ENVIRONMENTAL RELIABILITY AND CRACK PROPAGATION RESISTANCE OF 3D-PRINTED ALD-COATED NANO-CERAMICS

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3D-printed micro- and nano-architected ceramic metamaterials currently emerge as a class of lightweight materials with exceptional strength and stiffness. However, their application is hampered by the lack of knowledge of their mechanical reliability. Recently, the sensitivity of nano-ceramics’ crack propagation resistance to environmental conditions, triggered by the unavoidable presence of surface flaws introduced by the TPP-DLW 3D printing and pyrolization post-processing, has been evidenced [1], with a reduction of 20% in the average fracture toughness value reported at high relative humidity levels of testing ($RH > 60\%$) from the generally performed low-humidity-based testing.
In this work, we present a further step in understanding and improving the environmental reliability of those materials via the possibility of controlling their surface characteristics and obtaining enhanced independence of the crack propagation resistance to the testing relative humidity. To this scope, a population of TPP glassy carbon micro-pillars and uniform 2.5D structures have been coated with highly conformal ALD-deposited Al_2O_3 thin films (having a uniform thickness of 50 to 100 nm). The study focused on the application of pillar splitting protocols to elucidate the effects of the deposited films on the fracture toughness of 5.8 μm diameter micro-pillars (see *Figure*) as a function of two extremal testing RH levels ($< 5\%$ and $> 60\%$). A systematic study of the combined film-substrate mechanical properties variation (elastic modulus and hardness) with the testing conditions has also been performed on the 2.5D uniform structures.

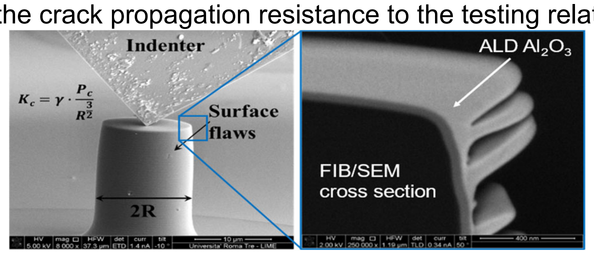

Figure 1 - Testing configuration for a TPP 5.3 µm micro-pillar coated with ALD conformal 50 nm thick Al_2O_3 film.

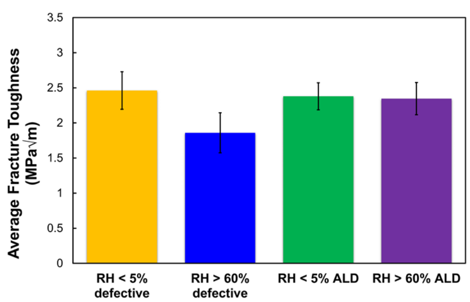

Figure 2 – Average K_c value of non-coated and ALD alumina coated 5.3 µm pillars at two different RH levels: <5% and >60%.

The study evidenced that the fracture toughness of ALD coated pillars is incremented from that measured for pristine defective high-humidity tested pillars (*Figure*), gaining independency of the reliability performances of TPP glassy carbon from the relative humidity conditions. The Surface Free Energy of the alumina films has been studied via a novel nanoindentation based testing protocol by the authors [2] to provide an enhanced understanding of the measured crack propagation resistance retention at high relative humidity levels envisaged as arising from a combined sealing effect of the pre-existent surface flows by the deposited thin film and prevention of water diffusion due to enhanced hydrophobic properties.
Moreover, the average K_c value for the ALD coated samples does not reach the values recorded for the non-coated pillars at low-humidity conditions. Studies on the residual stress state of the deposited films were conducted via high-depth resolved FIB-DIC ring-core testing protocols [3]. It was found that tensile residual stresses within the coating interact with the crack as an additional reservoir to the opening system. These new experimental observations suggested that the environmental reliability of glassy carbon 3D printed structures could be effectively augmented via ALD growth by carefully optimising the deposition process.

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[3] M. Sebastiani, E. Rossi, M.Z. Mughal, A. Benedetto, P. Jacquet, E. Salvati, A.M. Korsunsky, Nanomaterials 10 (2020) 853.