Polymer Testing 15 (1996) 381-395

0142-9418(95)00043-7
0142-9418/96/$15.00

Recent Developments in Heat Ageing Tests and Equipment

Göran Spetz

Elastocon AB, Göteborgsvägen 99, S-502 60, Borås, Sweden

(Received 21 September 1995; accepted 24 October 1995)

ABSTRACT

The types of oven in use in the rubber industry for heat ageing tests are reviewed, The results of interlaboratory tests are given which show the poor reproducibility obtained in ageing trials. The factors influencing heat ageing results are investigated in detail using conventional and new designs of oven. The new ovens are described, indicating the steps which were taken to minimise sources of error and hence poor reproducibility.

1 BACKGROUND

Heat ageing tests have a long history in the testing of polymers, especially rubber polymers. This can be seen by the early number of the IS0 standard for the heat ageing of rubber – IS0 188. It seems that the importance to specify the ageing conditions to get a reliable test result was understood very early.

2 TYPES OF AGEING OVENS USED

Three types of equipment have been used for the ageing of rubber; cell ovens, cabinet ovens and oxygen or air bombs. The ageing of polymers in oxygen bombs at high pressure will not be discussed, as with this type of ageing it is not possible to make any correlation with natural ageing.

Based on a paper presented at Polymer Testing 95, Shawbury, England.

2.1 Cell ovens

Cell ovens have been used for a long time for the ageing of rubber. The idea is to achieve a steady temperature by using a large block of aluminium and to separate the samples by using one cell for each material.

The first type of cell oven had a large aluminium block with a number of holes in the block. In the holes were glass test tubes placed for the samples. To get an exchange of air, two small glass tubes were placed in the cork of the test tube. One of the glass tubes went down to the bottom of the test tube and the other glass tube ended just below the cork. The idea was to achieve an air circulation caused by the temperature gradient in the test tube. No one has, to my knowledge, however been able to measure the air exchange rate in such an oven, even if attempts have been made.

A later type of cell oven had larger cells in the aluminium block and forced air exchange by using an air pump with a flow meter. This type of oven was a great improvement over the first type. The preheating of the air was however not always satisfactory, as the air in the cells could be up to two degrees Celcius lower than the temperature of the aluminium block.

2.2 Cabinet ovens

Until a few years ago, there were not any cabinet ovens designed especially for the ageing of polymers available on the market. As an alternative, the laboratories purchased standard drying ovens. The standard ovens on the market can be divided in two categories: with or without a fan. The ovens without a fan normally have very large variations of the temperature in space: up to 10°C is not unusual, especially when the throttle is open to get an air exchange. This type of oven is often used by the cable industry, as the standard they are using, IEC 811, states that no fan is allowed in the oven. To get an acceptable temperature tolerance of ± 1 °C the ovens are measured to find a usable area with this tolerance. Normally an area of a few litres is found in the middle of the ovens where the samples can be placed.

The ovens with a fan are normally used in the rubber industry, as companies have been more concerned with a uniform temperature than a low air speed, which is required in IS0 188. The detrimental effect on the test result of a high air speed has also not been known until recently, see Ref. 1 (paper presented in Polymer Testing 1994).

3 REPRODUCIBILITY IN HEAT AGEING TESTS

At the beginning of the 1980s it was decided to include within IS0 TC 45, Rubber and Rubber Products, a precision clause in all testing method stan-

TABLE 1
Ageing test reproduceability

12 different labs, ITP 1988	*Mean*	*s*	*R*
Change in tensile strength (%)	-18	5.3	15
Change in elongation at break (%)	-40	5.8	16
Change in micro hardness, IRHD	-13	3.8	10

s = standard deviation.
R = reproduceability in actual units of measurements.

dards. The precision clauses were established by carrying out interlaboratory test programmes, ITPs, to establish the repeatability (within a laboratory) and the reproducibility (between laboratories) for the test methods.

This work inspired us in Sweden to start an interlaboratory test programme, organised by the Swedish National Testing Institute. During the years 1982-1988, 14 interlaboratory tests were carried out. For two of the methods a retest was done. Up to 25 laboratories participated in these interlaboratory tests.

