Accelerated Ageing Through Thermal Cycling of Optical Coupling Samples to Investigate Performance Degradation in Detectors

K. L. Loughney, Z. Sharpe

NUCLEAR SECURITY TECHNOLOGIES

Introduction:

Thermal cycling of materials aims to accelerate the ageing process through an exaggeration of the daily diurnal temperature variations that occur, putting the materials under thermal stress without introducing thermal shock from rapid temperature changes.

The Coffin-Manson equation (right) can be used to estimate the number of thermal cycles required to accelerate the ageing to the equivalent of a year in real time. This enables an understanding of how materials will react and behave in real world situations, within a shorter time-frame.

For this investigation, samples of EJ550 optical grease were thermally aged up to the equivalent of 20 years real time through thermal cycling between 5 and 75 °C. The ΔT_{Life} value was obtained from research on average daily diurnal temperature variations reported by the Met Office [1].

To account for the 3 months required to complete this, additional control samples were created to ensure any variation observed was due to thermal ageing, not the passage of time.

$$C_{TOTAL} = \frac{t_{yr} \cdot C_d \cdot 365}{A_F}$$

$$A_F = \left[\frac{\Delta T_{Test}}{\Delta T_{Life}}\right]^3$$

t_{yr} = Time in years to emulate (converted to days in the equation)

C_d = Number of complete cycles per day

A_F = Acceleration Factor

ΔT_{Test} = The temperature variation being used for the thermal cycling

ΔT_{Life} = The average temperature variation that the material undergoes in life for the location of use

Table 1: Table of variables and calculated required number of cycles per year equivalent when using the Coffin-Manson equation.

Real Life Temp Variation Comparing Against (Δ°C)	15 [1]	Cycle Duration (hrs):	14
Min Temperature of Test Range (°C)	5	# of Completed Cycles per Day:	1.71
Max Temperature of Test Range (°C)	75	# of Cycles/Year Equiv. :	6.15
Temperature Ramp (°C/hr)	10	# of Cycles to reach total Years to Emulate:	123.14
Number of Years to Emulate? (Yr)	20	Days of Chamber Use Required:	71.83
Acceleration Factor:	101.63	Days of Chamber Use Required per Year Equiv. :	3.59

Samples:

A total of 66 samples were created, thermally cycled in a Kambic KK-50 Environmental chamber at the University of Surrey (see Table 1 for details of the thermal cycle) and optically characterised using a Varian Cary UV-Vis Spectrophotometer for the investigation, utilising four different substrate/dimension combinations of:

- **Microscope Slides**: 0.02g of optical grease between two glass slides, focusing on potential evaporation.
- **Petri Dishes**: 2ml of optical grease syringed onto the bottom of a 2" petri dish in a tight spiral and pressed between a secondary petri dishes to mimic realistic detector coupling. A steel weight was mounted on each sample that provided the equivalent weight to a standard 2" PMT (See left in Figure 1).
- **Petri Slides**: 1.5ml and 4ml samples within a petri slide to provide a 1" and 2" diameter sample, respectively. The excess space allowed for the potential migration of material to be observed, which was not as easily visible in the petri dish samples.

All samples were then divided into groups:

- **C Samples - Per Year:** Removed, recharacterised and returned into the chamber at the end of every year equivalent cycle.
- **D Samples - Full Duration:** Remained in the chamber and cycling until the final cycle was completed to determine if the removal of samples had an impact.
- **R Samples - Room:** A control set of samples left beside the chamber, at nominal room temperature, and not cycled to ensure changes were a result of cycling, not passage of real time.
- **NB Samples – "No Bubble":** Additional samples introduced after the 10th cycle to determine the impact of air bubbles present in the sample. Samples were created and given 1 month to settle prior to cycling.

Blank references were made for identical volumes of air to account for the presence of the substrate in optical characterisation and any ageing the substrate undergoes and for baseline/zero corrections performed when using the Varian Spectrophotometer. The percentage transmission (%T) of samples was measured over a wavelength range of 300 to 700 nm in order to cover the emission range of common scintillators, such as EJ200.

