ELASTIC-PLASTIC FRACTURE TOUGHNESS OF ELECTRODEPOSITED NI-W THICK FILMS USING IN-SITU MICROCANTILEVER BEND TESTS

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Nanocrystalline materials have been shown to exhibit properties superior to their coarse-grained counterparts. Nanocrystalline Ni-W is no exception and is a promising replacement for nanocrystalline Ni and hard chromium, with prospect in the electronics, microfabrication technology, automobile, and aerospace industries. In spite of having desirable properties, Ni-W exhibits a certain brittleness that if not mitigated, could lead to premature failure. This study investigates the fracture behavior of Ni-W and establishes structure-property relationships via correlation to the microstructure. Due to non-negligible plastic yielding, conventional linear elastic fracture mechanics was insufficient in quantifying the fracture toughness, and novel elastic-plastic fracture mechanics had to be used.

High-quality Ni–21 at.% W films were processed via electrodeposition in a controlled sulfate-citrate bath under direct current mode and heat treated in a reducing atmosphere at 437°C and 728°C for 24 hours. The microstructure, characterized via TEM and STEM techniques, ranged from being amorphous and nanocrystalline to ultrafine-grained with second phase precipitation. For mechanical testing, notched microcantilevers were fabricated in a dual-beam FIB, tested with in-situ bending under load-control, and analyzed with J-integral interpretation. The J-integral requires knowledge about the crack growth process, which is directly related to the stiffness (compliance) of the beam. The compliance of the beam was deduced from the unloading slope; thus, testing was conducted under quasi-static conditions using multiple partial unloads. Finite element analysis was used to establish the relationship between the crack length and the compliance, which allowed experimental compliance measurements to be linked to a particular crack length. Nanoindentation was conducted to complement the fracture analysis with hardness data and provide modulus values to input into the finite element model.

The as-deposited and 437°C alloys were nominally amorphous with mesotexture as evidenced by the assembly of clusters of grains into a cauliflower-like structure. This morphology, however, was absent from the fracture surface of the as-deposited alloy, which was instead characterized by extensive plastic deformation (also demonstrated by the load-displacement record). The 437°C alloy, however, failed in a brittle manner, but closer examination revealed dimple features, such as that commonly associated with microvoid coalescence. The dimple size was similar to the size of grain clusters, thus rendering the boundaries of the grain clusters as preferential crack paths, in which case deformation by grain boundary sliding would be a possible corresponding mechanism. The 728°C sample failed in an intergranular manner. Overall, the fracture toughness decreased after annealing at 437°C and remained constant at 728°C, while the hardness increased significantly after annealing at 437°C and regressed back at 728°C. Thus, there may be no benefit to annealing to such high temperatures. From a microstructural optimization standpoint, there appears to be a trade-off between the improvement in hardness and deterioration in fracture toughness after annealing at 437°C. It may be wise to use intermediate temperatures to increase the hardness while mitigating the decline in toughness.

Being a relatively new field, the practice of quantifying the fracture toughness of elastic-plastic materials on the micron scale is not fully developed. Many intricacies were encountered in shaping the methodology for testing and analysis in this study. For example, given the ambiguity and uncertainty with determining the extent of stable crack growth, two different methods were used to deduce the critical J value from the J-R curve. Despite minimal stable crack growth, the J-integral was successfully used in this study to quantify the fracture toughness.