THERMO OXIDATIVE AGING OF POLYMERS AND POLYMER-MATRIX COMPOSITES STUDIED WITH CYCLIC INDENTATION

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The growing use of polymer-matrix composites in aircraft structures leads to the necessity of understanding the degradation phenomena due to their exposure to high temperatures. For T<Tg, the main degradation phenomenon is thermo-oxidation that promotes the formation of a dark brittle oxidized layer. Depending on the aging conditions and the type of polymer material, the oxidized layers can reach a thickness of several hundreds of microns. A convenient way to study the gradients of mechanical properties induced by thermal oxidation is the instrumented indentation test at the relevant scale [1]. However, the classical indentation procedure [2] used in previous studies is not able to capture viscous nature of polymer behavior. In this work, local mechanical behavior of thermally aged polymers and polymer composites has been investigated through a cyclic indentation loading. Using this indentation method, it is possible to study the polymer behavior in a more complete way, focusing not only on the elastic unloading, but also on the time-dependent aspects.
The aging of three epoxy resins (PR520, Tactix and RTM6) in pure state and reinforced with carbon fibers has been characterized by Ultra-Micro Indentation on force-controlled Fischerscope H100C equipment. These materials were aged at 150°C, in air at atmospheric pressure up to 1000 hours and under pure oxygen environment at a pressure of 2 bar, to accelerate the oxidative aging.

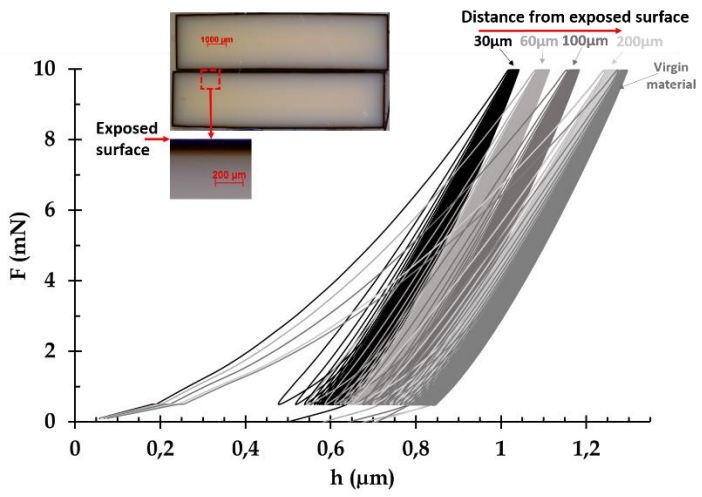

Figure 1 –Ultra-Micro Indentation test performed through the oxidized layer of an aged PR520 resin sample.

Firstly, the oxidized layer of aged bulk polymer samples has been characterized at different distances from the external surface. *Figure 1* presents an example of raw indentation data obtained with the cyclic procedure on an aged specimen of PR520 resin. This example clearly demonstrates a change in the mechanical response of the polymer due to aging. In particular, the maximum displacement is lower close to the surface, but similar to those of the virgin material at the sample core. The Elastic Indentation modulus (*EIT*) [2] calculated at each cycle, is higher at the surface. However, the area of hysteresis loop, which characterizes the time-dependent behavior, is rather similar for the aged and virgin polymer. This can be interpreted as a sign that only elastic behavior is affected by the oxidative aging.
The cyclic indentation method is then applied to the aged polymer composite samples. Previous work showed that fiber debonding and micro-cracks due to oxidative aging of the outer polymer layers offer new paths to oxygen to penetrate into the bulk material [3]. The cyclic indentation method has been applied to study the gradients of the local mechanical behavior of the aged polymer in composite close to the exposed surface and to the fibers. Results are compared to those obtained on pure polymer samples.

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[2] W.C. Oliver, G.M Pharr, Mat. Res. 7 (1992)
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