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EFFECT OF AUTOCLAVE CURE TEMPERATURE, PRESSURE, AND TIME ON THE GLASS TRANSITION TEMPERATURE AND THE DEGREE OF CURE OF EPOXY FILM ADHESIVE JOINTS

Nitesh Hirulkar, Shraddha Jagatap, Sayed A. Nassar

Fastening and Joining Research Institute (FAJRI)
Department of Mechanical Engineering
Oakland University, Rochester, Michigan 48309 U.S.A.

ABSTRACT

This study investigates the effect of autoclave curing variables on the glass transition temperature of and the degree of cure and strength of epoxy film adhesive single lap joints (SLJs) under static tensile shear loading. Studied autoclave variables include the cure temperature, cure pressure, temperature, and pressure ramp rates on the glass transition temperature as well as the cure time duration. Test joints are made of Aluminum substrates that are autoclave-bonded using epoxy film adhesive (AF163-2k). For each variable combination of the autoclave process, the corresponding glass transition temperature of cured Epoxy film adhesive is obtained using Dynamic Mechanical Analysis (DMA-Q800). Test data are generated for both baseline joints [uncycled] as well as for joints that have been heat-cycled in an environmental chamber after initial autoclave bonding. Results show a strong correlation between the autoclave process variable combinations and the corresponding glass transition temperature bond strength, and the failure mode of test joints.

1. INTRODUCTION

Recent increase in use of adhesive joints in structural applications is mainly motivated by the increased demand for light weighting, design optimization, and energy saving, as compared to conventional joining methods that use threaded fasteners, rivets, and weldments [1]. Recent advances in control methods and inspection have led to a significant increase the reliability and durability of adhesively bonded systems under complex service loads and severe environmental conditions including extreme temperature, water, and organic solvents [2].

However, moisture diffusion and humidity may still cause reversible or permanent changes to the adhesive (plasticization, swelling, hydrolysis) [3–5]. As a result, much work has been devoted to characterizing the strength of bonded joints. Extended exposure to moisture and humidity may also degrade the adhesion bond at the interface between the adhesive and joint substrate causing corrosion, cracking, and/or debonding, as pointed out by Bowditch et al. [6, 7].

Moisture may be absorbed by the adhesive in two different ways; namely, as free or bound water. Free water would occupy the free spaces in the adhesive layer, which causes adhesive plasticization. Bound water would form single or multiple hydrogen bonds with the adhesive polymer chain, which causes swelling of the adhesive, plasticization, and would consequently decrease the strength and glass transition temperature Tg. Hu et al. [8] studied different adhesives under extreme temperature environment. Their static test data showed that longer environmental exposure led to different failure modes. Da Silva et al. [9] studied effect of diffused moisture into the adhesive layer. Their results showed deterioration in bond strength after diffusion. Boubakri et al. [10] showed that aging temperature and water are the main reasons for adhesive degradation.

Strength of adhesively bonded structures depends on curing process parameters used during bonding. Nassar et al. [11,12] studied effect of cure temperature T, cure pressure P, temperature ramp rate Rt, pressure ramp rate Rp, cure time t on the strength of polycarbonate joint at room temperature and after environmental loading. Their result established relation between bonding process variables and their interactions on the strength of polyurethane film adhesive. This study investigates effect of bonding process variables on the glass transition temperature Tg that impacts the lap shear strength of the epoxy film adhesive joints.

Glass transition temperature Tg is one of the most important parameters of an epoxy polymeric system. Tg determines the temperature boundary of significant changes in the enthalpic, viscoelastic, dilatometric etc. properties of all glass-forming materials. Several cure monitoring techniques like Differential scanning calorimetry (DSC), rheology, dynamic mechanical analysis (DMA), and thermal gravimetric analysis (TGA) are used to characterize the thermo-physical and mechanical properties of an epoxy during cure. DSC is primarily used to observe phase transitions including the melting point and the glass transition temperature Tg as well as heat of reaction to measure degree of cure (DOC) before or after a cure cycle has been applied [13-17]. Another technique for material characterization is rheology which measures the viscosity, shear storage modulus, G′, which is a measure of the sample's elastic properties, and shear loss modulus, G′′, that is a measure of the sample's viscous properties [18-20]. Like rheology, DMA determines the viscoelastic properties of a material by applying a sinusoidal stress and measuring the strain in the material. DMA investigates the longitudinal moduli, E′ and E′′, and can be used to identify Tg as well as transitions associated with molecular motions [21]. This work uses DMA for Measurements of the glass transition temperature.

2. MATERIALS

The adhesives used in this work is AF 163-2K produced by 3 M Scotch-Weld. This is a film adhesive with a thermoplastic knit supporting carrier inside for bond-line thickness control. The film thickness of approximately 0.2mm in uncured state. This is a DGEBA based epoxy with an amine hardener. The reaction of Bisphenol A diglycidyl ether, DGEBA, (bisphenol A) and the amines is the typical network formation of epoxy resins, where epoxy and amine groups are linked.

