**Ozone · Dynamic rubber test · Woehler · Ageing test · Ozone climate simulation**

Aging factor in life is very important to measure, by doing this we can evaluate the life of materials before time. For example in cable, rubber, tire and aerospace industry we can't judge the actual service life of the rubber products. We need a time machine which could see in future to calculate its serviceable life. You can do this by Anseros Ozone Testing Systems, meets ISO, DIN and ASTM standards.

## Dynamische Gummiprüfung im Ozonklima

**Ozon · Dynamische Gummiprüfung · Wöhler · Alterungstest · Ozonklimasimulation**

Die Langzeitbeständigkeit von Gummi und Elastomerprodukten ist nicht nur ein Qualitätsanspruch, sondern mehr noch ein Sicherheitsfaktor geworden. Insbesondere verlangt die Mobilität Zuverlässigkeit für Kabel, Reifen, Dichtungen, Schläuche und Isolatoren, um Leben nicht zu gefährden. Mit Prüfgeräten zur Alterungssimulation lassen sich Vorhersagen treffen. Anseros Ozonsimulatoren entsprechen einschlägigen Testmethoden nach DIN, ISO, ASTM und bieten Innovationen, um die Sicherheit von Materialien weit zu verbessern.

# Dynamic Rubber Test by Ozone

Ageing of rubber products is caused by temperature, humidity, mechanical stress, and UV-light, but to a major part by the concentration of ozone in the atmosphere. Rubber goods like tires, cable insulation, O-rings, and hoses are manufactured using elastomers containing olefinic double bonds that are sensitive to ozone. Especially under strain cracks can occur. This process is accelerated under dynamic operation. In practice this can cause the bursting of a tire, ignition due to the loss of insulation of a cable, or the cracking of a petrol hose. Different standards like DIN, ISO, VDE, SAE, and ASTM describe static and dynamic methods for the investigation of the behaviour of rubber samples towards ozone. Usually, rubber samples are stretched and ozone is applied at defined temperature, humidity, and air velocity.
Anseros is a company with a focus on the application of ozone, providing a wide range of ozone test chambers from a small test unit (SIM pocket) to large units (SIM 8000) with space for testing several tires at the same time. Here, the latest developments are presented (e.g. Dr. Rohrbach [1]).

## Ozone Climate Chamber

Anseros ozone climate chamber SIM 7300 consist of an ozone generator (free of corrosion), ozone analyser (Patent DE 41 19346), and climate chamber. Ozone concentrations from 0.25 ppm to 400 ppm (0.5 mg/Nm³ to 800 mg/Nm³) can be realized. The climate chamber of 300 L exhibits temperature control in a range from 15 °C to 70 °C, humidity control from 10 % up to 95 % and air exchange providing > 0.6 m/s. A software is processing the tests. For the mentioned international standards special programs are already installed to facilitate the operation of testing. New programs can be easily added by the customer and applied. Consequently this chamber can be used to carry out all tests according to the above mentioned standards. Figure 1 shows the latest ozone climate simulation chamber SIM 7300.
The ozone climate chambers are used for quality tests of rubber products, hoses, and cables. New mixtures using waxes and antioxidants can be tested to increase the lifetime of rubber products. Special tools are offered to simplify the testing.

## Testing tools

The ozone climate chambers can be equipped with different tools. Stretching units allow to adjust the strain of barbell rubber samples at different percentages. They can be hang into rotating frames to improve the uniformity of the ozone exposition respectively. The latest development of Anseros is a dynamic rotating disc (Patent [2]). Up to 16 barbell or band samples can be clamped into a disc. This disk is rotating and stretches the samples 3 times per round at an adjustable stretch. Consequently, dynamic investigations can be carried out. Figure 2 shows a picture of the dynamic rotating disc.
The installation of a digital camera can provide auto-control of the testing. Current work is focusing on the digital recording and time dependent evaluation by Anseros Amacs software. The data is processed and depicted in the Woehler diagram. Figure 3 shows a crack in a rubber sample recorded by a digital camera.
The Woehler diagram depicts the stretch as percent over the period of strain as hours. Samples are put at different stretch into the test chambers under the required conditions. The time interval between "no observation of cracking" and "first observation of cracking" is drawn as a line into the diagram for all the samples. Thus, the area of the diagram can be divided into two parts: An area where cracks can be observed and an area without any cracks. Fatigue resistance is achieved when the resulting line of division has achieved a horizontal development. Figure 4 presents an example of the Woehler graph.
A dynamometer can be used inside the ozone test chamber to carry out torsion experiments. Figure 5 shows the set up.