All these interlaboratory tests within IS0 and in Sweden have shown that the spread in the test results is worse than anyone could have expected. Table 1 shows the result from an ITP for heat ageing done in Sweden in 1988

In another project, four rubber compounds, NR, SBR, NBR and EPDM, were aged for 168 h at 100°C in four ovens. Before and after the ageing, micro hardness and tensile tests were performed. The results are shown in Table 2 and compared with the result from the Swedish ITP in 1988 in Table 1.

When comparing the above results with the Swedish ITP in 1988, we can see that the ovens contribute to most of the variations in the ageing tests. It must be noted that before the test, the temperatures of the ovens were adjusted to an actual temperature of 100°C in the centre of each oven. So most of these differences are caused by factors other than temperature.

TABLE 2
Oven influence, micro IRHD and tensile test

4 different ovens	*Mean*	*s*	*R*
Change in tensile strength (%)	-20	5	15
Change in elongation at break (%)	-42	4	11
Change in micro hardness, IRHD	-3.5	1.9	5.8

4 INVESTIGATION OF FACTORS INFLUENCING HEAT AGEING TEST RESULTS

The problem of obtaining reproducible test results when doing heat ageing tests have been shown in several interlaboratory test programmes (ITP) carried out in Sweden and within ISO TC 45 during the last ten years. To improve the situation a project within the Swedish Plastic and Rubber Institute, has studied the different factors influencing ageing results.

The project started with an inventory of the ovens used for ageing tests among the twelve participating Swedish rubber companies. Based on this inventory, four of the ovens and a new designed oven were chosen for a closer study. The ovens were investigated for the following:

1 temperature uniformity in time;
2 temperature uniformity in space;
3 set, shown and actual temperature;
4 air speeds;
5 air exchange rates;
6 ageing results in different ovens.

The following factors influencing the ageing were then further investigated in different ways:

1 temperature;
2 air speed.

4.1 Measurements of the ageing ovens

Based on the inventory the following ovens were measured in one laboratory:

1 Heraeus UT 5042;
2 Heraeus UT 5060E;
3 Salvis TSW 60;
4 Elastocon EB 01;
5 Elastocon EB 04.

4.2 Temperature variations in time

A PT 100 sensor was placed in the centre of each oven. The sensors were connected to a data logging system connected to a PC-computer, with a resolution of ±0,1°C. The temperature was adjusted as close as possible to 100°C. The ovens were run for 5 days to see temperature variations in time. The results are shown in Table 3.

The UT 5042 oven had a mechanical controller, the other ovens had elec-

TABLE 3
Temperature variations in time

5042 (°C)	5060E (°C)	TSW 60 (°C)	EB 01 (°C)	EB 04 (°C)
13.8	0.1	0.2	0.1	0.1

tronic controllers. With a modem electronic controller temperature variations in time seems to be no problem.

4.3 Temperature variations in space

A frame with five PT 100 sensors located in each comer and in the centre, connected to a data logging system, was placed inside each cabinet and was moved between three positions. The outer sensors were placed about 50 mm from the walls. The results are shown in Table 4 and Figs 1 to 3.

4.4 Air speeds

The air speeds have been measured in 27 points in the ovens and the results are shown in Table 5.

The air speeds of the ovens EB 01 and EB 04 are dependent of the air exchange rate only and have not been possible to measure. The value is calculated from the air exchange rate.

The traditional heating cabinets show great variations in air speed from almost 0 up to 5 m/s. The flow is rotating and turbulent. The air movement is created by a fan behind a baffle, either in the back or on one side of the cabinet.

TABLE 4
Temperature variations in space

Location	5042 (°C)	5060E (°C)	TSW 60 (°C)	EB 01 (°C)	EB 04 (°C)
Inner	0.9	0.5	1.3	NA	0.4
Centre	0.7	1.7	1.3	NA	0.3
Outer	0.7	1.1	2.7	NA	0.2
Total	1.2	1.7	3.1	0.5	0.4

NA = not applicable.
The table shows the difference between five points in each location and the total difference (all points all locations).

