

ALEKSANDER KOZAK¹ULTRAVIOLET TEST CHAMBER – APPLICATION IN
SIMULATED WEATHERING AND ARTIFICIAL AGING
TESTSKOMORA STARZENIOWA Z LAMPAMI ŚWIATŁA
ULTRAFIOLETOWEGO – ZASTOSOWANIE W TESTACH
SYMULOWANEGO I PRZYSPIESZONEGO STARZENIA

Abstract

The paper deals with description of the UV test chamber used in artificial aging tests. The apparatus has been designed and built in Institute of Building Materials and Structures at Cracow University of Technology. It allows to control the weathering cycles, temperature and spraying intensity with distilled water. Moreover, the example of the application of the chamber described for epoxy coating tests used to protect concrete against carbonation has been presented.

Keywords: weathering chamber, UV lamp, weathering, protective coatings, coating degradation

Streszczenie

W artykule przedstawiono opis komory z lampami UV używanej w badaniach przyspieszonego starzenia. Aparatura została wykonana w Instytucie L-1 Politechniki Krakowskiej. Pozwala ona kontrolować cykle starzeniowe, temperaturę oraz intensywność zraszania wodą destylowaną. Ponadto przedstawiono przykład wykorzystania opisanej komory do badań epoksydowej powłoki stosowanej do ochrony betonu przed karbonatyzacją.

Słowa kluczowe: komora starzeniowa, lampa UV, proces starzenia, powłoki ochronne, degradacja powłok

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1. Introduction

Natural weathering of polymeric materials refers to the exposure of polymers to natural outdoor conditions under which these materials undergo degradation. There are different environmental factors influencing durability of polymeric materials. These factors include solar radiation, moisture, temperature, oxygen, ozone, chemical pollutants (sulphur and nitrogen oxides) and microorganisms. These factors also influence other components of the polymeric materials such as dyes, pigments, stabilizers etc. Weathering is a complex process, since many factors usually act interactively. That is why we can observe synergistic effect among different elements. Moreover, the action of these factors exhibit some variations even within one day. Some information on above elements is presented below.

Solar energy is the main factor, which contributes to the degradation of polymeric materials. The total solar energy, which reaches the earth's surface consists of three regions:

- a. Infrared (2500 nm ÷ 780 nm) accounts for about 42÷60% of the total spectral radiation. The energy level is not high enough to initiate photochemical reactions but can accelerate some chemical reactions.
- b. Visible light (780 nm ÷ 380 nm) is around 39÷53% of the total radiation. It can influence some photochemical reactions.
- c. Ultraviolet (380 nm ÷ 295 nm) is no more than 5% of the total spectrum, nevertheless it is mainly responsible for the photochemical reactions occurring in polymers. It significantly influences durability of organic materials. The region has the highest energy level in the solar radiation and the energy is high enough to cause cleavage of chemical bonds present in polymers. Thus, it causes damage to polymeric materials.

Water is present in the outdoor environment in different forms such as humidity, fog, dew, rain, snow, hail etc. It can cause hydrolysis of polymers and is also responsible for freeze-thaw effect, which leads to formation of physical stresses and then material degradation.

The elevated temperature influences the rate of chemical reactions and usually high temperatures are associated with the high radiation level. Hence, degradation of the polymeric materials induced by solar radiation (photochemical reactions) is accelerated by the elevated temperature. It is worth noting that the same temperature can differently influence materials depending on the colour of samples. Sometimes the temperature gradients can be as high as 30°C. Moreover, the elevated temperature has high impact on hydrolysis and oxidation of the polymeric materials as well as the increase in the rate of water and oxygen diffusion into the material. Nevertheless, even the highest measured temperatures are not high enough to cause the cleavage of the chemical bonds.

Oxygen and ozone also play important role in the degradation of the polymeric materials. Oxygen takes place in many photochemical reactions and low-concentration ozone influences fast degradation of the organic materials, since the materials are exposed to higher UV radiation. On the other hand, elevated concentration of ozone has an effect on oxidation of polymers.

