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Investigation of Accelerated Aging Effects in Phenolic Ablative Composites

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Abstract: C Phenolic and Si Phenolic are the special composite materials used for ablative thermal protection in solid motor nozzles for aerospace launch vehicles. These materials are highly specialized composite materials made from C / Si fibres and special phenol-formaldehyde resins. These materials can withstand very high temperature of 1500C to 3000C in the nozzle of the solid rocket motors by the principle of self sacrificing and protecting the backend hardware to a very low temperature of less than 100C when the actual nozzle temperature is of 3000C. The amount of fibre (C, Si), the matrix material (phenolic resin) and the different combination play a key role in the ablative performance. This paper deals with preparation of carbon phenolic laminate which is subject to accelerate the aging conditions to determine the effects of aging on ablative, thermal and mechanical properties of carbon Phenolic for 1yr, 2yrs & 3yrs respectively and comparing the values with an un-aged laminate part. This entire work shows the significance of accelerated aging effects in phenolic ablative composites.

Keywords: Phenolic laminates, Aging, Thermal effects, Mechanical properties, Aerospace nozzle

1 Introduction

The advanced composite materials like phenolic ablatives used in aerospace has definite variations in its original size of the structure due to extreme heat load developed above the parts of aerospace vehicles. The environment effects eventually affect the designed properties of the material. This effect is known as aging. This may penetrate in the material structure and degrades the mission-critical components which are used in space vehicles and have a potentially catastrophic effect on both the vehicle and payload. Hence, investigating the significance of aging process in advanced aerospace materials is a huge task in construction, property design and safe operation. Such materials consist of various degradation mechanisms viz. Physical, Chemical, Mechanical and Thermal. These mechanisms are depends on the environmental effects and mechanical loads.

Sanjana [1] has explained about the aging behaviour of unidirectional Carbon fibre / epoxy system with contents of resin having 42% and 35% by weight under various humidity and heat conditions. This work shows that due to aging the dynamic mechanical analysis and dielectric analysis are used to correlate the loss of tack [1]. Kempner has studied the effects of aged materials on the autoclave cure of thick composites. The aging occur due to exposure of AS4/3501-6 graphite/ epoxy prepreg to ambient conditions can be calculated. It is found from the study on extent of reaction that could result such as outtime was calculated to be 0.06 after 10 days and 0.16 after 30 days [2]. Chung [3] has presented an evaluation of thermal degradation on carbon fiber cyanate ester composites to identify the kinetics parameters at room and high temperatures.

This work shows that curing is not much reliable at room temperature. Similarly, the experiment were based on accelerated aging environment to investigate the degradation and deterioration process. The results reveal that the interface between fibre and resin of unaged cured laminate is uniform without any void content in the resin matrix. Due to aging, the glass transition temperature of cured composite laminate was found to decrease dramatically and the relaxation time was also decreased [3]. Akay [4] has studied the effects of prepreg aging in

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its behaviour of carbon fiber epoxy laminates. The obtained result shows that the hot/wet conditioning causes reductions in Inter Laminar Shear Strength (ILSS), compressive strength, flexural strength in the range of 10%, 17% and 7% respectively [4]. D.M. Taylor and K.Y. Lin [5] has studied the aging effects on the ILSS of high performance composites at various temperature and its results were compared with previous data. It is found that for thermoset bismaleimide laminate, the aging effects are highly significant.

After the intial increase, the interlaminar shear strength steadily decreases due to degradation of matrix properties. Degradation in its strength occurs not only for an increase in aging temperature, but also its time increases [5]. N. Winya [6] explained about the factors affecting the ablation rate of phenolic resin and glass fibre by using two ways factorial design to screen the factors to those factors that affect the ablation rate significantly [6]. From various experimentation done by many researcher it is identified. The effect of ablation rate is depends upon curing time, curing temperature, amount of phenolic resin and interaction between each of them. Results show that for 75% wt of phenolic resin, the curing should be at 160C for 35 minutes to give the ablation rate of 0.121 mm/s which is less than 0.14 mm/s. Hence accordingly the MIL 1- 24768 were the best condition.

