# Raycnem Energy Division

**Report\*** 

Pages: Title: 7 HEAT AGING STUDY OF WCSF COMPOUND Enclosures: Date: Report Number: 10 August 1978 EDR 2001 Approved by: Prepared by: Tested by: Ɗan Magay∩ David D. Nyberg Signature: Signature: Signature: bard A. Myberg Ca Jan C \*Report may only be **Raychem Corporation** used unchanged. **Energy Division** 

## ABSTRACT

Radiation crosslinked slabs of WCSF compound and WCSF 070/250 tubing were subjected to oven aging in air at 136°C. 150°C, 162°C and 175°C. Based on an Arrhenius analysis of the oven aging data, the time to 50% retention of original elongation plotted on semilog paper against the reciprocal of absolute temperature in degrees Kelvin yields a predicted service life for WCSF compound of 40 years at a continuous operating temperature of 91°C.

## <u>CONTENTS</u>

SECTION	TITLE	PAGE				
1.	Objective	1				
2.	Test Procedure	1				
	2.1 Specimen Preparation	1				
	2.2 Oven Aging	2				
3.	Test Results	2				
	3.1 Retention of Elongation	2				
	3.2 Arrhenius Thermal Plot	3				
	3.3 Calculation of Heat of Activation	3				
4.	Conclusions	4				
5.	Tables					
	5.1 Oven Aging Data - WCSF Compound					
6.	Figures					
	.1 Oven Aging Data - WCSF Compound					
	6.2 Arrhenius Thermal Plot - WCSF Compound	7				

#### 1. <u>OBJECTIVE</u>

To assess the thermal oxidative resistance of radiation crosslinked slabs of WCSF compound by oven aging of specimens in air at 136°C, 150°C, 162°C and 175°C.

#### 2. <u>TEST PROCEDURE</u>

#### 2.1 Specimen Preparation

Standard, pelletized, virgin WCSF compound was used. Slabs (6" x 6") were compression molded. Thickness was  $75 \pm 12$  mils. The slabs were crosslinked by radiation to the same crosslink density as WCSF tubing. Two Die D specimens from each slab were tested for tensile strength, ultimate elongation and tensile stress at 50% and 100% elongation (crosshead speed 2 inches per minute). The stress-strain values from this were averaged and used as the original values to which the heat aged samples were compared.

Forty three (43) slabs were prepared. Standard deviations for tensile strength, ultimate elongation and tensile stress at 50% and 100% elongation were calculated based on the original data from the two Die D specimens from each slab. Slabs were discarded if the above original stress-strain values were outside the mean value  $\pm$  twice the standard deviation for that value.

From the acceptable slabs (41), Die D specimens were cut and <u>all</u> combined. Five Die D specimens were chosen at random from the combined lot to provide one data point for the heat aging study. The original cross-sectional area of the specimens (pre-measured) were used to calculate the stress-strain properties after heat aging.

3

A few specimens of WCSF 070/250 plant production tubing were included in this oven aging study to allow some comparison to the slab data. The tubing was recovered prior to heat aging. Three specimens of recovered tubing were used to calculate an average original value for the stress-strain properties of interest. As with WCSF compound. five specimens were used for each data point.

## 2.2 Oven Aging

Forced air type Blue M ovens were used for heat aging at 136°C, 150°C, 162°C and 175°C. The ovens were calibrated with a 12 point recording thermocouple set-up at 12 different zones in the oven chamber. The temperature was monitored regularly with a single thermocouple permanently assigned to each oven. The temperature variation was less than  $\pm 2\%$  of the specified exposure temperature in degrees centigrade.

The specimens in groups of five were hung vertically from the oven tray utilizing metal clips and hooks in the conventional Raychem manner.

## 3. TEST RESULTS

#### 3.1 <u>Retention of Elongation</u>

The property of prime interest in this study was retention of original elongation which is a measurement of flexibility (or lack of brittleness). Both slabs and tubing oven aged at essentially equivalent rates with a reduction of tensile strength and with a slow increase in tensile stress (modulus) or stiffness.

4

The retention of original elongation versus time was plotted during the oven aging periods at the four different temperatures. In Table 5.1 the time (hours) to various percent retentions of original elongation are tabulated and this data is presented in graphical form in Figure 6.1.

## 3.2 Arrhenius Thermal Plot

In order to construct the Arrhenius thermal plot in Figure 6.2, we chose an end-point for retention of original elongation of 50X. The four experimental points for 136°C, 150°C, 162°C and 175°C yield a reasonably straight line predicting a service life of 40 years at a continuous operating temperature of 91° based on a conventional Arrhenius analysis of the time to 50% retention of elongation plotted on semilog paper against the reciprocal of absolute temperature i n degrees Kelvin.