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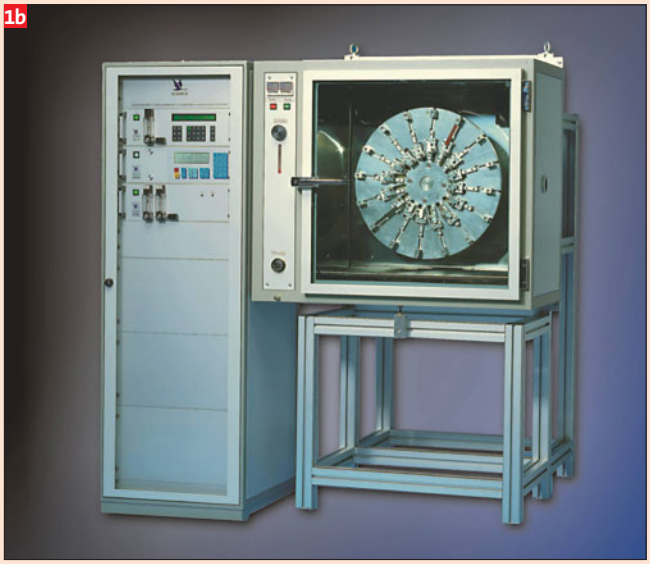
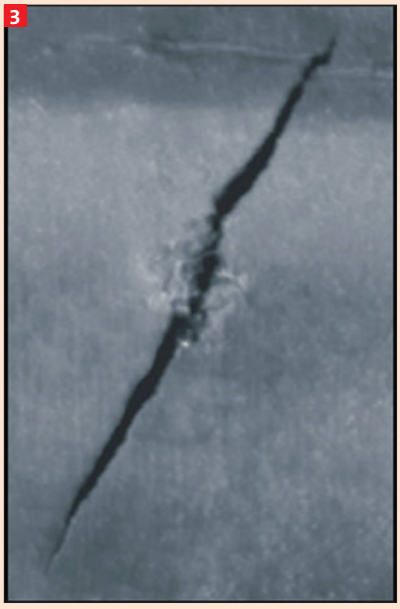
1a Ozone climate simulation chamber SIM 7500

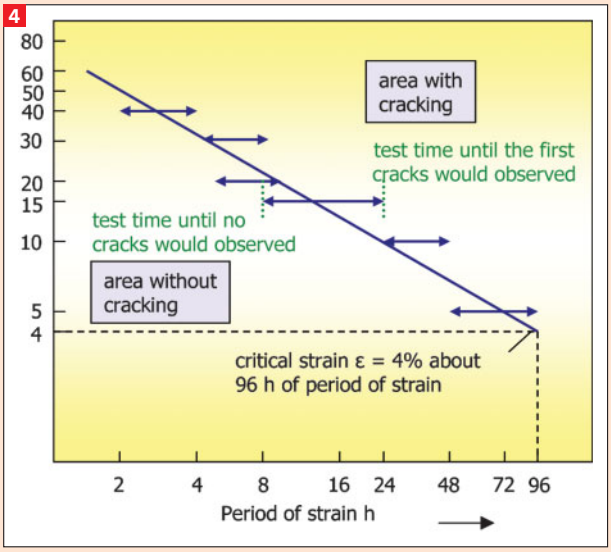


1b Ozone climate simulation chamber SIM 6300

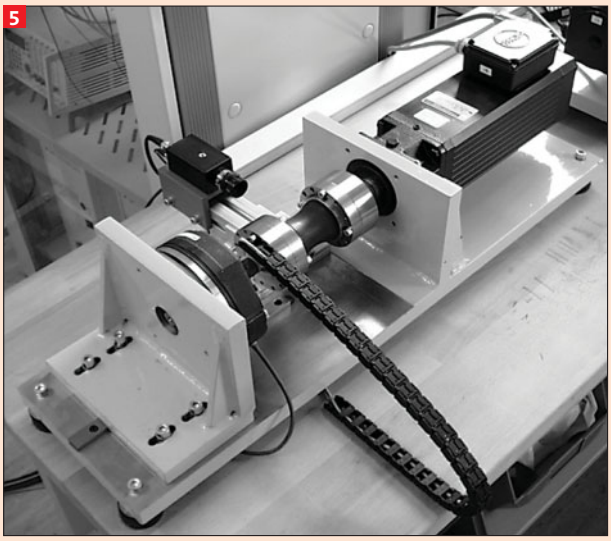
2 Dynamic disk for continuous stretching and release of 16 samples.