Some pollutants such as sulphur oxides and nitrogen oxides lead to degradation of the materials. The compounds contribute to the formation of acid rains, which results in reactions among polymeric materials and aggressive chemicals.

Mechanical factors are usually connected with the action of thermal stresses, wind, hail etc.

The influence of the microorganisms on the durability of polymeric materials is also important. Some bacteria and fungi may feed on polymeric materials and treat them as a substrate to grow. Presence of carbonyl groups is beneficial to bacteria, because it is the source of carbon. Moreover, the microorganisms can increase permeability of liquids and gases in polymers.

Weathering of the polymers leads to degradation of these materials and then to reduction of their service life. Depending on the conditions under which the materials are used, the rate of the process can vary considerably. In many cases, some materials even after one year after depositing can lose desired properties. The properties include both mechanical ones such as tensile strength, elasticity, adherence as well as barrier ones such as diffusion coefficient D , diffusion resistance R and equivalent air layer thickness R_{air} that is equal to diffusion resistance of coating. Weathering particularly influences thin polymeric protective coatings [1-5]. In order to explain the changes in the structure of the coatings FTIR spectroscopy has been used.

2. Weathering chambers

Simulating outdoor conditions is usually time consuming that is why accelerated laboratory tests are commonly used to check if any material is durable under environmental conditions or not. It is of great importance to achieve good correlation between artificial weathering and natural one. A very important factor is designing the weathering cycles that should reflect degradation mechanism similar to that occurring under natural conditions. We should simulate natural processes in the weathering chambers keeping in mind that weathering processes are synergistic ones. It is very difficult to ensure the same conditions, especially in one chamber, which occur in natural environment. The chambers can only simulate the natural processes to some extent. In artificial testing climatic factors are intensified in order to rapidly simulate natural weathering. Nevertheless, the cycles designed to imitate natural weathering should reflect them as much as possible. We can control different conditions and lead to faster degradation of polymeric materials by means of the chambers. The chambers are usually equipped with three types of lamps: Xenon, UVA and UVB ones. Xenon lamps reflects solar radiation most accurately, but since UV is mainly responsible for damage to polymers, UV lamps are widely used. Moreover, xenon lamps are much more expensive than UV ones. In many tests UVB-313 lamps are used, but because they utilize short-wave UV, the mechanism of weathering can be different from that taking place under natural conditions. Fig. 1 presents the comparison between solar radiation and UVA-340 lamps [6].

