# *"PREDICTING THE LIFE OF TPE SEALS IN SEVERE SERVICE ENVIRONMENTS"*

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## BACKGROUND

Historically, use of thermoplastic elastomers in severe service environments has been limited by their inherent intrinsic properties; the "softer" grades of most TPE's possess neither high temperature nor oil/solvent resistance to displace the TSE's that have dominated severe service applications for many years. With the development of newer thermoplastic materials and changing perceptions, however, it is evident that some TPE's, e.g., TPV's and co-polyesters, are finding expanding markets in these environments as the press toward TPE's with improved heat resistant/solvent resistant attributes continues to result in more resins in the marketplace which are performance-competitive with TSE's.

One of the TSE mainstays over time has been automotive engine compartment applications, particularly oil and transmission seals -- applications "off limits" for conventional TPE's. Increasing underhood temperatures have driven the upper end of the required performance temperature window to 175°C for certain elastomeric components. TPE's do continue to make inroads into several underhood applications, usually where spec requirements are typically a maximum of only 125°C. At the same time, seals in contact with various fluids at these service temperatures further eliminate a number of TPE's from consideration for use as gaskets, O-rings and fluid-component sealing applications. Since both temperature and fluid exposure in combination represent particularly difficult challenges for TPE materials in sealing applications, it becomes increasingly important to be able to predict performance of new resins in these combination environments if one expects to be able to predict the serviceability of a new or modified resin.

As the industry pursues the manufacture of these improved resins, appropriate performance evaluation tools continue to evolve as well. Historical single-point material property characterization testing, e.g., stress-strain, hardness and flexural properties, is useful for routine quality control/SPC, but is really less useful when attempting to predict the expected life of a part, particularly in severe service. The advent of computer modeling and Finite Element Analysis as applied to thermoplastic parts is particularly useful in identifying localized stress concentration areas, but is less useful when complicating and transient environmental variables are introduced. For more detailed discussion of a complete life prediction methodology as used at the Materials Engineering Research Laboratory from a rubber engineering point of view see Reference #1 pp. 204-206; the work addresses fracture mechanics, stress analysis, thermal analysis and diffusion analysis in detail as well.

For several years now, manufacturers of high performance TSE's, e.g., Dow/STI and GE as well as automotive manufacturers, most notably Ford and GM, have been investigating the use of compression stress-relaxation testing as a means of predicting service life of TSE seals in simulated service environments (Ref. 2 - 5). While most of this test work has emphasized higher service temperatures (150° and 175°C), there has also developed an interest by other TSE and TPE polymer manufacturers, notably Shell, AES and Zeon in testing for compression stress-relaxation at reduced temperatures (70° - 125°C).

At the same time, SAE under the auspices of the SAE CARS subcommittee on Long Term Aging, has recently issued SAE J2236 which defines the continuous upper temperature limit for elastomeric materials based on long-term aging performance (1008 hours) (Ref. 6).

Also forthcoming from Ford and GM are spec performance standards for automotive seals based on compression stress-relaxation testing, which will most likely replace existing compression set test requirements. ISO/BS compression stress-relaxation standards have been in existence for some time Ref. 7 - 9.

## DEFINING "SEVERITY"

The term "severe service" as applied to elastomeric materials is commonly associated with hostile temperatures and chemical environments, either singly or more often in combination. For the manufacturers of TPE components for automotive underhood applications, the term "severity" varies considerably, as can be readily appreciated, depending not only on the environmental variables but on the TPE polymer itself. In the family of all TPE's, severity varies considerably. Consequently, service life prediction for an individual TPE should be made only within near-reasonable temperatures and environments.

## THE CLASSIC ARRHENIUS APPROACH -- ACCELERATED AGING

In 1990, Rapra Technology's R.P. Brown (Ref. 10) conducted a survey on the status of test methods for accelerated durability testing of polymers. Input from three hundred and fifty companies worldwide was solicited. Rapra concluded in part that, ".... normally single-point (tests) are really only effective as QA procedures." and that, "For thermal effects the only recognised procedure is Arrhenius."

The classic Arrhenius equation,

$$(d \ln k)/dt = -(E/T^2)$$

can be modified to: $\log k = (E/2.303R)\ (1/T) + C$

where
- $k$ = specific activation rate
- $E$ = activation energy for the reaction
- $R$ = gas constant per gram molecular weight
- $T$ = absolute temperature in degrees Kelvin
- $C$ = mathematical constant

A straight line is produced when the log of a specific reaction rate is plotted against the reciprocal of absolute temperature since the above equation is of the form $y = mx + b$. It has been empirically demonstrated that many reactions double or treble their rates for every 10°C. As a result of both empirical observation and the above linearity, if one carefully chooses a meaningful property that relates to service life, it should be possible to predict performance at extrapolated temperatures based on observed results at several actual temperatures. This technique has been used successfully for many years when dealing with thermoset elastomers in severe environments. Two particularly good presentations on this methodology may be found in References 11 - 12.

## LIFE PREDICTION FOR SEALS

The automotive industry has focused on % retained sealing force as a function of time as being the multi-point parameter for useful service life prediction: when sealing force has decayed to 25% of the original sealing force, the seal is considered to have failed.

The stress decay of polymer components under constant compressive strain is known as compression stress-relaxation. The test measures the sealing force exerted by a seal or o-ring under compression between two plates (Fig. 1). It provides definitive information for prediction of the service life of materials by measuring the sealing force decay of a sample as a function of time, temperature and environment.

The ARDL test apparatus used for the compression relaxation measurements is the ISO 3384 Wykeham Farrance device. The device measures precisely the counterforce exerted by a specimen maintained at constant strain between two stainless steel plates inside the compression jig over a period of time. The decay in force is then plotted against time to generate the stress-relaxation curve.

