

## Effect of Additives on Post-hydrogen Decompression Microstructure and Mechanical Behaviour of PA11 Involved in Type-IV Hydrogen Tank Liners

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**Abstract :** In light of the ongoing energy transition, "Infrastructure developments" for hydrogen transportation and storage raise studies on the materials employed for hyperbaric vessels. Type IV tanks represent the most mature choice for gaseous hydrogen storage at high pressure - 70MPa. These tanks are made of a composite shell and an internal hydrogen-exposed polymer liner. High pressure conditions lead to severe mechanical loading requiring high resistance. Liner is in contact with hydrogen and undergoes compression - decompression cycles during system filling and emptying. Stresses induced by this loading, coupled with hydrogen diffusion, were found to cause microstructural changes and degradation of mechanical behaviour after decompression phase in some studies on HDPE. These phenomena are similar to those observed in elastomeric components like sealing rings, which can affect permeability and lead to their failure. They may lead to a hydrogen leak, compromising security and tightness of the tank. While these phenomena have been identified in elastomers, they remain less addressed in thermoplastics and consequences post-decompression damages on mechanical behaviour was not studied either. Different additives are also included in liner formulation to improve its behaviour. This study aimed to better understand damage micro-mechanisms in PA11s exposed to hydrogen compression-decompression cycles, and understand if additives influence their resistance. Samples of pure, plasticized and impact-modified PA11s are exposed to 1, 3 and 8 pressure cycles including hydrogen saturation at 70MPa followed by severe 15-second decompression. After hydrogen exposure and significantly later than full desorption, the residual mechanical behaviour is characterized through impact and monotonic tensile tests, on plain and notched samples. Several techniques of microstructure and micro-nano damage characterization are carried out to assess whether changes in macroscopic properties are driven by microstructural changes in the crystalline structure (SAXS-WAXS acquisitions and SEM micrographs). Thanks to WAXS acquisition and microscopic observation, the effects due to additives and pressure consequences can be decorrelated. Pure PA11 and PA11 with a low percentage of additives show an increase in stress level at the first yielding point after hydrogen cycles. The amplitude of the stress increase is more important in formulation with additives because of changes in PA11 matrix behavior and environment created by additives actions. Plasticizer modifies chain mobility leading to microstructure changes while other additives, more ductile than PA11, is able to cavitate inside PA11 matrix when undergoing decompression. On plasticized formulation, plasticizer migration are suspected to enhance impact of hydrogen cycling on mechanical behaviour. Compared to the literature on HDPE and elastomers, no damages like cavitation or cracking could be evidenced from SAXS experiments on every PA11 formulation tested. In perspectives, on all formulation, experimental work is underway to confirm influence of residual pressure level after decompression on post-decompression damages level.

**Keywords :** additives, hydrogen tank liner, microstructural analysis, PA11

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