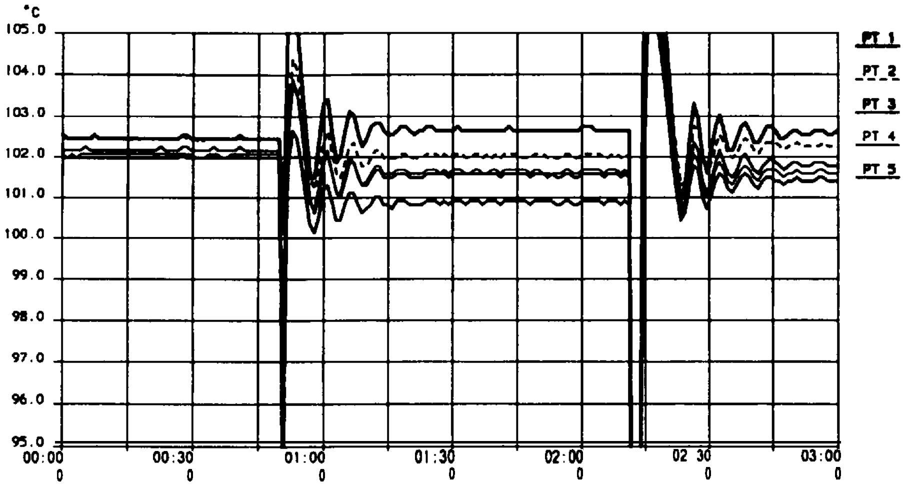

Fig. 1. Temperature variations in space, UT 5060E.

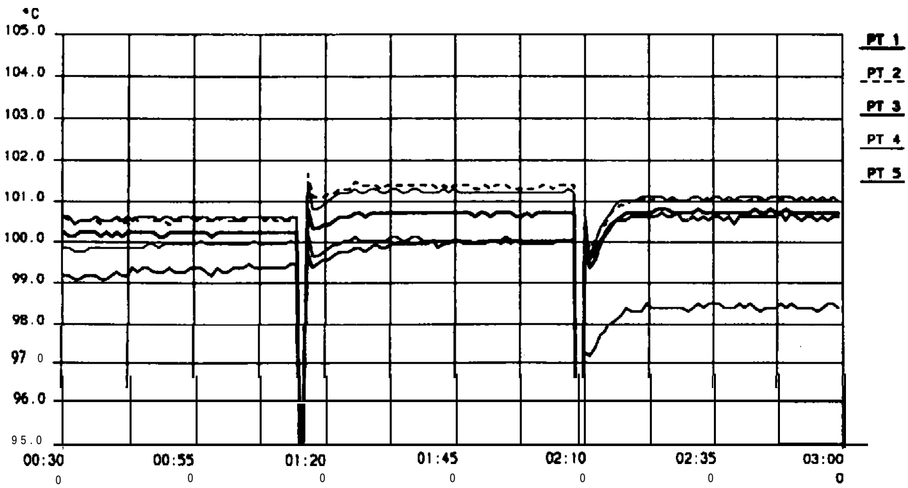

Fig. 2. Temperature variations in space, TSW 60.

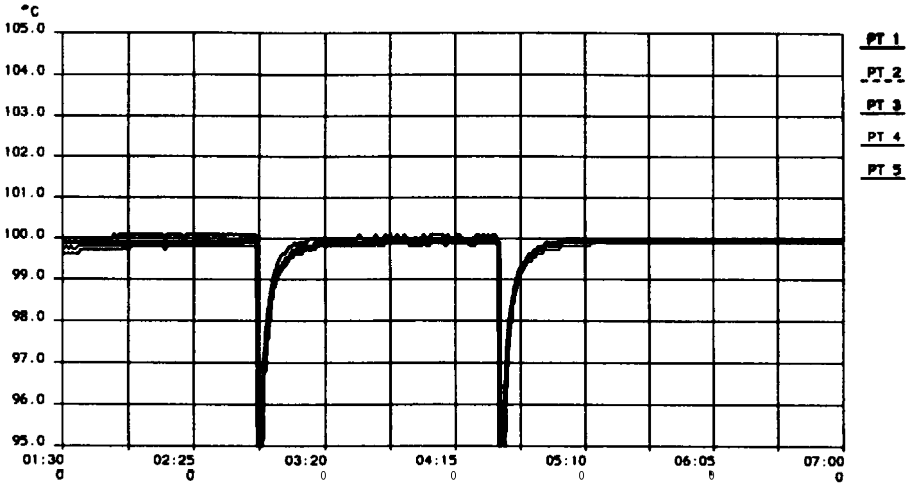

Fig. 3. Temperature variations in space, EB 04.