The instrument has a variety of jigs for accommodating test pieces of o-rings up to 100 mm O.D. Various service environments, liquid, gas or a mixture of liquid and gas can be introduced into the stainless steel compression jig and maintained during aging and testing. A typical cross sectional view of the compression jig is shown in Figure 2.

A typical sealing force decay graph of a Thermoplastic gasket is shown in Figure 3. These curves were obtained at 25% constant compressive strain at three accelerated aging temperatures (70°C, 85°C and 100°C). In this example, the specimens are aged until an arbitrary "Failure Point" retained sealing force is reached, i.e., when the sealing force decays to 25% of the original unaged sealing force. Sealing force readings may be found in Fig. 4.

Figure 5 is the Arrhenius service life plot from data obtained from the 3 decay plots from Figure 3. The abscissa is the reciprocal of the absolute temperature, but for convenience, the equivalent Celsius temperature is shown.

Compare the single point compression set data on the two TPE materials (Fig. 6) with the multi point compression stress-relaxation curves (Fig. 7, 8 and 9). Note that TPE #2 does not totally degrade at 100°C, but that TPE #1 does. This information is valuable for both TPE manufacturer as well as end user and partially explains OE's preference for the stress-relaxation data.

Besides elevated temperature testing, the environment during compression stress-relaxation can also be varied to obtain data at low temperatures and/or in corrosive, oxidative or fluid environments. An example of multi-media predicted service life curves from a recent TSE study is shown in Figure 10.

Compression stress-relaxation testing is now underway at ARDL on a variety of elastomeric seals in several "severe" environments for 1008 hours aging. Results of this program will be given in Denver at the ACS Rubber Division meeting in May.

## UTILITY AND APPLICABILITY OF PREDICTIVE TESTING

Data to date has shown two particular benefits from a TPE perspective:

1. Behavioral characteristics of thermoplastics under constant compressive strain;
2. The concept of maximum service temperature for TPE seals.

The thermoplastic behavior short term is consistent with compression set results obtained after short duration; thereafter the retained sealing force "plateaus" in contrast to TSE materials which show progressive deterioration.

As expected, TPE's are more temperature sensitive than TSE materials, with a tendency to degrade beyond certain temperature limits. While this is a characteristic of thermoplastic material, it can be used to establish the continuous upper temperature for a given material application, particularly when tested in the proper media.

## SUMMARY

Compression stress-relaxation is the current testing methodology being used to assess performance and predict the service life of thermoset elastomeric seals in severe environments, and will most likely replace the single-point compression set test on material specifications.

As thermoplastic elastomers are developed for more severe service applications, this testing methodology can be used to predict service life for TPE seals. It can also be used for comparative testing against TPE controls or other TSE's already being used in a particular application.

The life prediction methodology is soundly based on continuous multi-point stress-relaxation coupled with classical Arrhenius aging. Screening TPE materials utilizing this approach yields insight into long term performance in severe environments.

## REFERENCES


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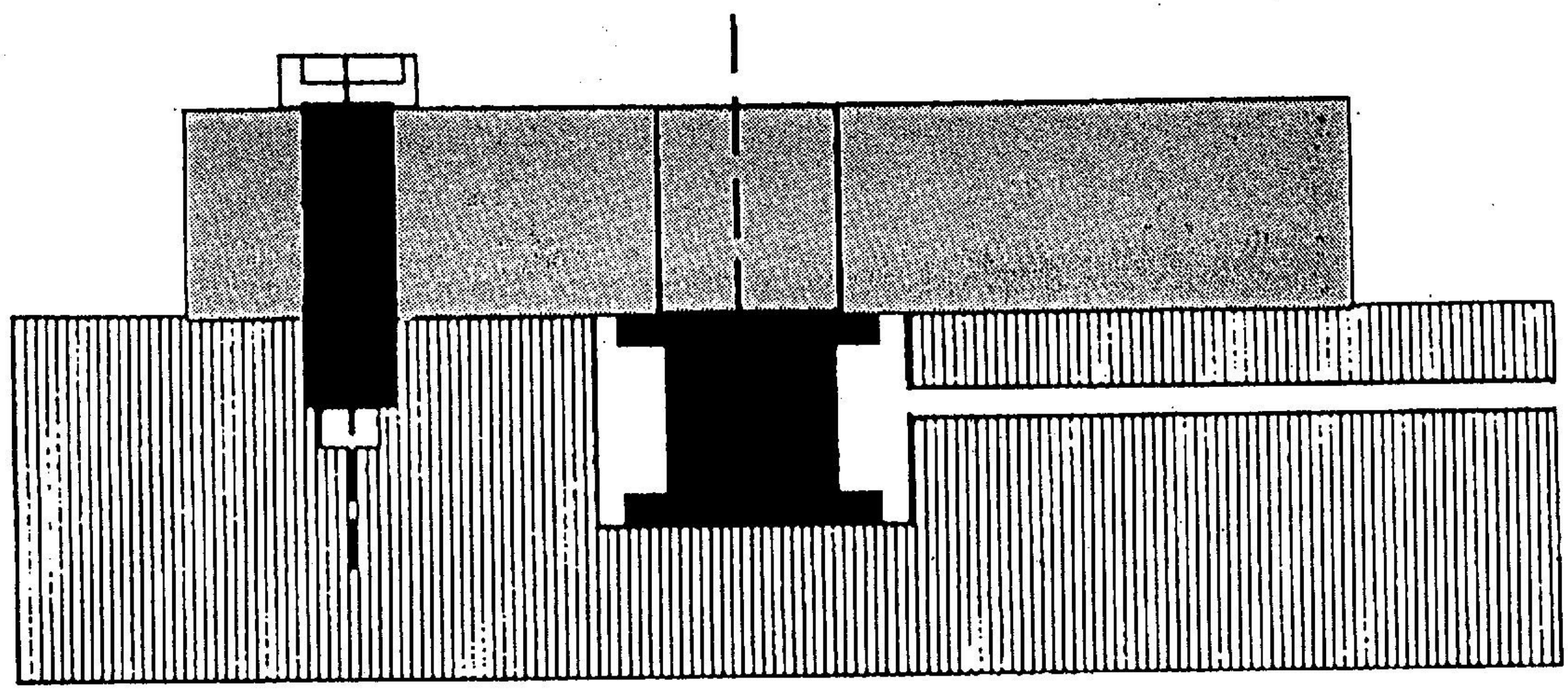


