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# In situ fracture mechanics of oxide-mediated Ni-Al-based nanometallic multilayers at the micro- and nanoscale

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## Abstract

Nanoscale multilayers provide a powerful platform to tune mechanical behavior through interface design, yet the fracture response of oxide-mediated nanometallic architectures remains insufficiently understood. This gap is particularly important for multilayers combining reactive metals with amorphous oxides, where competing effects of confinement, interfacial strength contrast, and local ductility can produce unexpected combinations of hardness and toughness. In this work, we investigate the nanomechanical behavior of Ni–$Al_2O_3$–Al–$Al_2O_3$ multilayers composed of 15 nm Ni and 15 nm Al layers separated by amorphous $Al_2O_3$ interlayers of variable thickness (0, 2.5, and 8 nm). The design enables a systematic comparison between interfacially intermixed Ni–Al reference multilayers and oxide-mediated systems in which interdiffusion is suppressed and fracture pathways are redirected by the presence of brittle–ductile–brittle stacking sequences at the nanometer scale. The films were characterized systematically using an experimental chain combining nanoindentation, in situ SEM clamped-beam micro-fracture testing, and in situ TEM tensile experiments. Nanoindentation reveals that hardness decreases with increasing $Al_2O_3$ thickness, demonstrating that the exceptional strengthening previously observed in intermixed pure Ni–Al multilayers is progressively diminished as amorphous interlayers interrupt interfacial mixing, modify bonding,

and relax the confined slip pathways. Despite this reduction in strength, the oxide-mediated multilayers exhibit unexpected fracture resistance. From stable crack-growth experiments analyzed with an elastic–plastic J-integral framework, we obtain JIc ≈ 100 J/m2 and KJc ≈ 2.5 MPa m1/2. These values are noteworthy considering the significant volume fraction of nominally brittle $Al_2O_3$, especially in the 8 nm-interlayer architecture, and they place the fracture performance of these multilayers on par with fully metallic Ni–Al systems. Mechanistic insights come from in situ TEM tension, where the tensile axis is parallel to the layers and crack propagation occurs perpendicular to the layers, closely mirroring the SEM fracture tests. The fracture process unfolds as a repeated sequence: (i) initial failure in the nanoscale-ductile $Al_2O_3$ and Al layers; (ii) crack arrest at the weaker $Al_2O_3$–Ni interfaces, which initiates controlled delamination and interfacial energy dissipation; (iii) pronounced plastic flow and necking in the Ni layers, serving as ductile bridges; and (iv) restart of the sequence as the crack advances. This multi-stage, interface-mediated process produces a highly dissipative fracture zone that accommodates both brittle and ductile constituents in a cooperative manner. Overall, these results reveal a previously underexplored toughening mechanism in oxide-mediated nanometallic multilayers. They demonstrate how the hierarchy of interfacial strengths, combined with nanoscale confinement and layer-by-layer switching of deformation modes, allows decoupling hardness from fracture resistance. Our findings therefore provide design principles for creating hard, damage-tolerant nanolaminate coatings with tailored energy-dissipation pathways.

**Keywords:**

In situ electron microscopy Micro-/nanomechanical fracture Oxide-mediated nanolaminates