TABLE 5
Air speeds

Speed	*5042 (m/s)*	*5060E (m/s)*	*TSW 60 (m/s)*	*EB 01 (m/s)*	*EB 04* ep*(m/s)*
Minimum speed	0.5	0.0	*0.4*	<0.001	<0.001
Maximum speed	*2.6*	4.5	3.0	<0.001	<0.001

In EB 01 and EB 04 the air is passed through a flowmeter with a control valve, going from the bottom to the top of the cabinet more as a laminar flow.

In IS0 188 rubber accelerated ageing test, no exact air speed is required, but the standard states that 'provision shall be made for a slow circulation of air through the oven of not less than three and not more than ten changes per hour.' This may be interpreted as no fans are allowed inside the testing cabinet and this means then that the three standard ovens tested do not meet the requirements for low air speed.

4.5 Air exchange rates

The air exchange rates have been determined both by calculating the air volume by measuring the air speed and area in the exhaust hole and by measuring the time needed to fill a 125 1 plastic bag attached to the exhaust hole. The results are shown in Table 6.

The air exchange rate in EB 01 and EB 04 is set by a flow meter and a control valve.

In IS0 188 rubber accelerated ageing test, the requirement for air exchange is 3-10 exchanges/h, while the IEC 811 requires 8-20 exchanges/h.

4.6 Long term ageing, two ovens

One rubber compound of a NBR/PVC blend was aged in two of the ovens for 1000 h at 100°C. One Salvis TSW 60 and one Elastocon EB 01 cell oven were used. The micro hardness and tensile test were performed after 72, 336 and 1000 h. The temperature in the ovens were adjusted to be 100°C in the centre of each oven. The results are shown in Fig. 4.

The results in the figure show that ageing does not follow the same reaction in the two ovens. The surface of the tested rubber was aged much more in the heating cabinet than in the cell oven. The main difference between the ovens were the air speed. The micro hardness test is mainly measuring surface effects. The tensile test however did not show the same big differences. The air speed seems to be of greater importance for heat ageing than is presently recognized.

4.7 Temperature influence on ageing

Four rubber compounds, NR, SBR, NBR and EPDM, were aged at three temperatures, 95, 100 and 105°C, in a cell oven for 168 h. Before and after the ageing, micro hardness and tensile test were performed. The results for NR are shown in Figs 5 and 6 and the results for EPDM are shown in Figs 7 and 8.

The results shows a clear influence of the temperature, even in the limited

TABLE 6
Air exchange rates

Oven	5042	*5060E*	*TSW 60*	*EB 01*	*EB 04*
Exhaust			Air changes/h		
Open	-160	-40	-300	20	16
Closed	0	0	-20	0	0

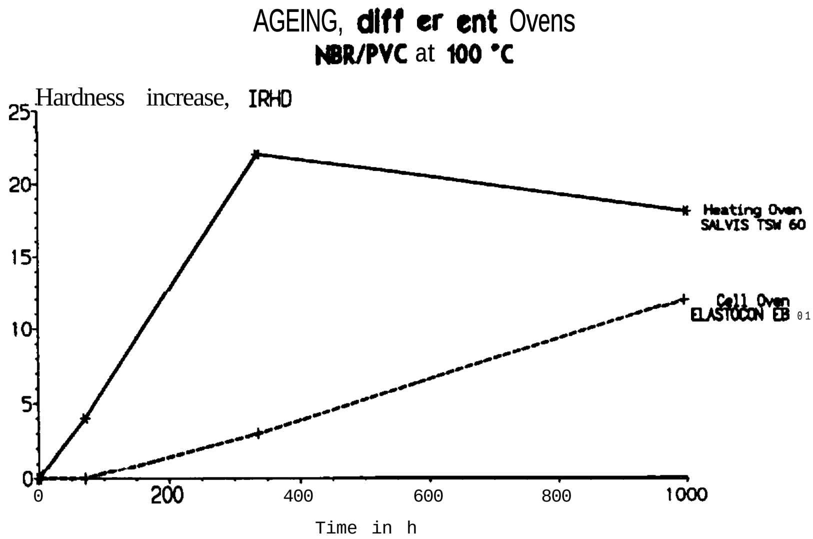

Fig. 4. Long term ageing, different ovens, micro IRHD.