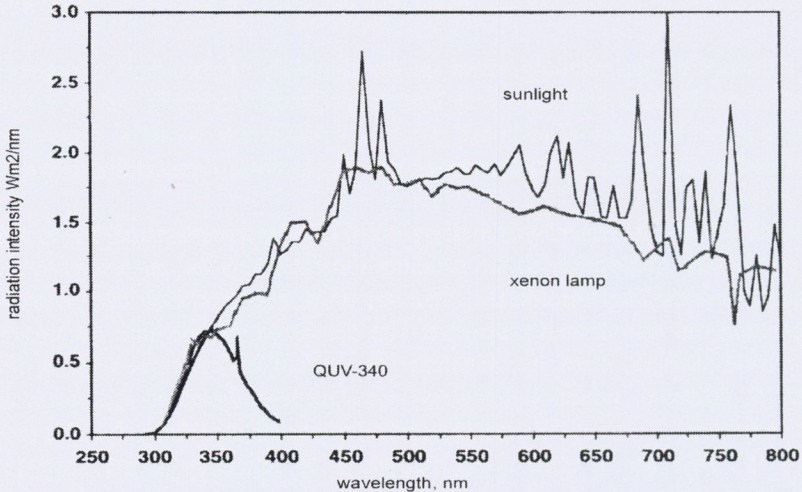


Fig. 1. Comparison of spectrum of sunlight with spectra of xenon and UV lamps

Rys. 1. Porównanie widma światła słonecznego z widmem lampy ksenonowej i UV

The chambers used for accelerated weathering are usually equipped with UVA lamps, a spraying system and a system to control temperature. The chambers are very useful for coatings applied on metal substrate, whose thickness is very small, since the distance between specimens and the lamps is 5 cm. The problem occurs when we would like to test the coatings applied on concrete. Because of such limitations and high cost of purchase and maintenance, the author of the article has decided to design and build the chamber which would be appropriate for the samples mentioned above. The lamps used in the chamber have spectra that are nearly the same as the spectra of UVA-340 light source and give a total radiation intensity similar to that of UVA-340 lamps. The radiation intensity has the value similar to that measured in sunny day during summer in Cracow and the value is $40 \pm 2 \text{ W/m}^2$. The radiation intensity is measured by means of Solarmeter. Moreover, the chamber allows to control the temperature and spraying samples with distilled water. In order to control and choose different weathering cycles, special software has been written. The constant temperature in the chamber is ensured by application of four digital sensors. The average temperature is calculated as mean value out of these sensors and the value is then chosen to control the temperature in the chamber. In order to ensure constant temperature for all samples, air conditioning is applied. The air is taken from the chamber and transferred to the heater outside the chamber, heated and then comes back to the chamber. The system is closed. By means of the software, one can also download parameters of the cycles during and/or after weathering. Currently the software is being updated in order to have a possibility to control the chamber cordlessly from a computer located far from the room in which the chamber is located. The pictures of the radiometer and an interior of the chamber are presented below.

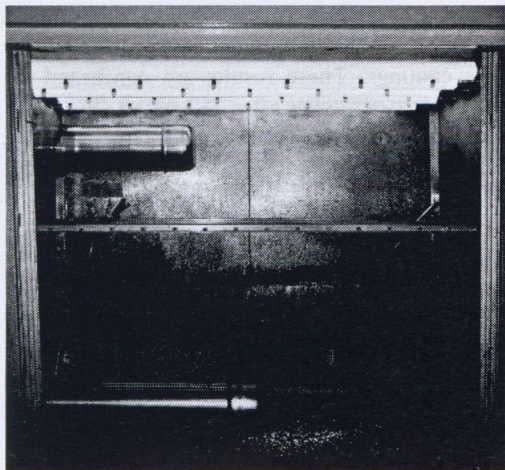


Fig.2. Weathering chamber

Rys.2. Komora starzeniowa

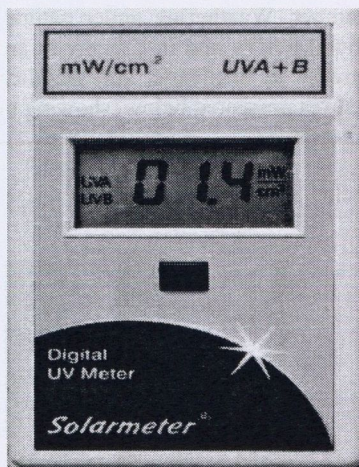


Fig.3. Radiometer Solartech

Rys. 3. Radiometr firmy Solartech

3. Experimental part

A plasticized epoxy coating (EP) was tested. The coat was weathered according to ASTM G154a standard for 252 cycles as well as under natural weathering in Cracow and in Tatranska Strba (Slovakia), which is located near High Tatras and exposed to a high level of UV. The choice of two different places was made in order to compare the influence of the high UV radiation in Slovakia with an effect of a municipal atmosphere on durability of the coating. One weathering cycle in the chamber consisted of 6h of irradiating by UV lamps at $60 \pm 3^\circ\text{C}$, and then 2 h of sprinkling with distilled water at $40 \pm 3^\circ\text{C}$. The time of the accelerated weathering was 2160 h (3 months). Radiation range was between 295 do 400 nm. The value of the radiation was $40 \pm 2 \text{ W/m}^2$ and it was also the highest radiation intensity of UV measured during summer in Cracow in 2006.