Figure 1

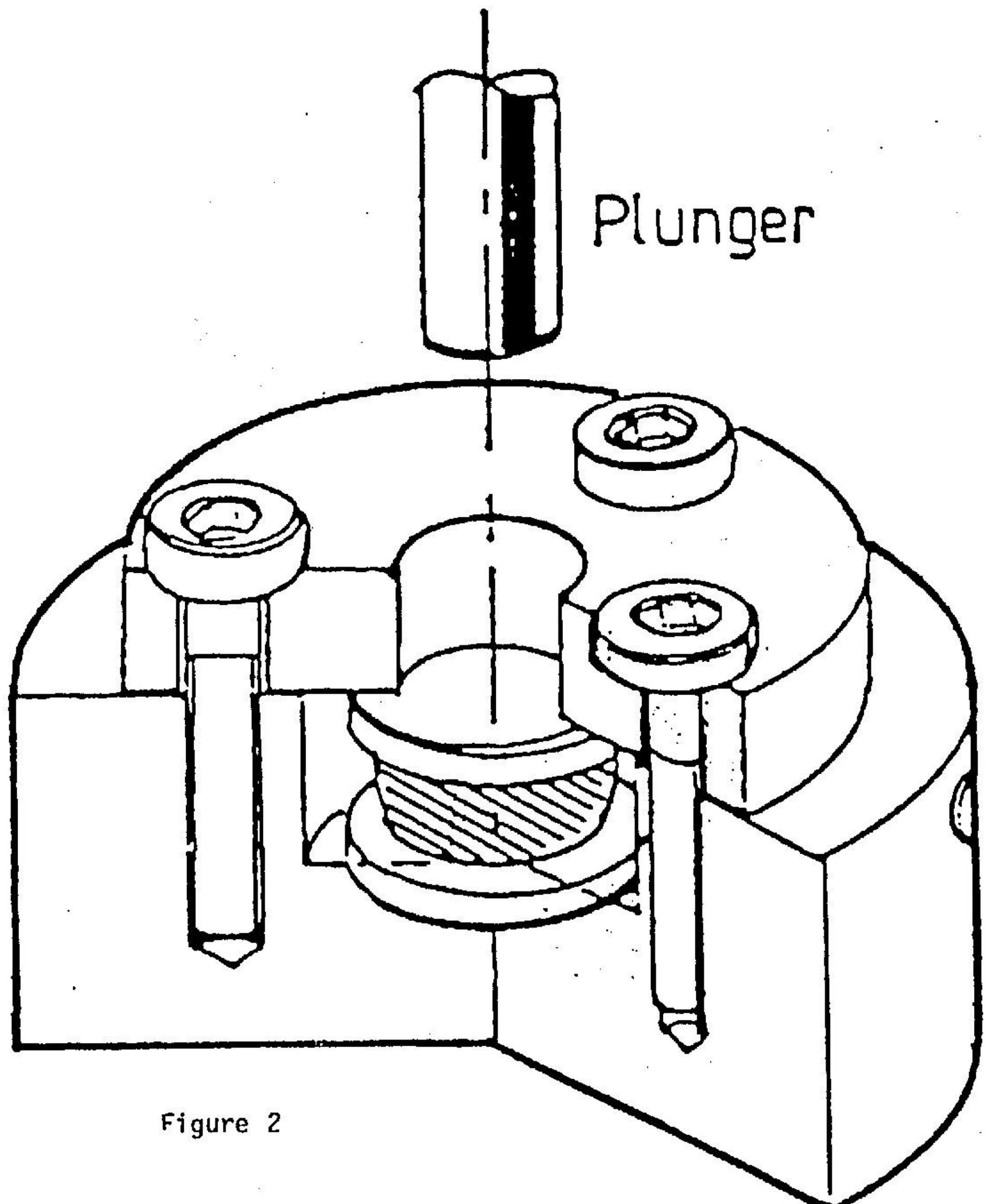



Figure 2