2 Accelerated Aging

To determine longterm effects in shorter times accelerated aging is essenitial [7],[8]. The aggravated conditions of heat, oxygen and vibration etc are used to speed up the normal aging [9],[10]. All its properties and characteristics change in rapid time is termed as accelerated aging [11],[12]. Accelerated aging methods are needed to provide guidance for materials selection and to accurately assess aging of new materials [13],[14]. Several researchers tried different ways of accelerated aging but till date no pinpoint method is splet out [15],[16]. The highly empirical approaches taken for the majority of accelerated aging studies dictate that the primary objective of an accelerated aging method is to screen and characterize new material systems [17],[18]. Material testing requires costly laboratory equipment [19],[20].

Accelerated aging may reduce the expense and time [21],[22]. The empirical method for accelerated aging may address the concerns for specific applications and environments, but the need for predicting performance in broader service conditions required the development of empirical methods coupled with analytical methods [23],[24]. The development of accelerated aging methods requires extensive testing to define critical environmental stress factors and their interactions [25]. The researcher shows the characteristics of ablative composites and its aging effects [26]. The applications of accelerated aging and its few testing techniques is confirmed by some researchers [27],[28].

3 Theoretical Calculations For Accelerated Aging

From the below formula one can theoretically calculate the accelerated aging:

$$AAT = \frac{DRT}{Q_{10}[\frac{T_{AA}-T_{RT}}{10}]} \tag{1}$$

where; AAT is Accelerated Aging Temperature; DRT is Desired Real Time. Where,
TAA = Accelerated Aging Temperature, 85°C
DRT = Desired Real Time
TRT = Ambient Temperature, 30°C
Reaction Rate Factor (Q) = 2.2

This shows that the chemical reaction is approximately doubles for every 10C rise in temperature from ambient temperature. Using the above formula it is identified that the values of AAT for different years results 4.7, 9.5 & 14.5 for the first three years of acceleration.

4 Preparation of Carbon Phenolic Laminate

In this process, dry carbon fabric is passed through a phenol-formaldehyde resin bath and taken through a set of squeeze rollers and finally through heating chamber to get carbon phenolic prepreg. This stack of prepreg plies to the required size over a flat surface. Then the stacked prepreg sheets are loaded into a layup machine after which it enters a computer controlled vacuum press for curing under pressure which is carried out on a 250Ton Hydraulic Press. It consists of platten size $1000 \times 1000 mm$. The ram diameter and stroke are 360 mm & 1200 mm. It can operate at Max. temp of 250°C. Curing process starts from room temperature which is ramp to 90°C at 10 ton load. The laminate is soaked for 1hr at this level. Next the temperature is slowly raised to 120°C at 20 ton load. Soak for another 1hr at this stage. Temperature is slowly raised again until it reaches 155°C, the load is also increased to 30 tons. In this stage of the curing cycle, the laminates are soaked continuously for a period of 3hrs. The curing details are shown in Table 1 & 2. The machine is switched off and the laminate inside is allowed to cool for a period of 12 hrs. Using similar manner two numbers of carbon phenolic laminates are made. The dimensions of the laminate are shown in Table 3. Each laminate is divided in half. So there are totally 4 parts. Each part is marked as L1, L2, L3, and L4. The laminate part L4 is un-aged and taken as reference. The parts L1, L2, L3 are subjected to accelerated aging. In this study, accelerated aging by thermal means is adopted.

Test temperature needs to be chosen considering the trade-off between test duration and degradation of material. The test temperature of $85°C$ is chosen upto which the phenolic composite is stable. The duration of temperature application to simulate accelerated aging for 1yr, 2yrs and 3yrs are calculated. The calculated durations are 5 days for 1yr, 10 days for 2 yrs and 15 days for simulating 3 yrs of normal aging. The aging chart with laminate dimensions before and after heating is clearly explained in Tables 4, 5 & 6.