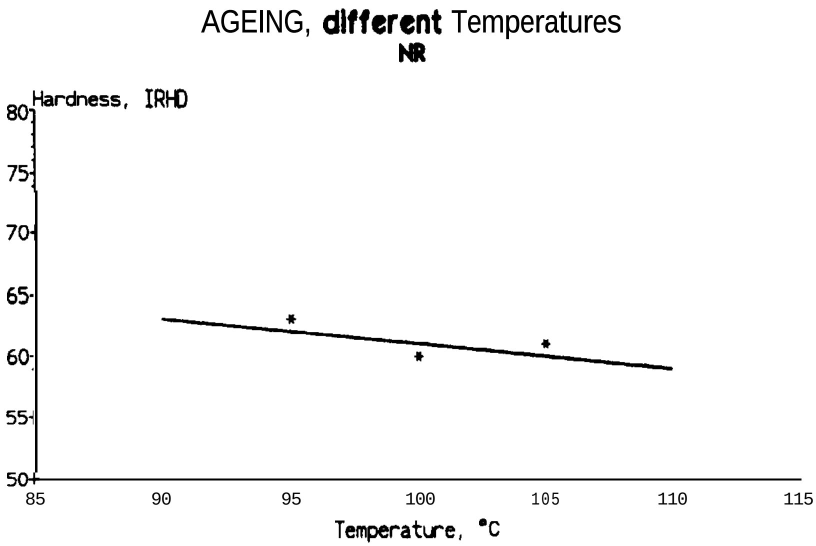

Fig. 5. NR, hardness.

range of ±5°C. The NR compound shows a dramatic reduction in properties in the temperature range of 100°C and the temperature seems to be too high for ageing of NR. To keep the temperature variations within close limits seems to be an important factor in reducing variations in ageing of polymers.

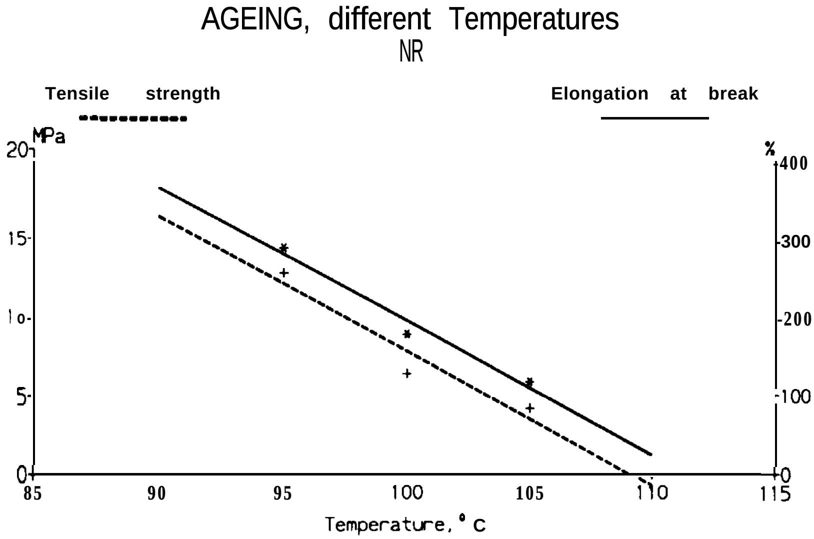

Fig. 6. NR, **tensile test.**

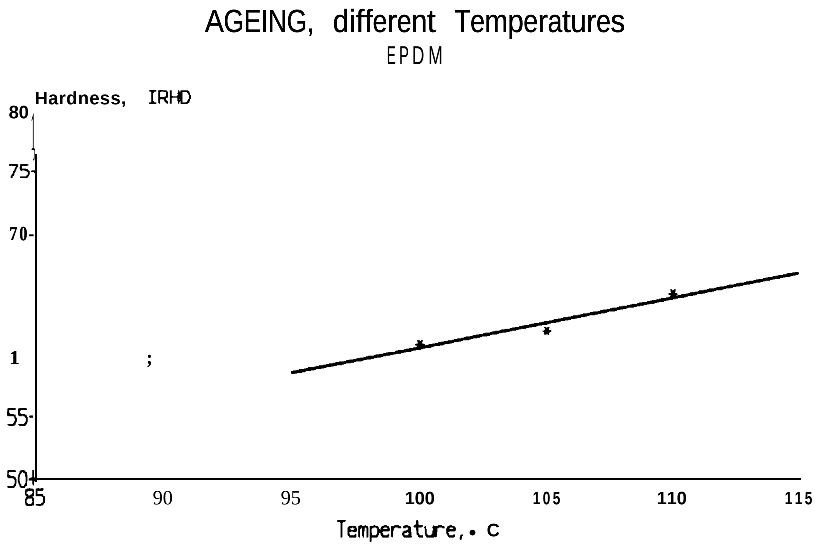

Fig. 7. EPDM, hardness.