The coatings tested were either deposited on mineral substrate (concrete) or studied in the form of foils. The following diagnostic features were determined: adherence to concrete, changes in elongation and tensile strength. Adherence to concrete was checked by means of Dyna 715. The adherence to concrete before and after weathering of the coat was in the range from 5.0 to 6.5MPa, i.e. much above requirements ($>1 \text{ MPa}$) [7]. No changes in the adherence of coatings tested were observed during both natural and artificial changes. Studies of changes in elongation of foils of coatings as a result of weathering were done using ZWICK 1445 universal machine. Fig. 4 presents test results (mean values).

Test results showed that weathering influenced the ultimate elongation of the samples, which have been subjected to long UV exposure. EP coatings are known not to be resistant to UV radiation. Test results of tensile strength showed that samples exposed to the action of artificial weathering exhibited very low values of the strength. They dropped more than 70 times from around 14 MPa to around 0.2 MPa even after 43 cycles of weathering in the

chamber. Weathering under natural conditions in Slovakia led to decrease in strength to around 6 MPa and in Cracow to around 8 MPa, respectively. Aging caused the significant increase in brittleness and fragility of the coatings. These results are confirmed by the pictures presented below, showing numerous micro-scratches after 252 cycles (Fig. 5).

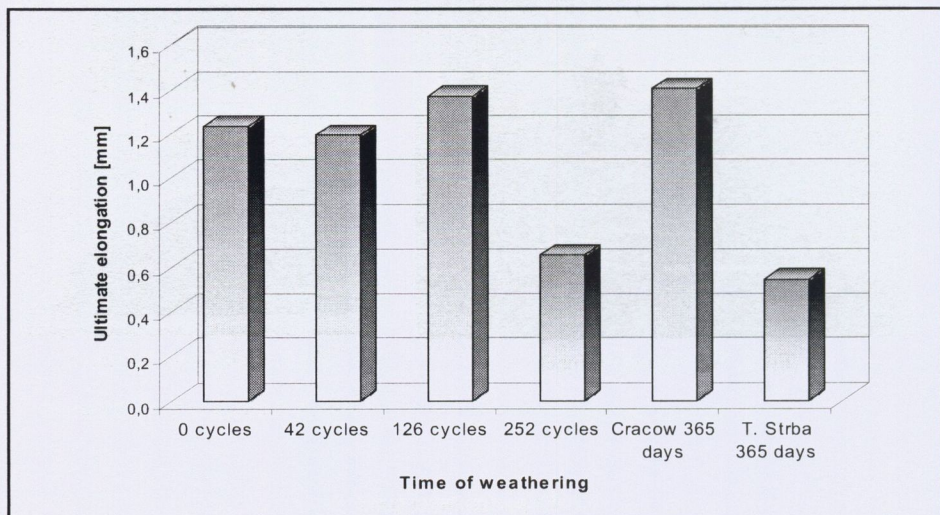


Fig. 4. Ultimate elongation of EP coating before and after weathering

Rys. 4. Wydłużenie powłoki EP przed i po starzeniu

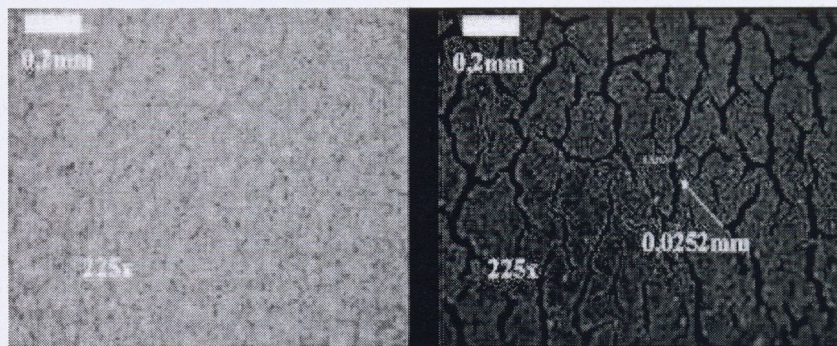


Fig. 5. a) Picture of initial EP coating, b) Picture of EP coating after 252 cycles