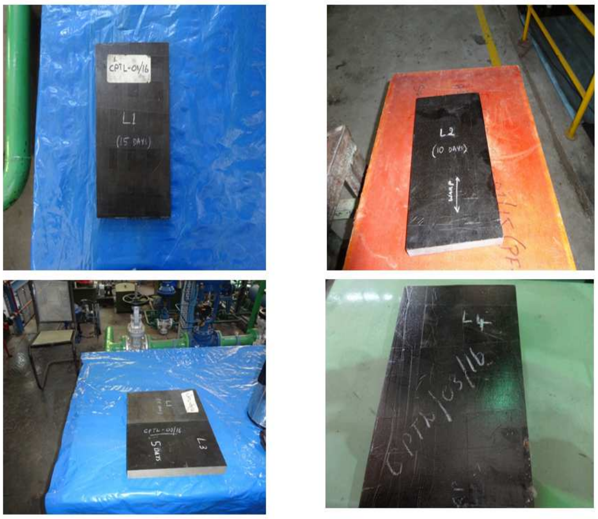

Fig. 1: Various types of prepared laminates

5 Determination of Ablative Properties

The prepared laminates are shown in Fig.1. Its dimension details are shown in Table 7. The ablative properties like heat of ablation & erosion rate are determined by using plasma jet operation. An inert gas Argon or Nitrogen is passed through a glass chamber consisting of an anode and a cathode. An arc is developed between anode and the cathode. The specimen and the holder unit is placed at a distanced from the source of plasma jet. The plasma jet of heat flux, q = 750 w/cm^2 is allowed to strike on the specimen for time, t = 15s. The length and weight of the specimen before and after the test is noted.

5.1 Determination of Thermal Properties

In this technique, a sample is placed within a controlled atmosphere furnace and subjected to a finite impulse of radiant energy on its front surface, through the use of a laser. The transport of heat through the sample, as a result of the laser impulse, causes a transient temperature rise on the rear surface of the specimen. This temperature rise is measured by an IR detector placed below the rear sample surface. From the recorded data of the resulting transient temperature rise at the back face of the sample, Thermal conductivity (k) , Specific heat (C_p), and Thermal Diffusivity along ply and across ply can be deducted. The results are ploted in various graphs. The various mechanical testing are conducted using the specified equipment.

5.2 Effect of Aging on Ablative Properties

The ablative properties such as erosion rate and heat of ablation are tested for the four samples along the ply direction (parallel) and across the ply direction (perpendicular). The average properties are plotted in figures 2 and 3 respectively for heat of ablation and erosion rate.

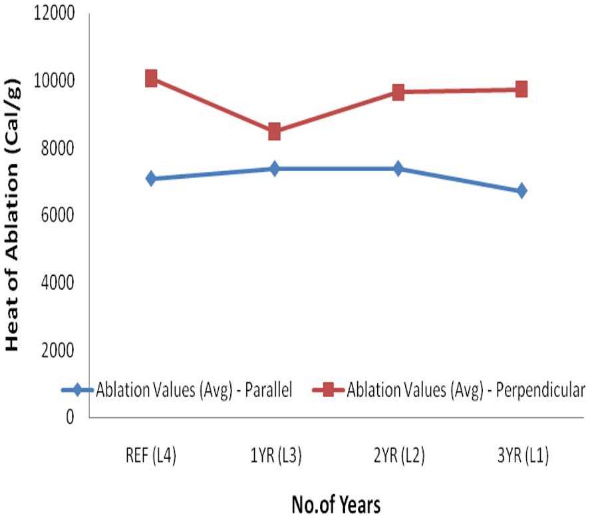

Fig. 2: Heat of ablation chart

The heat of ablation and erosion rate in parallel direction shows that no appreciable change for 2 years but it shows a minor deterioration after 3rd year. The erosion rate increases by 18.6% of reference whereas the heat of ablation reduces by 5.17% of reference value after 3 years. The ablative properties in perpendicular direction shows some deterioration after 1 year but recover and stay nearly stable after second years. This may also be due to testing problems of the particular set of specimen. The heat of ablation reduces by 3.2% of reference after 3 years whereas erosion rate increases by 25% of reference after 3 years. A higher heat of ablation and lower erosion rate desirable for giving better ablative performance. It is seen that ablative performance is not affected by aging for

Table 1: Temperature cycle of curing process

Oven Set Temperature	Soak	Remarks
$90^{\circ}C$	1Hr	Platen Temperature
$120^{\circ}C$	$\frac{1}{2}$Hr	Platen Temperature
$155 \pm 5^{\circ}C$	3Hrs	Product Temperature

Table 2: Load cycle of curing process

Product Temperature ($^{\circ}C$)	Load (T)
RT-10	3
60	6
70	7
80	8
90	10
95	15
100	20
105	25
115-155	30