4.8 Air speed influence on ageing

To make a closer investigation of the influence of air speed on heat ageing results, two special ovens were developed. One oven with an air speed of about 0.3 m/s and one with about 3 m/s. Four rubber materials, NR, SBR, NBR and EPDM, were then aged at 70°C (NR, SBR) and 100°C (NBR, EPDM) for

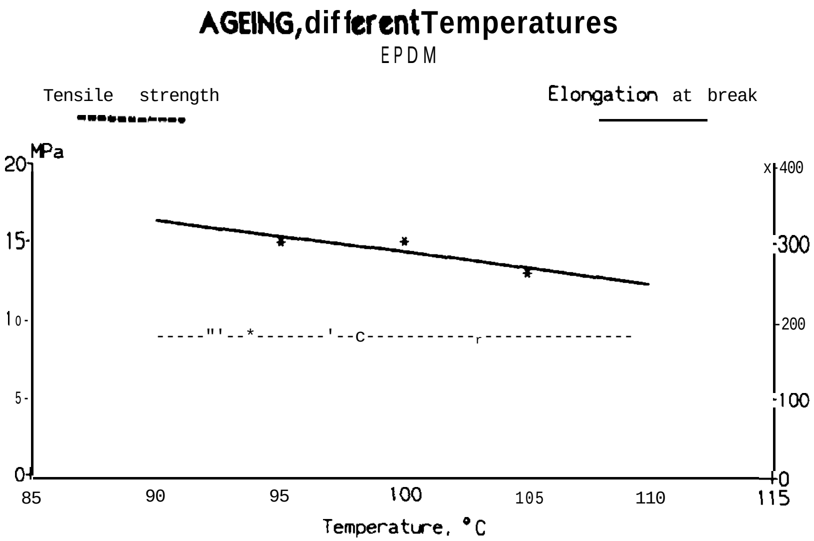

Fig. 8. EPDM, tensile test.

1000 h in these ovens plus in a cell oven with an air speed of about 0.001 m/s. Weight loss, micro hardness and tensile tests were performed on the four materials after 1, 3 and 6 weeks of ageing. Some of the results are shown in Figs 9 and 10.

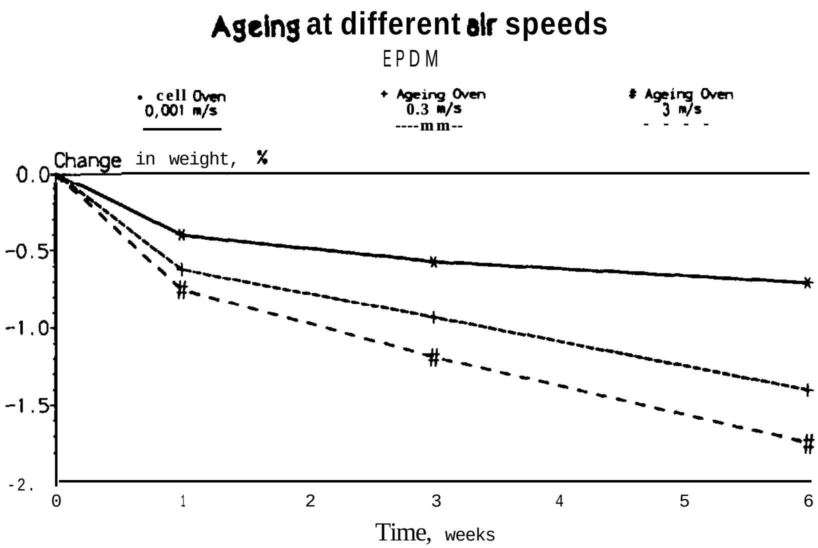

Fig. 9. EPDM, weight loss at different air speeds.