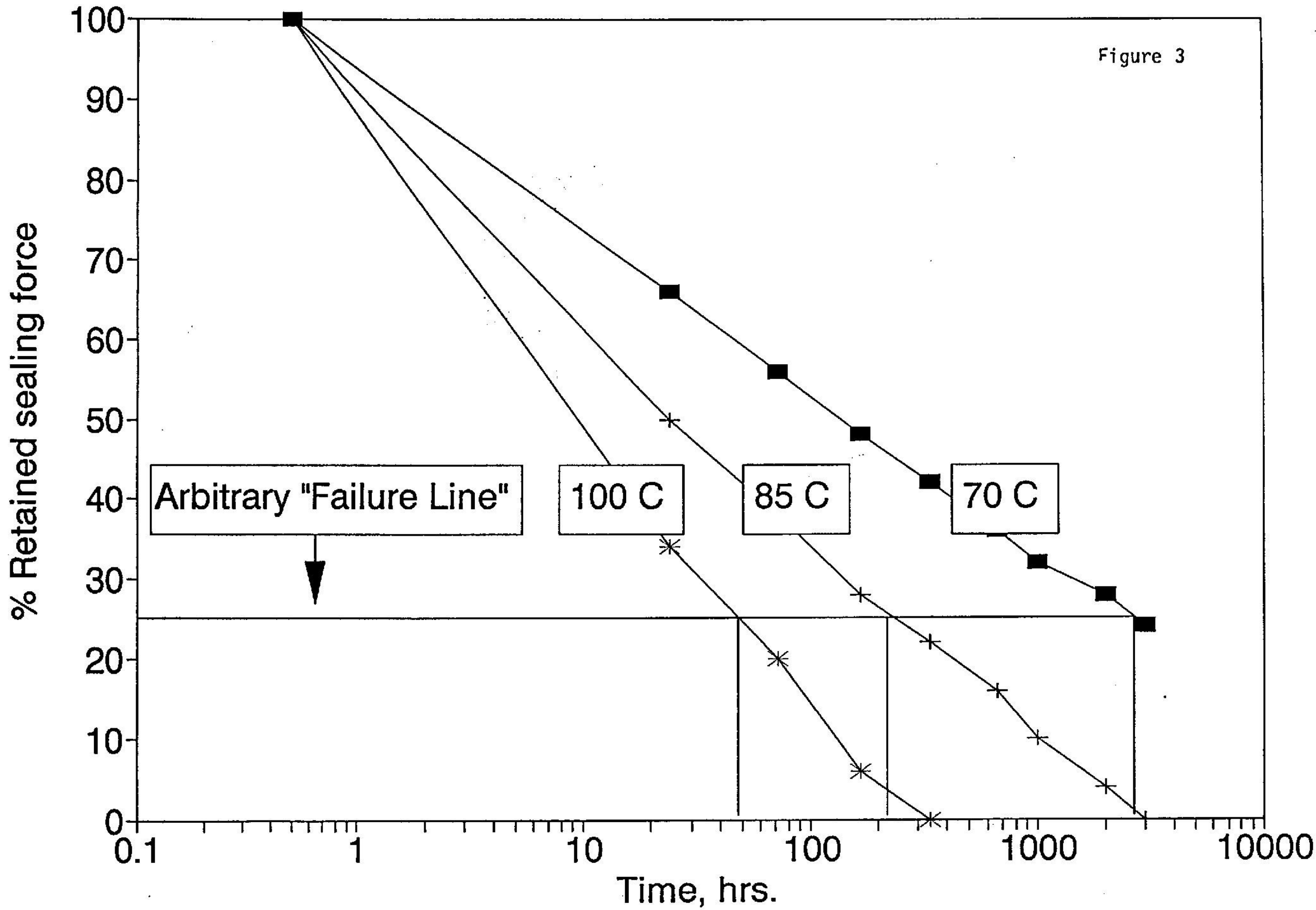
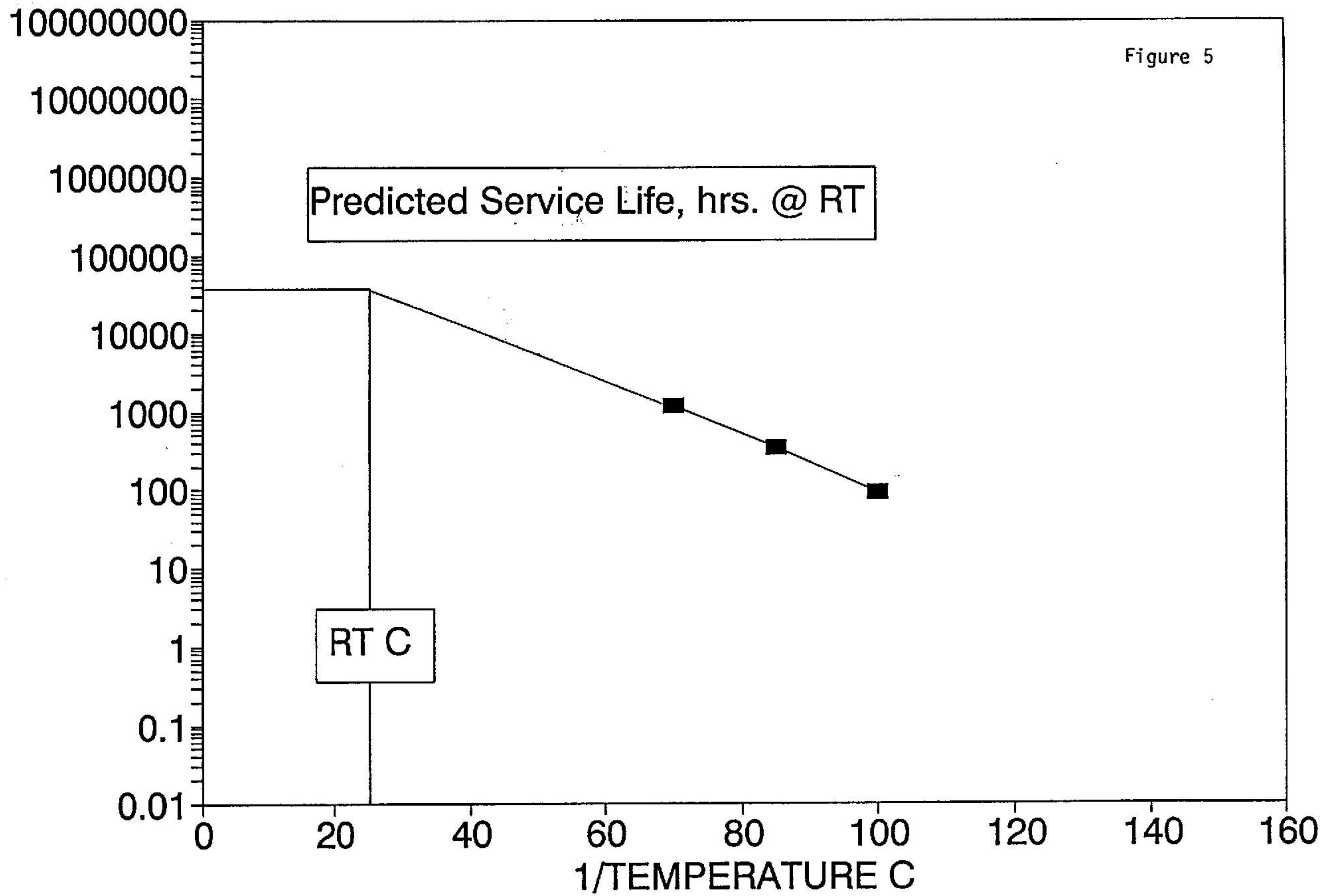