Table 3: Detailed specifications of laminates

No	Dim. before curing	Plies	Wt. Before Curing	Wt. After Curing	Thickness after curing	Dim. after trimming
Laminate 1	330x300x30	102	5.801	4.07	26	329x294.5x26
Laminate 2	330x300x35	120	6.98	5.19	33.2 to 34.2	328x293x33.5

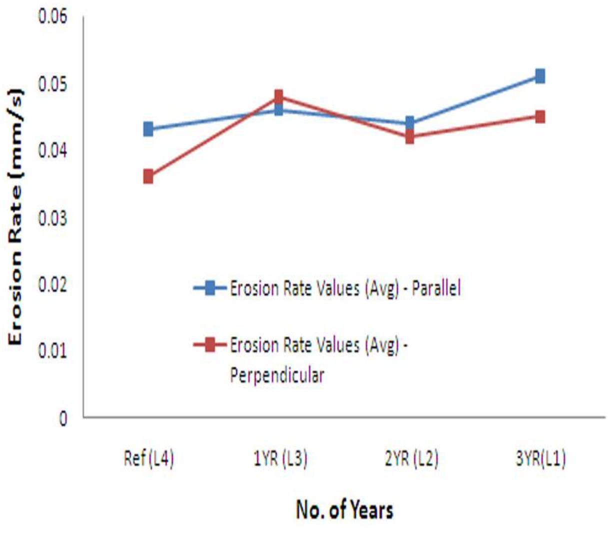

Fig. 3: Erosion Rate Chart

3 years but show tendencies for reduction in ablative performance after third year, and the effect is more predominant for erosion rate.

5.3 Effect of Aging on Thermal Properties

The thermal properties such as diffusivity, thermal conductivity and specific heat are tested for the 4 samples along the ply direction (parallel) and across the ply direction (perpendicular). The average values of diffusivity, specific heat and thermal conductivity are plotted in figures 4 to 9 respectively.

Thermal diffusivity in both parallel and perpendicular directions remains nearly stable till 1 year but shows an increase of 13 to 22% by the end of 3rd year at 30C and 100C respectively. Specific heat remains nearly stable and changes below 3%. Thermal conductivity show some peculiar behaviour.

Thermal conductivity in parallel direction at 30C showed a reduction after 1 year but monotonically

Table 4: Aging Chart

Laminate	Duration	Remarks
L1	15days (360 hrs)	3 yr
L2	10 days (240 hrs)	2 yr
L3	5 days (120 hrs)	1 yr
L4	-	Un-aged

Table 5: Dimensions before heating

Laminate No	Length (mm)	Width (mm)	Height (mm)	Weight (Kg)
L1	295	166	26.60	1.964
L2	293	166	26.00	1.922
L3	293	164	33.5	2.433
L4	293	164	34.00	2.467

Table 6: Dimensions after heating

Laminate No	Length (mm)	Width (mm)	Height (mm)	Weight (Kg)
L1	293.6 - 294.5	164 168.6	26.6 27.1	1.958
L2	293	166	26 27.8	1.917
L3	293 294	163.7 164.2	33.5 35.5	2.430
L4	294	164.2-164.4	33.5 35.5	2.463

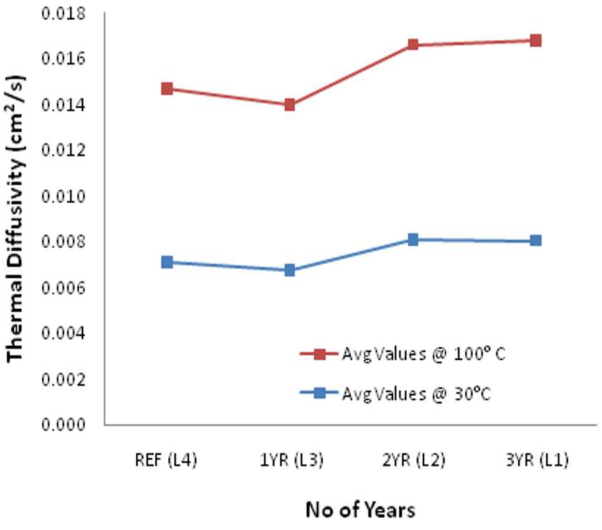

Fig. 4: Thermal Diffusivity Chart (Parallel)

