack tip. Digital Image Correlation was used to determine crack tip opening displacements by post processing the SEM images that were acquired during the experiments. Similar experiments on bulk stoichiometric single-crystal NiAl was conducted for calibration. Stable crack propagation was used to determine R-curve behavior, Fig. 1b. It has been observed that the initiation fracture toughness increases from 5 to 15 $MPa.m^{1/2}$ with increasing Ni:Al ratio across the coating thickness from the free surface towards the coating-substrate interface for a particular PtNiAl coating as shown in Fig. 2a. For a given thickness, the coating with higher Pt content showed higher K_{IC}. Although cleavage fracture was observed in all the cases at room temperature, crack propagation was catastrophic in the Al-rich zone and stable in the Ni-rich zone (Fig 1b and 1c). This could be attributed to the change in precipitate morphology from small equiaxed shape in the Al-rich zone to large elongated shape in the Ni-rich zone. While the spherical precipitates do not impede crack growth, the elongated particles resist crack propagation by forcing it to kink along the interfaces, and also act as bridging agents, enhancing the fracture toughness. A CAD model of PI 85 that was used in the *in situ* experiments is shown in Fig. 2b.

References:

[1] D. K. Das, V. Singh and S. V. Joshi, Metallurgical and Materials Transactions A **29** 8 (1998), p. 2173.

[2] N. Padture, M. Gell and E. h. Jordan, Science **296** 5566 (2002), p. 280.

[3] N, Jaya B, V, Jayaram and S. K. Biswas, Philosophical Magazine special issue on Nanomechanical Testing in Materials Research and Development, **92** 25-27 (2012), p. 3326.

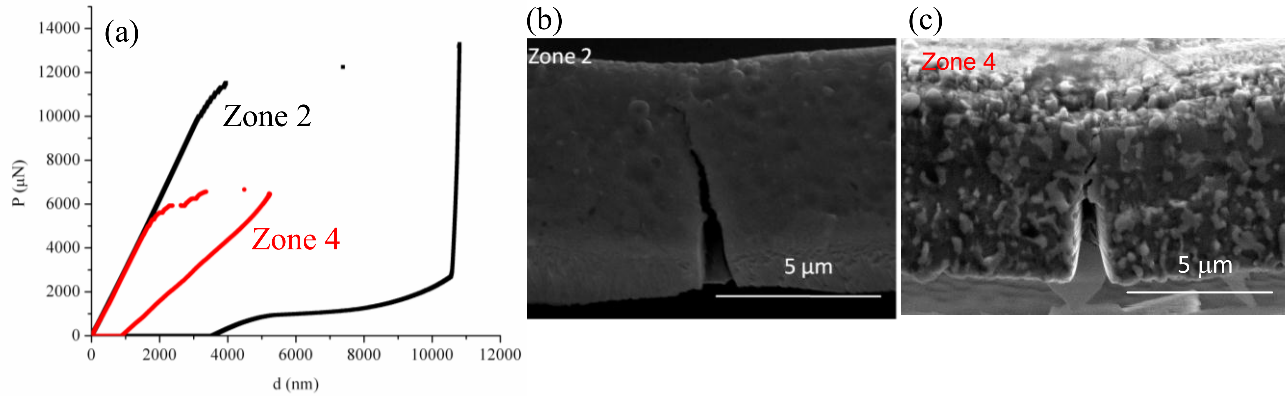

Figure 1: (a) Catastrophic and stable fracture shown in load – displacement curve, (b) and (c) crack trajectory in microbeams in zone 2 and zone 4. Zone 2 data in (a) shows single large displacement jump at a particular load, while zone 4 shows multiple incremental jumps with increasing loads. The corresponding crack path in zone 2 shows a through thickness crack while in zone 4 shows crack arrested at precipitates.

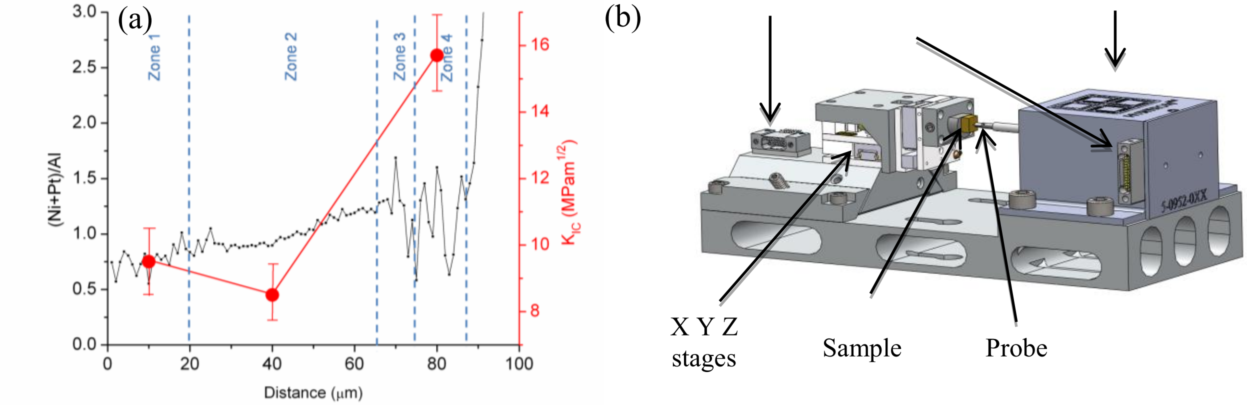

Figure 2: (a) K_{IC} across coating thickness as determined from *in situ* test, superimposed on the Ni:Al equivalent ratio, (b) A CAD model of PI 85 showing different components.