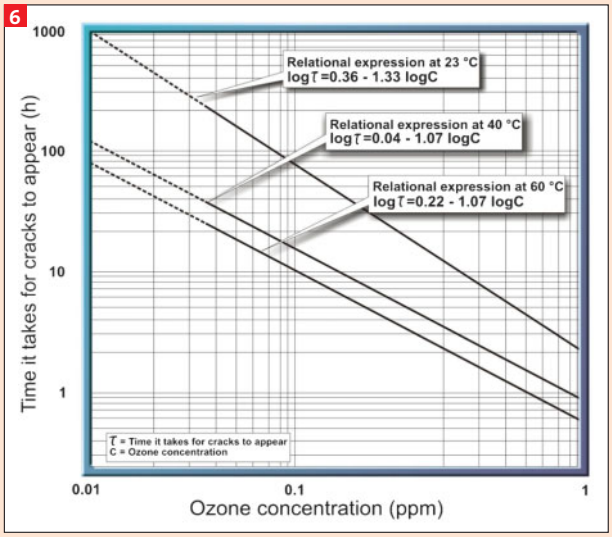
3 Photo of the crack on a rubber sample




4 Woehler diagram [3]



5 Dynamometer for torsion experiments




**6 Ozone concentration and the time it takes for ozone to generate cracks(deterioration) on rubber [5]**

## Ozone attack on rubber in relation with temperature

In most cases, there is a clear-cut relationship between ozone concentration (C) and the time it takes for ozone to generate cracks ($\tau$)*.

$$\tau \cdot C^n = \text{Const.} \quad \log\tau = K - n\log C$$

- : Time it takes for ozone to generate cracks (h) [4]

C : Ozone concentration (ppm)

n : Constant that varies depending on the rubber material

K : Constant

Linear relationships were obtained when plotting the logarithms of the experiments SIM conducted on ozone concentration and the time it takes for ozone to generate cracks. What became evident is that as ozone concentration increases, the time it takes for ozone to generate cracks decreases. These tests were conducted under conditions that are much more severe than those of a normal operating environment, and were likewise evaluated more stringently.

The solid lines in the graph indicate actual measurements gathered during the tests, and the dotted lines are estimated values obtained from calculations. Changes in ozone concentration greatly affect the time it takes for ozone to generate cracks (Fig. 6).

Ozone concentration and its direct relation to the appearance of cracks (time factor) (NBR)

<Test conditions> Specimen: I-shaped dumbbell, Tensile strain: 20 ± 2 % Evaluation: Cracking detection using a magnified 50 x metallurgical microscope

## Basic structure and ozone resistance

### *Molecular structure*

Ozone resistance of NBR and HNBR varies depending on its structure S1.

### *Double bond*

Deterioration caused by ozone occurs when ozone acts on the NBR double bond to break the rubber molecules apart S2.

On the other hand, HNBR's basic structure, which is becoming more and more standard, eliminates the double bond of NBR by adding hydrogen. Although a minute amount of double bond remains in the structure of HNBR to allow it to maintain its physicality as a rubber, its structure has an excellent ozone resistance S3.

### *Ozone resistance*

An experiment was performed under test conditions conforming to ISO 1431, and using air containing 1 ppm of ozone. The test conditions (ozone concentration, temperature, and tensile strain) were much more severe than those in a normal operating environment; therefore, cracks were evident in NBR in just 1 to 25 hours of exposure to ozonic air. On the other hand, when the same stringent conditions were applied to HNBR, cracks did not appear even after 1000 hours of exposure S4.

<Test conditions>Ozone concentration: 1 ppm, Specimen: I-shaped dumbbell, Tensile strain: 20 +2 %, Testing temperature: 40 ± 1 °C

## Conclusions

Ozone has a high impact on rubber ageing and fatigue. Therefore rubber products have to be tested in an ozone atmosphere. Anseros provides ozone test chambers that provide a safe control of rubber goods using static and dynamic strain tests. Requirements of all international standards can be fulfilled. Special tools simplify the investigations.

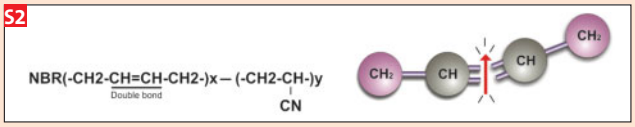
## Refernces

[1] Dr. G. Rohrbach, Anseros GmbH, Poster presentation at DKT 2006 Nuremberg, Germany.
[2] Patent DE 100 24 490 (2002), Erf. Klaus Nonnenmacher.
[3] DIN 53509.
[4] YU. S Zuev and S. I. Pravednikova "Rubber Chemistry and Technology" (1962) Page 411 to 420.
[5] JIS K6259: Testing of Resistance to Ozone Cracking for Vulcanized Rubber.

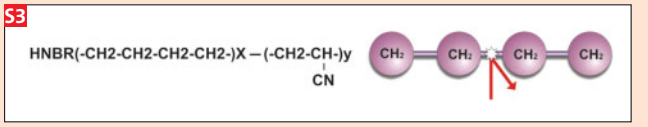
S1 Basic molecular structure

| Rubber Type | Basic molecular structure |
|---|---|
| NBR (acrylonitrile-butadiene rubber) | (-CH2-CH=CH-CH2-)x – (-CH2-CH-)y, CN |
| HNBR (hydrogenated nitrile butadiene rubber) | (-CH2-CH2-CH2-CH2-)X – (-CH2-CH-)y, CN |

S2




S3




S4 Ozone Resistance

| Rubber type | Time it takes for ozone to generate cracks |
|---|---|
| NBR | 1 to 25hrs. |
| HNBR | 1000hrs. or more |