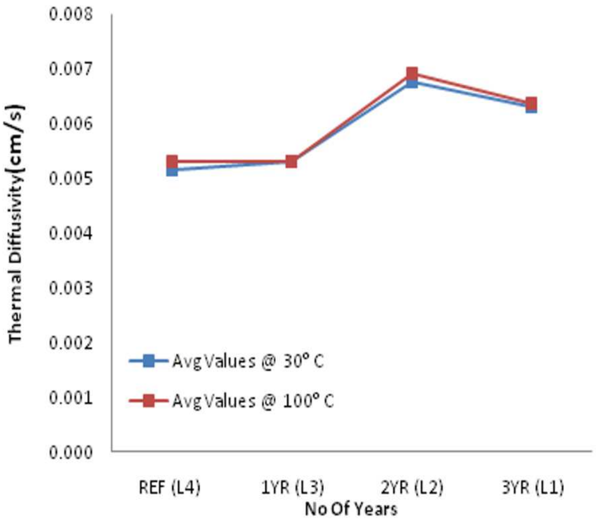

Fig. 5: Thermal Diffusivity Chart (Perpendicular)

increase after that, ending up 13% higher than reference value. On the other hand, the same property at 100C increases monotonically for first two years and reduces during third year ending 7.6% lower than reference.

Table 7: Dimensions after heating

Sl.No	Specimen Name	Dimension (mm)	Qty (Nos.)
1	Ablation Specimen with holder (Parallel)	$\phi 10 \times 20$	3
2	Ablation Specimen with holder (Perpendicular)	$\phi 10 \times 20$	3
3	Thermal Conductivity Specimen for Laser flash (Warp)	$\phi 12.7 \times 2$	3
4	Thermal Conductivity Specimen for Laser flash (Perpendicular)	$\phi 12.7 \times 2$	3
5	Compressive Strength (Warp)	$\phi 8 \times 16$	5
6	Compressive Strength (Perpendicular)	$\phi 8 \times 16$	5
7	Compressive Modulus (Warp)	$12.5 \times 12.5 \times 12.5$	5
8	Compressive Modulus (Perpendicular)	$12.5 \times 12.5 \times 12.5$	5
9	ILSS Specimen	$5 \times 9 \times 20$	10

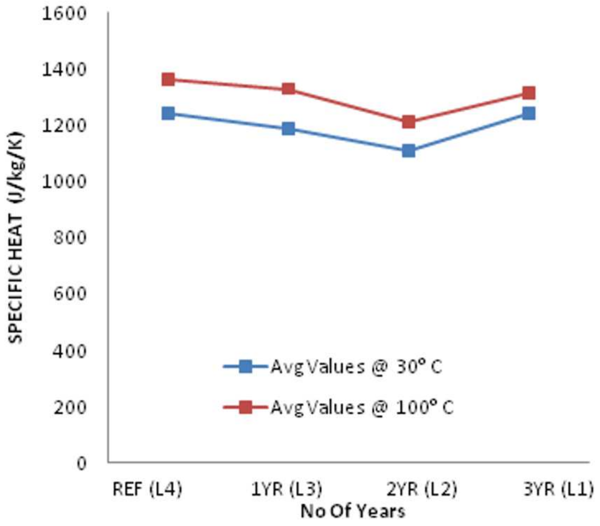

Fig. 6: Specific Heat Chart (Parallel)

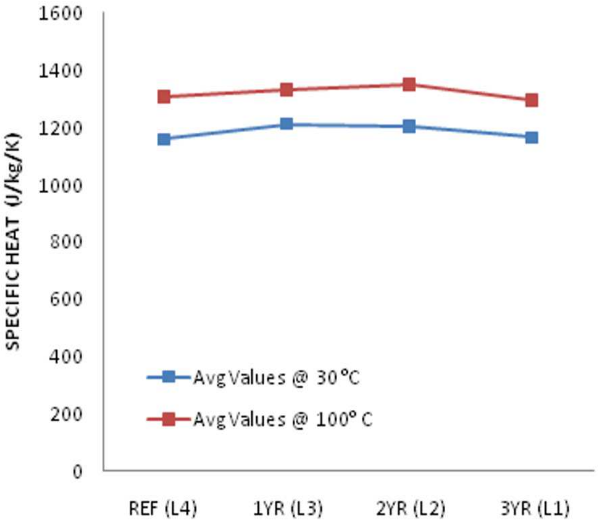

Fig. 7: Specific heat Chart(Perpendicular)