Figure 3
% Retained sealing force
100
90
80
70
60
50
40
30
20
10
0
0.1
1
10
100
1000
10000
Time, hrs.
Arbitrary "Failure Line"
100 C
85 C
70 C

ARDL,INC., PLASTICS DIVISION
COMPRESSION STRESS RELAXATION TEST

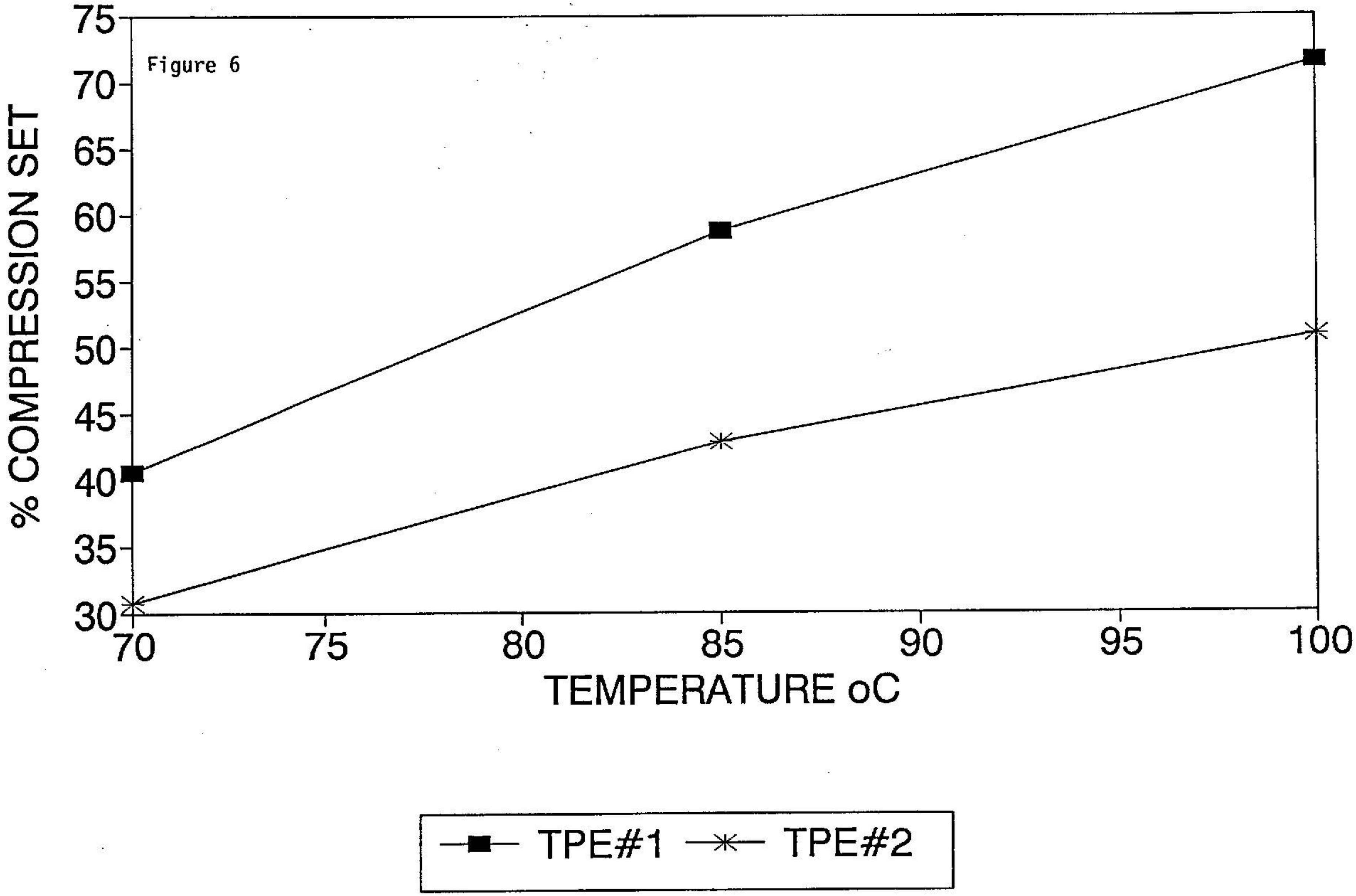
| TIME, Hr. | 70oC % S.F. RETAINED | | 85oC % S.F. RETAINED | | 100oC % S.F. RETAINED | |
|---|---|---|---|---|---|---|
| | TPE#1 | TPE#2 | TPE#1 | TPE#2 | TPE#1 | TPE#2 |
| 0.5 | 100 | 100 | 100 | 100 | 100 | 100 |
| 22 | 42 | 49.9 | 26 | 35.3 | 14 | 27.6 |
| 72 | 37.1 | 43.7 | 20.4 | 30.9 | 13.2 | 27.1 |
| 166 | 31.4 | 37.6 | 19.5 | 28.6 | 0 | 26.1 |
| 336 | 29.4 | 33.6 | 18 | 25.9 | 0 | 24.5 |
| 666 | 27.3 | 31.5 | 17.8 | 25.2 | 0 | 23.6 |
| 1000 | 25.2 | 31.8 | 13.9 | 24.5 | 0 | 21.8 |
| 2000 | 25.4 | 31.8 | 0 | 23.8 | 0 | 16.8 |
| 3000 | 25.6 | 33.2 | 0 | 22.9 | 0 | 11.7 |

