

## The Use of Autoclaves to Assess the Oxidation Resistance of Plastics made from Polyolefins

### 1 Introduction

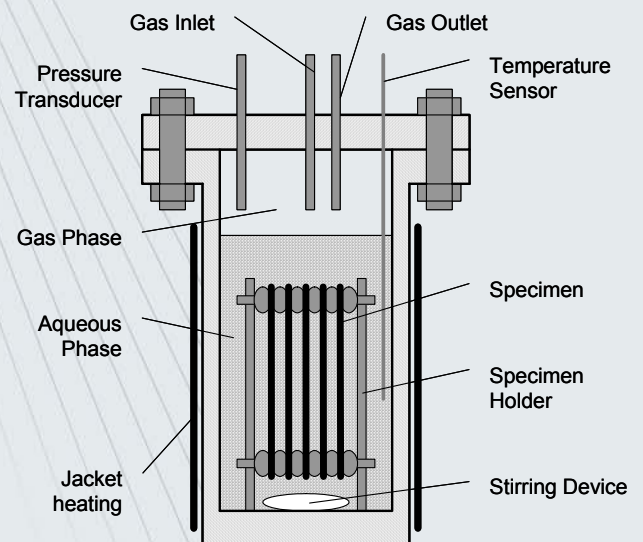
The durability of polymeric materials for the use in civil structures with a scheduled service life of more than 25 years has to be proven by accelerated tests. This is the case for the aging of construction materials in particular. Their durability might be essential for the safety of a construction and has to be taken into account for maintenance purposes. A repair of the construction due to an insufficient durability of polymeric parts can cause enormous financial expenses. Based on the recent EOTA Guidelines (Assumption of working life of construction products in Guidelines for European Technical Approvals and Harmonized Standards, 1999) a proof of an expected service life of at least 100 years is requested for relevant building products. This topic is part of the discussions in technical and standardization committees which are working on recommendations and rules for a durability assessment.

### 2 Test Procedure

Practical investigations to assess the durability of polymeric materials and products are done by using defined test conditions (e.g. elevated temperature) to cause an accelerated aging which affect typical material properties (e.g. mechanical tensile strength). The evaluation can be done either as a simple index test for rough estimates based on minimal requirements or as a more significant result based on the time dependent behaviour of relevant material properties. The second option includes an extrapolation using the stress parameters of the tests and their interdependence. To get realistic results the circumstances must be chosen according to the practical use of the material.

The working group VI.33 of the Federal Institute for Materials Research and Testing (BAM) in Germany has developed a method to assess the oxidation resistance of geosynthetics made of polyolefins, such as polypropylene (PP) and polyethylene (PE), by using autoclaves. The combination of specific temperatures, an increased oxygen pressure and the presence of an aqueous medium allows an acceleration of the auto-oxidation with simultaneous extraction of additives.

These autoclave tests represent a promising method to replace conventional oven tests. The moderate temperatures during the test procedure provide suitable conditions to assess the effect of stabilizers and anti-oxidation-agents at their working temperatures. Due to the high oxygen pressure and the continuous circulation of the aqueous solvent a sufficient amount of oxygen is available for the auto-oxidation of the polyolefins.



Scheme of an autoclave

In cooperation with working group VI.33  
„Chemical Resistance of Polymers“ of the

The time frame for the autoclave tests depends on characteristics and quality of the material and the prevailing application conditions. Generally three temperature levels of 60 °C, 70 °C and 80 °C at 50 bar oxygen pressure and two additional oxygen pressure levels of 20 and 10 bar at 80 °C have been proven to be suitable for the testing of polymeric materials. In this case the time needed to get reliable results varies from approximately 300 days for the lowest temperature to 30 days for the highest.

That allows both, rather short index tests at elevated temperatures and a calibration of these short term tests with long-term tests at lower temperatures. It is a comfortable approach to estimate the residual service life of polyolefins in a construction with accelerated tests. The described standard procedure for synthetics can be modified according to specific environmental conditions. This might be interesting for investigations regarding the aging effect of water which penetrates the mountain rock until it reaches a liner and drain system of a tunnel. Amongst other components the infiltrating water may contain heavy metal ions which will have a catalytic effect on the oxidation.

### 3 Advantages

The defined liquid environment inside the autoclaves leads to a new quality of testing. Combining the various test media and conditions with each other is a promising approach to get a more reliable formulation for

- a fast and efficient procedure with enhanced differentiating ability compared to the conventional oven test, which is based on temperature increase and evaporation of stabilizers only
- a moderate variation of the influencing stresses (temperature  $\leq 80$  °C)
- a simulation of the most essential characteristics of the installation environment (temperature, oxygen concentration, extraction of additives and anti-oxidants - in a specific aqueous surrounding)
- modelling an individual stress scenario to evaluate the durability for various application areas of the polymeric materials
- an improved test with a higher efficiency regarding the results and a shorter test duration to provide a durability assessment for the design process of new products.

The steady acceleration of product development cycles and the use of polymeric materials in more demanding applications has caused an urgent need to predict the oxidation resistance of building materials over the whole service life in a reasonable time.

### 4 Acknowledgements

Autoclaves are available on the market for money but their use and the evaluation of the measurements need detailed background knowledge of the physical and chemical processes. This knowledge has been exchanged between the scientists of the Federal Institute for Materials Research and Testing (BAM) and the engineers of the tBU. Special thanks for this fruitful cooperation and the granted support to us go to Berlin.



Autoclaves in use