Thermal conductivity in perpendicular direction at both the test temperatures increased till 2 years then decreased ending at 19 to 21% higher than the reference value. A lower thermal diffusivity which results from lower thermal conductivity and higher specific heat is preferred for ablative applications as it results in less thickness of the ablative liner and hence lower weight. Thermal diffusivity show increasing trend after 1 year of aging which is mainly contributed by thermal conductivity as specific heat remains stable. The behaviour of thermal conductivity shows no particular trend which needs to be investigated further.

5.4 Effect of Aging on Mechanical Properties

The mechanical properties namely youngs modulus, compressive strength and inter-laminar shear strength are tested for the 4 samples along the ply direction (parallel) and across the ply direction (perpendicular) and average values of these properties are plotted in figures 10, 11 & 12 respectively.

Compressive strength in both parallel and perpendicular directions show no appreciable change during 3 years of aging, with change of less than 4% from reference value. Compressive modulus in parallel & perpendicular directions shows an increasing trend. An increase of 7.8% and 10.1% of reference value is observed after 3 years in parallel and perpendicular directions respectively. ILSS shows a downward trend, ending at 17.3% lower than the reference value at the end

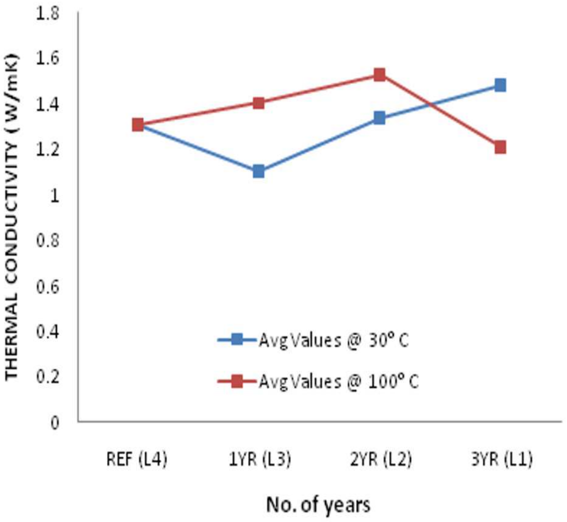

Fig. 8: Thermal conductivity chart (Parallel)

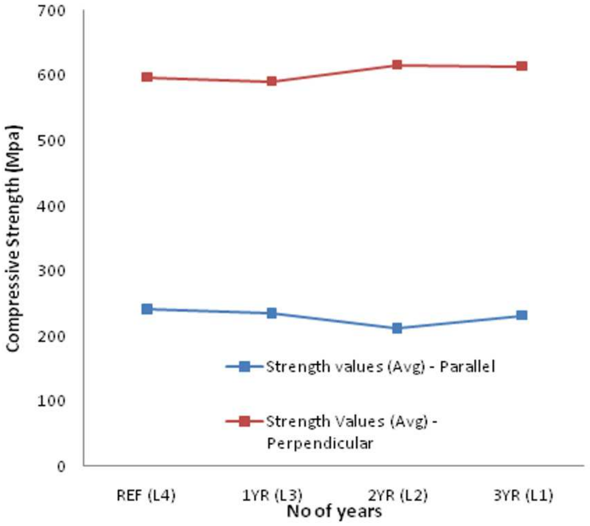

Fig. 10: Compressive strength chart (Parallel)