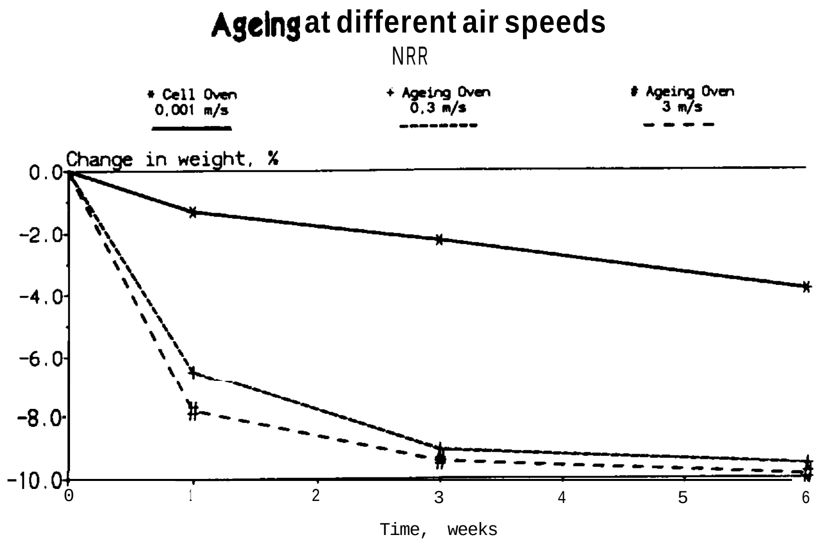

Fig. 10. NBR, weight loss at different air speeds.

4.9 Summary

This project has shown that the main factors contributing to poor reproduceability between laboratories when doing heat ageing tests are air speed and temperature. The results have been presented to IS0 TC 45 and have been taken into consideration during the present revision of IS0 188 'rubber vulcanized and thermoelastic heat ageing tests'. Sweden has suggested that the new version of IS0 188 shall include more clearly defined testing conditions for the ageing test especially regarding air speed. Besides the ageing in still air Sweden has suggested the addition of an ageing test at high air speed (1 m/s), for the study of air speed influence.

5 DESIGN OF NEW OVENS FOR HEAT AGEING TESTS

Elastocon has during the last 8 years been involved in the development and design of new ageing ovens for achieving better reproducability and repeatability when doing heat ageing tests of polymers. The work has been carried out in parallel with the research project reported above. We have developed a series of both cell ovens and cabinet ovens for the ageing of polymers.

5.1 Cell ageing ovens

Elastocon designed the first cell oven EB 01, 8 years ago, a 4 cell oven with large cells and a diameter of 100 mm. The oven is now improved and the third generation is on the market, see Fig. 11.

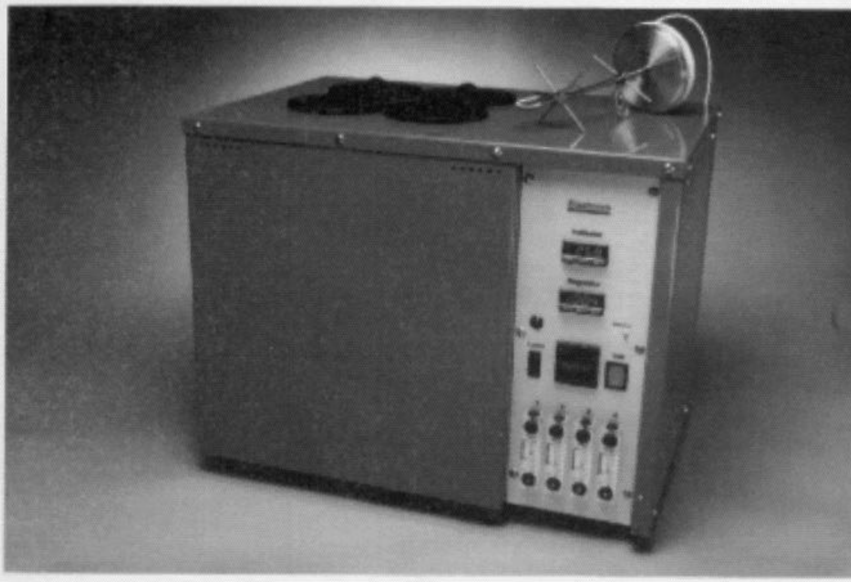

Fig. 11. Cell oven EB 01 with four cells.