## 3.3 Calculation of Heat of Activation

From the slope of the Arrhenius thermal plot, the heat of activation for the thermal oxidation of WCSF compound was calculated to be 29 kcal/mole for the end-point of 50% retention of original elongation. This was calculated using the following equations:

 $b = 2.303 (log_{10} t_2 - log_{10} t_1)$ 

 $\frac{1}{T_2} - \frac{1}{T_1}$ 

Heat of Activation =  $\Delta$  H act = R x b

where	$t_2$ = time to endpoint (100,000 hours)					
	$t_1 = time to endpoint (1000 hours)$					
	$T_2 = °C + 273$ corresponding to $T_2$					
	$T_1 = °C + 273$ corresponding to $T_1$					
	$b = \Delta H \operatorname{act/R}$					
	R = 1.98 cal mole $^{-1}$ °C $^{-1}$					

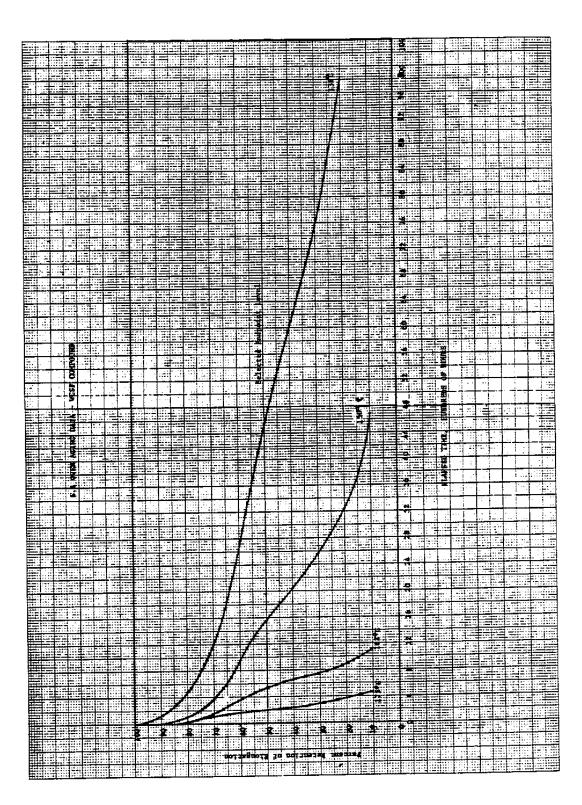
## 4. <u>CONCLUSIONS</u>

On the basis of the oven aging study described in this report and the use of a conventional Arrhenius analysis of the data, it is concluded that the useful service life of radiation crosslinked WCSF compound is predicted to be 40 years at a continuous operating temperature in excess of 90°C. The heat of activation for the thermal oxidation of WCSF compound was calculated to be 29 kcal/mole.

# 5. <u>TABLE</u>

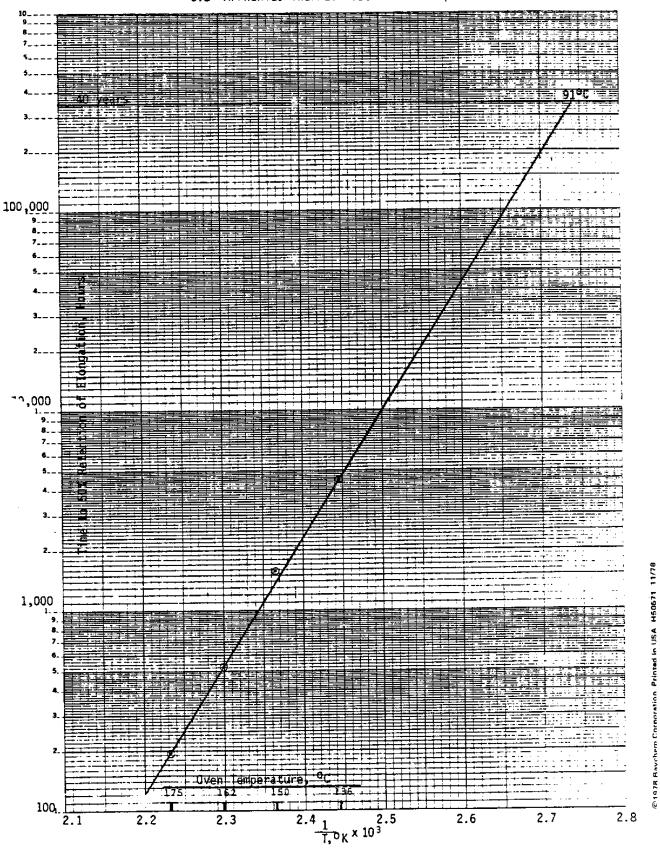
## 5.1 OVEN AGING DATA WCSF COMPOUND

Oven									
Temperature,			TIME (HOURS) to						
°C		Various Levels of Retained Elongation							
	90%	80%	70%	60%	50%	40%	30%	20%	10%
136	71	470	1230	2700	4500	6020	7510	9810	
150	35	107	331	960	1570	2030	2600	3230	4720
162	12	30	130	350	521	637	744	876	1100
175	4	20	112	154	194	228	279	361	450



#### EDR-2001 8/10/78

EDR-2001 8/10/78



6.2 Arrhenius Thermal Plot - WCSF Compound