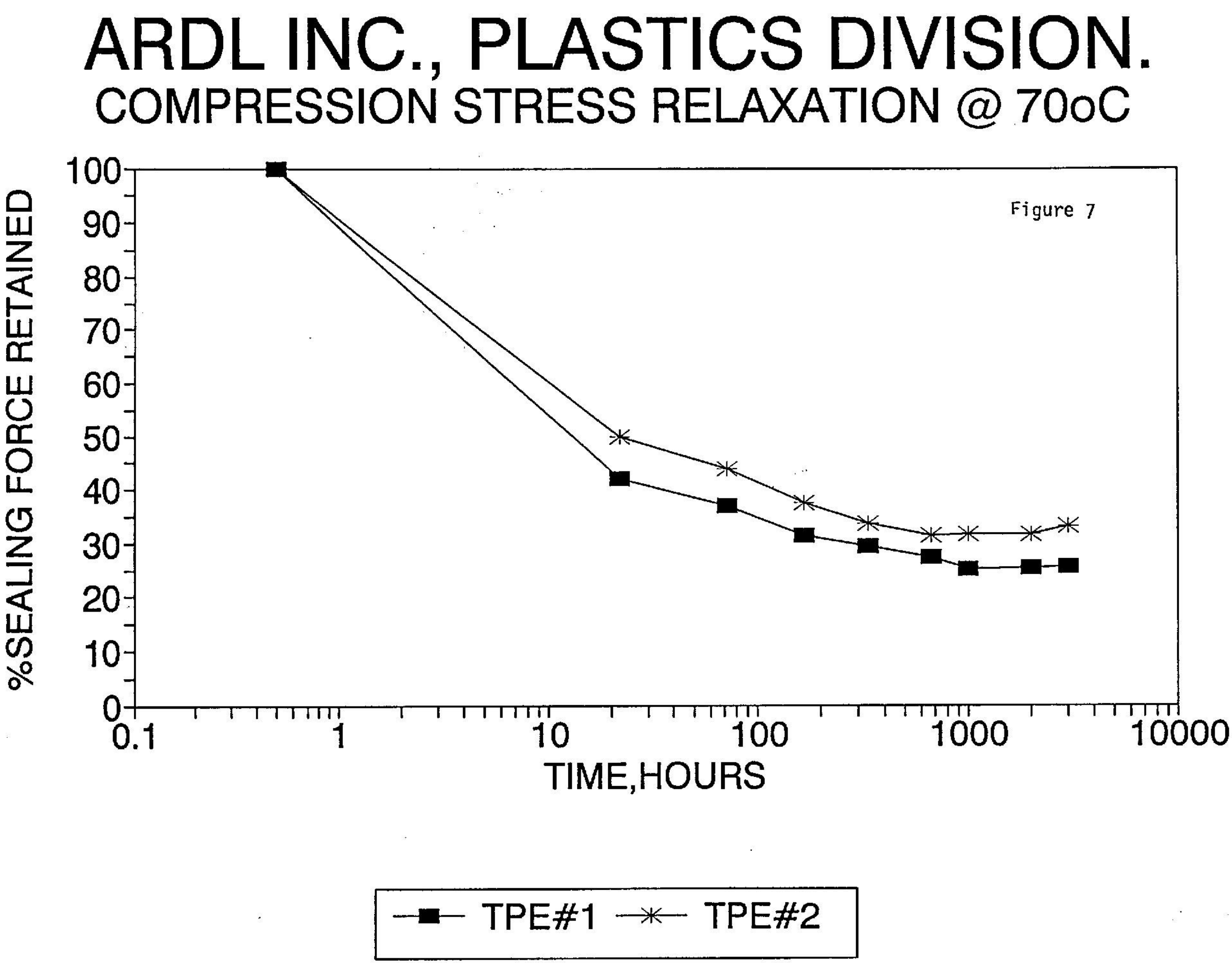
Figure 4


Predicted Service Life, hrs. @ RT
RT C
100000000
10000000
1000000
100000
10000
1000
100
10
1
0.1
0.01
0
20
40
60
80
100
120
140
160
1/TEMPERATURE C
Figure 5