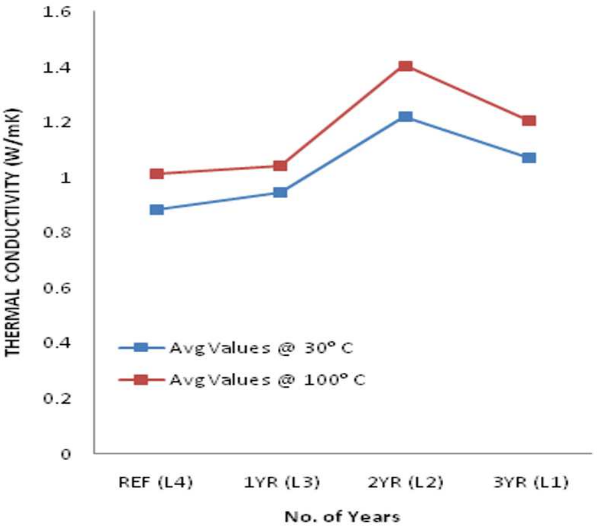

Fig. 9: Thermal conductivity chart (Perpendicular)

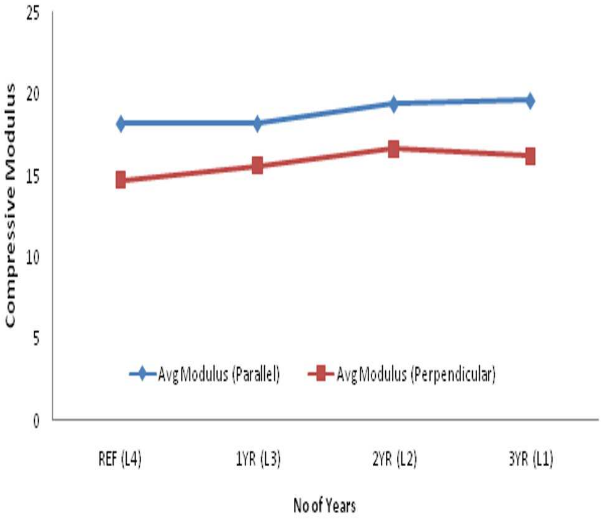

Fig. 11: Compressive modulus chart (Perpendicular)

of 3 years. Ablative materials are not designed to take structural loads and as such mechanical properties do not form part of design specifications of ablative materials. However, it is desirable to have higher mechanical properties. The aging shows mixed effect on mechanical properties while modulus increased and ILSS decreased.

6 Results And Conclusions

This experimental work indicates that the properties of carbon-phenolic ablative composites are not greatly affected by aging for 3 years but showed some signs of deterioration. Moreover, higher heat of ablation and lower erosion rate was desirable for giving better ablative performance. Similarly, the results of ablative performance shows some reduction in its performance after third year, and the effect is more predominant for erosion rate. Also, low thermal diffusivity result from low thermal conductivity and higher specific heat is preferred for ablative applications which results in light thickness/ weight of the ablative liner. Thermal diffusivity shows an increasing trend after 1 year of aging which is mainly contributed by thermal conductivity and pecific heat both remain almost stable. However, the behaviour of thermal conductivity shows no particular trend which needs to be investigated further. These studied materials are not designed for structural loads and hence such mechanical

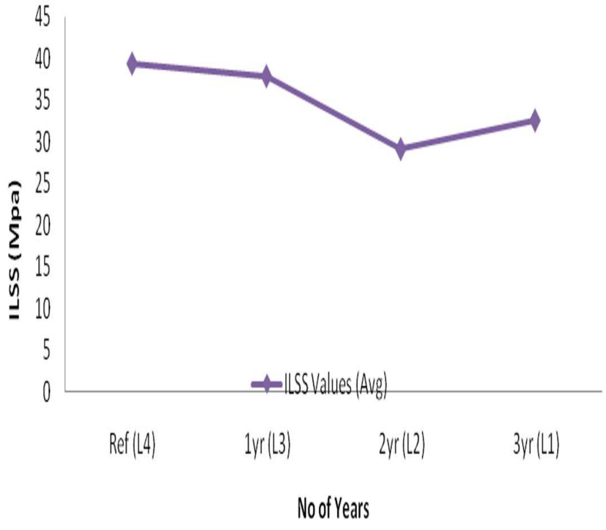

Fig. 12: ILSS Chart

properties do not form part of design specifications of ablative materials. However, it is desirable to have higher mechanical properties. Finally, it is observed from aging results showed that the combined effect on mechanical properties result the modulus increased and ILSS decreased. From various research it is further identified that accelerated aging studies on carbon phenolic for more than 8 years showed considerable variations which need more tests.

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