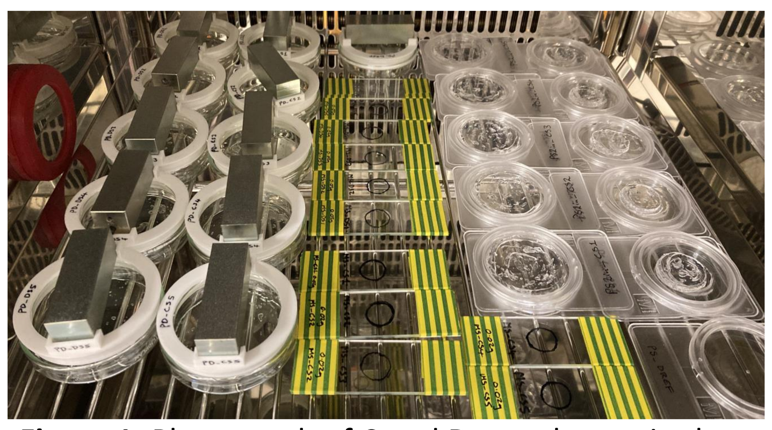

Figure 1: Photograph of C and D sample sets in the Kambic Environmental chamber used for thermal cycling.

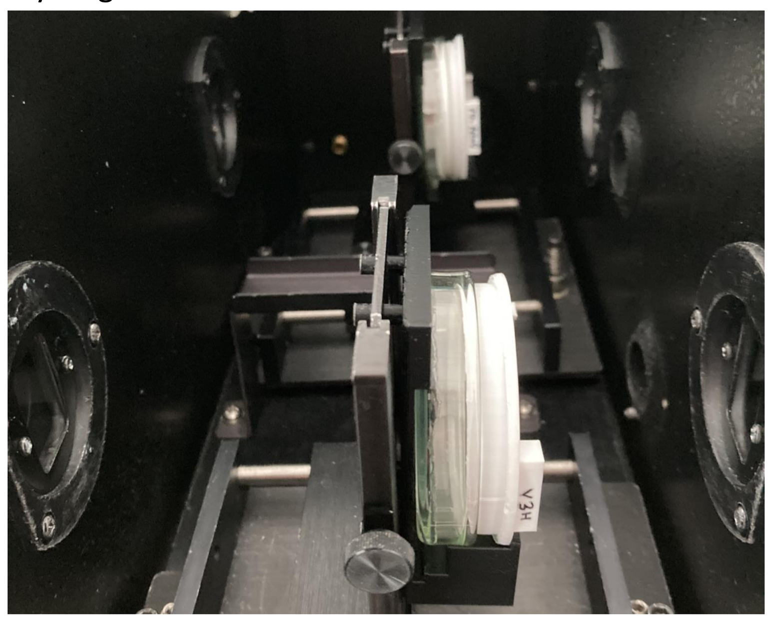

Figure 2: Photograph of the sample chamber for the Varian spectrophotometer, showing an example of a petri dish sample (bottom) and blank reference (top) in position.