The temperature system in the oven uses two digital PID controllers with 0.1°C resolution. One of the controllers is used to control the temperature in the aluminium block. The other controller is used to measure and display the air temperature in one of the cells, close to the samples. The temperature alarm system is also connected to this controller. The temperature variation in time and space can be kept to half of what is required in the ISO 188 heat ageing standard, or ±0.5 up to 100°C and ±1 up to 200°C.

The exchange of air is made using an air pump, which pumps the filtered air through a separate flow meter into the bottom of each cell. Before the air enters the cell, it is preheated to the test temperature by passing through channels in the aluminium block. The air pressure is monitored by a piezoelectric pressure sensor, connected to the alarm system. The air exchange rate can be set with the flowmeter to 3–20 exchanges/h. The air speed is dependent of the air exchange rate only and is calculated to be below <0.001 m/s.

Two years ago Elastocon made the first triple temperature cell oven, EB 07. The triple oven has the same specification as the EB 01 cell oven, but the temperature can be set individually in each cell. This allows for very flexible use of the oven, see Fig. 12.

5.2 Cabinet ageing ovens

Three years ago Elastocon presented its first cabinet oven for ageing with the same specification as the cell oven, see Fig. 13.

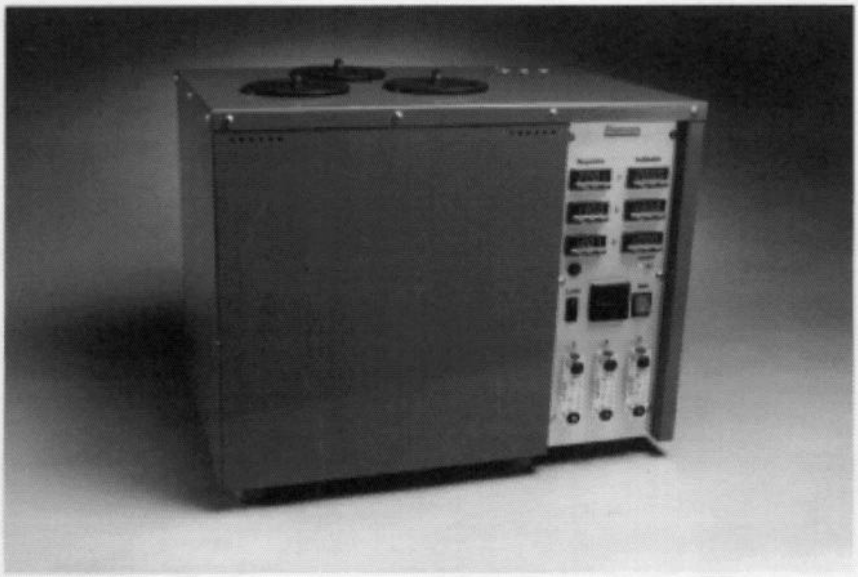

Fig. 12. Triple temperature cell oven EB 07.

Fig. 13. Cabinet ageing oven EB 04.

The basic temperature system is the same as in the cell ovens, but to achieve a uniform temperature in space is more difficult in a cabinet oven. The normal way of achieving a uniform temperature in space in a cabinet oven is to use a fan and have a high air speed stirring the air. A high air speed is however not suitable for ageing tests and the only way to get a uniform temperature in still air, is to have all sides of the cabinet at the same temperature. In the cell oven this is achieved by keeping the aluminium block at correct temperature. In the cabinet oven we get the same results using an inner chamber and having a fan blowing the air with high air speed around the inner chamber.

To exchange the air, fresh air is pumped through a flowmeter into the air stream around the inner chamber, close to the heating elements. The preheated air enters the testing space though a distribution chamber in the bottom of the inner chamber and moves slowly (<0.001 m/s) up to the top, where it is evacuated through a small chimney.

We are also, at the moment, designing a budget priced version of our cabinet ageing oven. To reduce the price we exclude the air pump, flowmeter and window. To still get an air exchange rate within the specification, we use the fan to get fresh air into the system and we set the air exchange rate by adjusting a throttle in the chimney.

REFERENCES

1. Spetz, G., Improving Precision of Rubber Heat Test Methods. Part 2: Ageing. *Polymer Testing,* 13 (3) (1994) 239-270.