Rys. 5. a) Zdjęcie wyjściowej powłoki b) Zdjęcie powłoki EP po 252 cyklach

FTIR measurements were performed using BIO-RAD FTS 175C device. Spectra of samples were recorded using KBr pastille technique. Initial spectra were compared with spectra of specimens after weathering and then, comparing characteristic functional groups

of given coating, an attempt was made to explain changes in the structure of the coats. Analyzing spectra of the EP coatings one can observe broadening of band $\sim 1700\text{ cm}^{-1}$, which is characteristic for carbonyl groups. Oxidation process leads to the formation of additional carbonyl groups. Moreover, broadening the region may be prescribed to -C=N groups if hardener of the coating was amine. Fig. 6 presents spectra of initial samples and specimens after weathering.

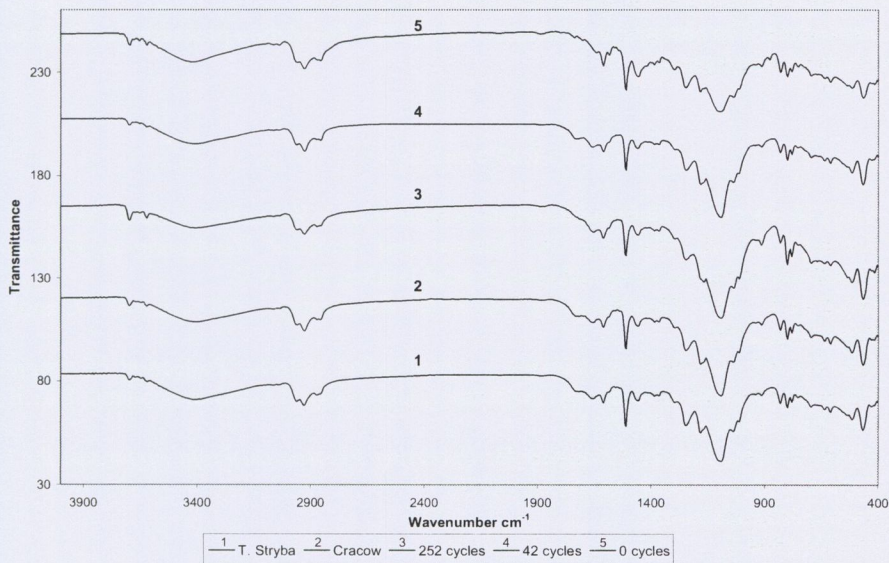


Fig. 6 Spectra of EP coating before and after weathering.

Rys. 6. Widma powłoki EP przed i po starzeniu

4. Conclusions

On the basis of the test results obtained the following conclusions have been drawn:

1. Natural weathering leads to degradation of polymeric materials. Different environmental factors can influence the durability of these materials. Of these factors, solar radiation is the main weathering factor.
2. In order to predict service life of polymeric coatings exposed to natural weathering chambers for artificial weathering are commonly used. The weathering cycles and application of the light sources should reflect natural aging as much as possible, i.e. the mechanism of the weathering caused by the chamber ought to be similar to that induced by natural weathering.

3. The designed and built chamber for artificial weathering at Faculty of Civil Engineering successfully performed weathering tests according to ASTM G154a standard. Currently the software of the chamber is being upgraded to control it cordlessly.
4. Weathering of EP coatings proved that the material is not resistant to the action of UV light, hence the application of the material on concrete as protective layer is questionable. Aging of the coating, especially artificial weathering, led to dramatic decrease in tensile strength and increase in stiffness. The reason was a formation of numerous micro-cracks on the surface of the samples observed by means of microscopic observations. The changes were induced as a result of oxidation processes and formation of extra carbonyl groups.

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