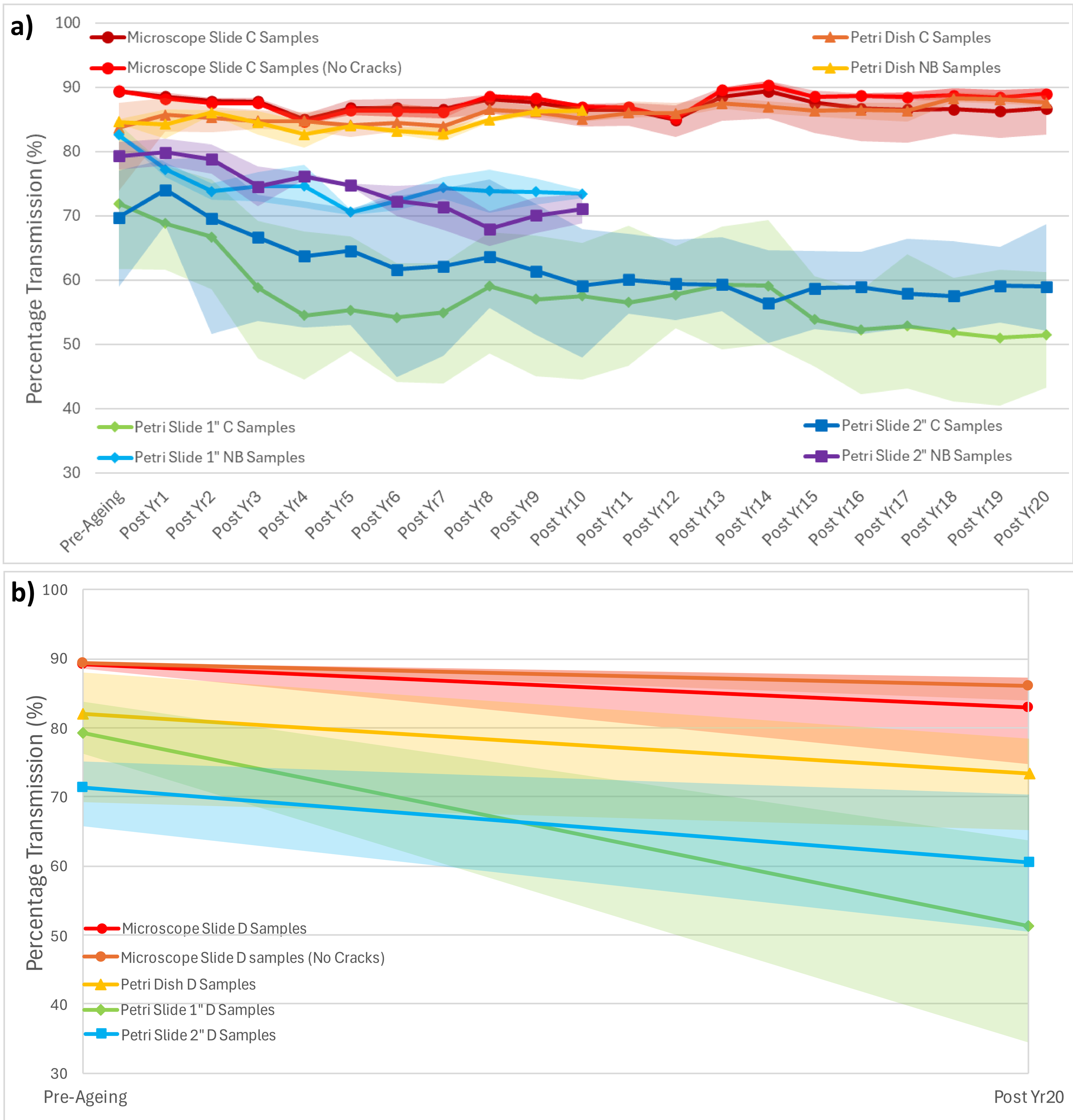

Figure 3: Comparison of the average transmission for each substrate/dimension combination for a) C and NB samples for each year equivalent and b) D samples following 20 years equivalent. The shaded regions show the variation across the five samples for each substrate/dimension combination. An additional line has been included for the microscope slides to highlight the impact on transmission of cracks that appeared in the microscope slides during the thermal cycling.

Post Thermal Cycling up to 20 Years Equivalent:

Figure 3 shows the averages for each sample substrate/dimension combination for the C and D samples. R samples have not been included as they remained consistent with pre-ageing characterisation values or showed minor increases due to dissipation of bubbles, meaning any variations in the C, D and NB samples is a result of thermal cycling.

- Microscope slide and petri dish C samples displayed no significant variation with the "yearly" characterisation
- Both 1" and 2" petri slide samples displayed degradation for all C and NB samples, with a consistent average decrease in %T value of 10% by the 10th year equivalent, with further degradation to a total decrease in %T value of 20.
- All D samples that remained in the chamber displayed degradation in the transmission, with the 2" petri slide and the 2" diameter petri dish samples showing a consistent average decrease of 11% and 9% in %T value, respectively.
- It was observed that the C and D petri slide samples displayed different behaviour with the presence of bubbles when comparing before and after photographs (Figure 4 & 5). Bubbles in C samples were observed to noticeably increase in size with the thermal cycling while D samples were seen to have minor variations, with smaller bubbles dissipating or merging with others. Both show that there was a migration of material throughout the cycling.