# ARDL INC., PLASTICS DIVISION.

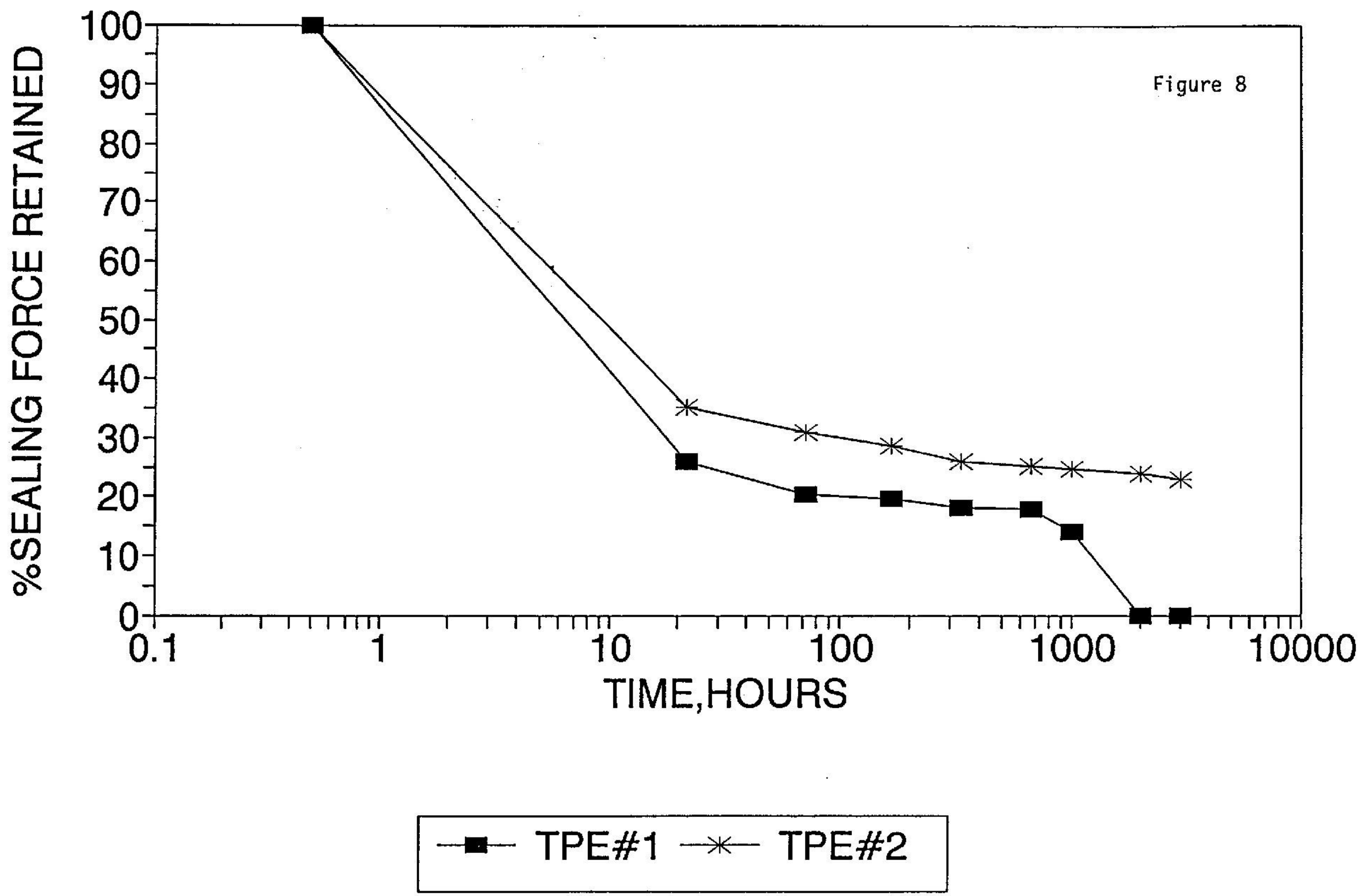
## COMPRESSION SET, ASTM D-395



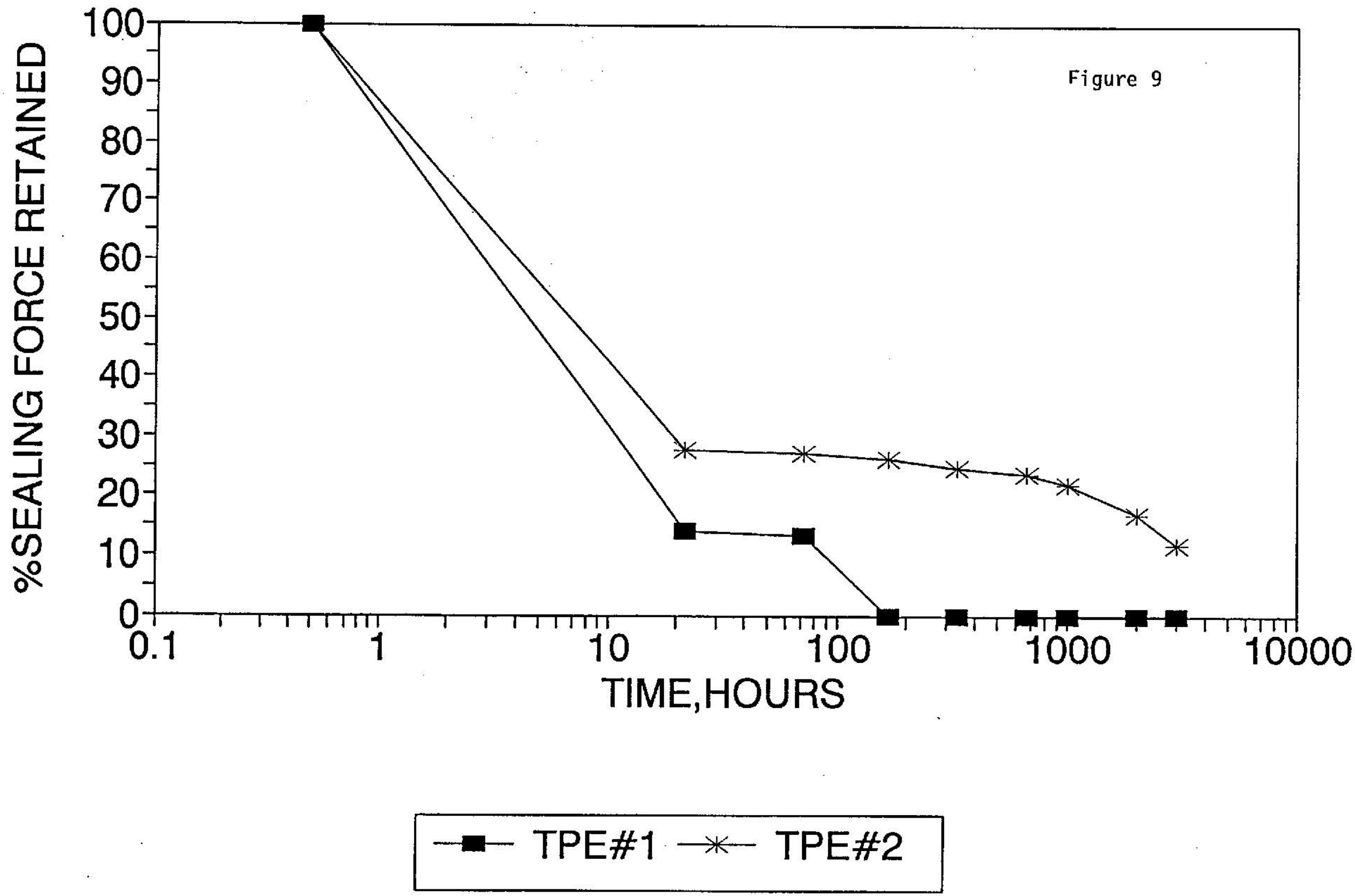

ARDL INC., PLASTICS DIVISION.
COMPRESSION STRESS RELAXATION @ 70oC
Figure 7
%SEALING FORCE RETAINED
100
90
80
70
60
50
40
30
20
10
0
0.1
1
10
100
1000
10000
TIME,HOURS
TPE#1
TPE#2