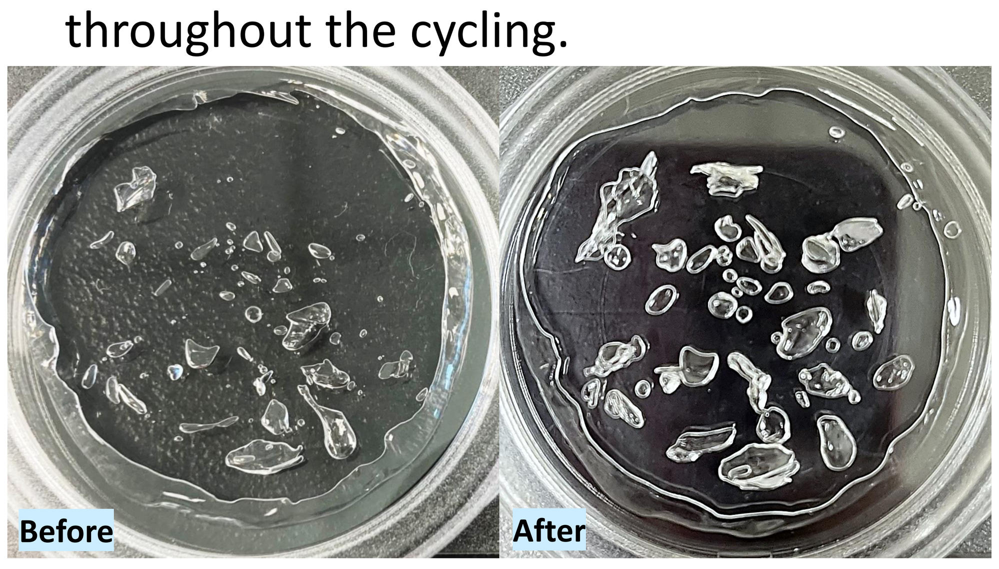

Figure 4: Characterised (C) 2" petri slide sample before and after 20 years equivalent ageing.

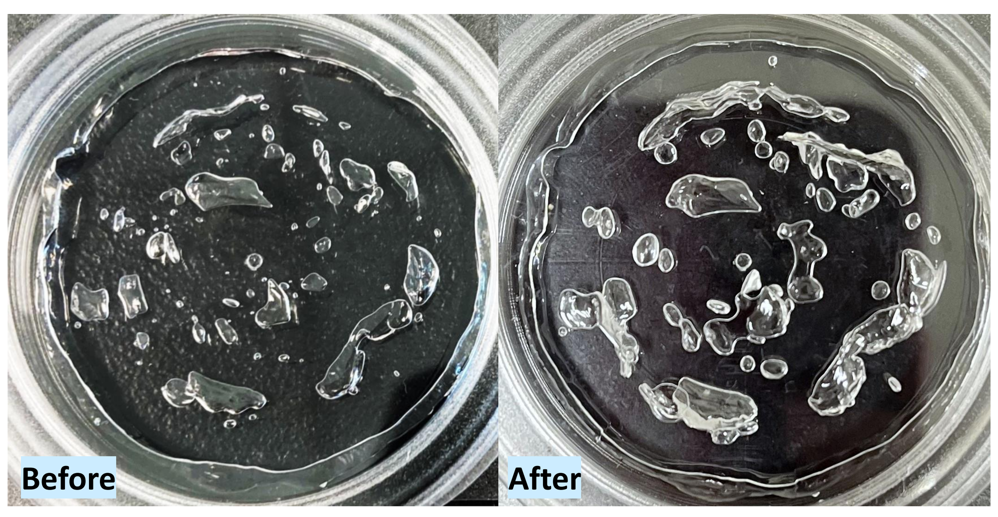

Figure 5: Duration (D) 2" petri slide sample before and after 20 years equivalent ageing.

References:

[1] Met Office, "A review of the UK's Climate in 2024", Met Office: A review of the UK's climate in 2024 - Carbon Brief

AWE, Aldermaston, Reading, Berkshire, RG7 4PR, UK