# ARDL INC., PLASTICS DIVISION.

## COMPRESSION STRESS RELAXATION @ 85oC



# ARDL INC., PLASTICS DIVISION.
## COMPRESSION STRESS RELAXATION @ 100oC



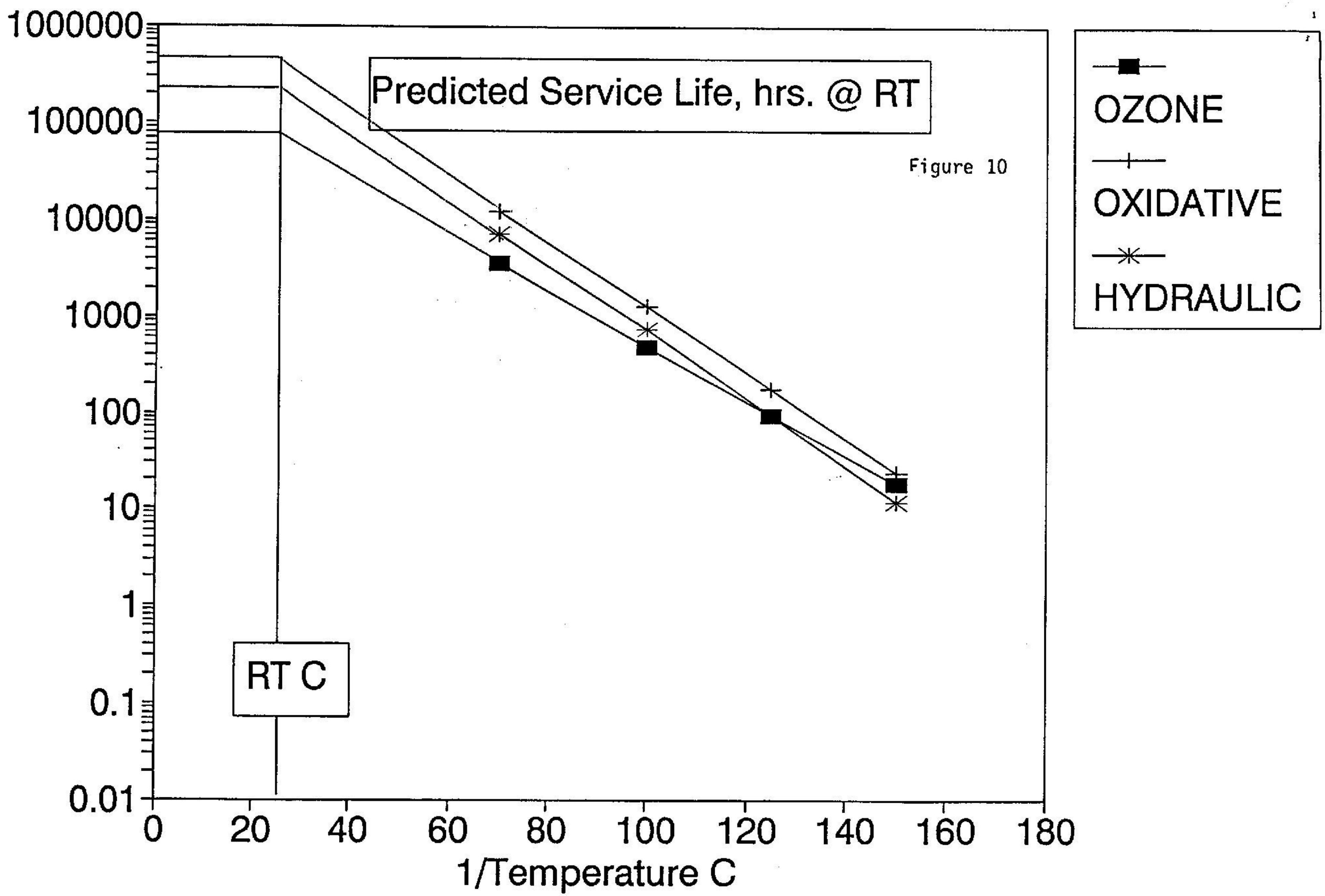

Predicted Service Life, hrs. @ RT
RT C
1000000
100000
10000
1000
100
10
1
0.1
0.01
0
20
40
60
80
100
120
140
160
180
1/Temperature C
OZONE
OXIDATIVE
HYDRAULIC

Figure 10