TABLE 1. ADHESIVE PROPERTIES (SHEET FILM EPOXY ADHESIVE (AF163)

Property	Value
Tensile Strength (MPa)	45
Elongation @ break (%)	500
100% Modulus (MPa)	2
Specific Gravity	1.07
Hardness (Shore A)	80
Glass Transition Temperature (˚C)	-36

TABLE 2. SUBSTRATE MECHANICAL PROPERTIES (ALUMINUM 6061)

Property	Value
Tensile strength (MPa)	310
Yield strength (MPa)	276
Shear strength (MPa)	207
Elastic modulus (GPa)	68.9
Poisson's ratio	0.33
Elongation (%)	12-17

Aluminum alloy, Al 6061-T6, is the used as adherend. Joint surfaces are sanded using emery paper to eliminate variation due to surface roughness. Lab grade acetone is used for the removal of foreign particles from the substrate surface and Figure 1 shows 3d surface roughness inspection.

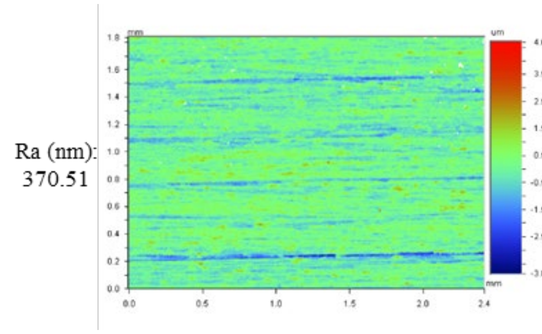


FIGURE 1. 3D SURFACE ROUGHNESS INSPECTION WITH RA = 370.51 NM

3. AUTOCLAVE PROCESS VARIABLES AND TEST PROCEDURE

In this study five autoclave process variables were investigated which include, cure temperature T, cure pressure P, temperature ramp rate R_t, pressure ramp rate R_p, cure time t. Table 3 illustrate selected two levels of five autoclave process variables. Full factorial experiment was designed to consider all 32-possible autoclave bonding process combinations with two levels of five process variables. For each autoclave bonding process combination six test coupons were bonded; each autoclave bonding process has a different combination of five autoclave process variables. MaGill Air Pressure LLC ™ Autoclave used for joint bonding. The autoclave is rated for a maximum pressure of 1.15 MPa and a maximum temperature of 204 °C with a vacuum pump that connects. to a vacuum bag, which encloses aluminum alloy adherends and adhesive film inside the vessel during the bonding cycle.

TABLE 3. AUTOCLAVE BONDING PROCESS VARIABLE

Autoclave Bonding Process Variable	2-level Autoclave Process Variables	
	Minimum	**Maximum**
Cure Pressure [MPa]	$P_1 = 0.345$	$P_2 = 0.689$
Cure Temperature [°C]	$T_1 = 93.33$	$T_2 = 126.67$
Temperature ramp rate[°C/min]	$R_{t1} = 1.11$	$R_{t2} = 3.89$
Pressure ramp rate [°C/min]	$R_{p1} = 0.069$	$R_{p2} = 0.103$
Cure time [minute]	$t_1 = 40$	$t_2 = 100$

4. EXPERIMENTAL PROCEDURE AND TEST SETUP

The dimensions and geometry of the specimens are shown in Fig. 2, Thirty-two (32) Al/Al single lap joints (SLJ) with different combinations of five autoclave processes variables at two (2) levels tested in tensile mode for to determine static strength. Six Al/Al SLJ were autoclave bonded for each 32-autoclave bonding process. From which three Al/Al SLJ were tested for baseline static strength at ambient conditions and three

Al/Al SLJ were tested after cyclic heat at high relative humidity (RH) of 85%. Average test data is reported.

4.1 Baseline Tests

Static tensile strength of Al-Al SLJ was evaluated using MTS 810 Material Testing System. SLJ were held inside specially designed fixture and gripped vertically on MTS system. A quasi-static axial load is applied to the coupon at a constant speed of 1.27 mm/min. Displacement and forces are recorded by MTS data acquisition system. Failure modes have been documented for all test joints.

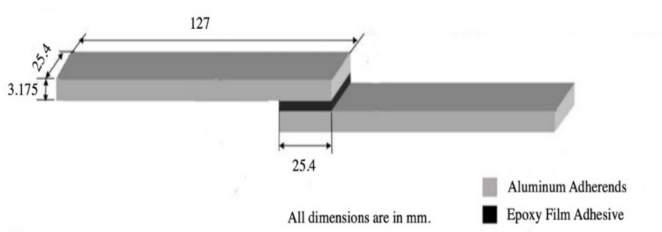


FIGURE 2. ILLUSTRATION OF SLJ

4.2 Environmental Cycling

Single lap joint (SLJ) of Al-Al tested for two different environmental conditions including ambient condition as baseline and cyclic heat at high relative humidity (RH) of 85%. Three SLJ coupons bonded from each of 32 different autoclave processes were transferred into an environmental chamber (Thermotron SE-300). The temperature cycles in the ranges of 20 – 75 °C for six 12-hours cycles following the profile illustrated in Figure 3

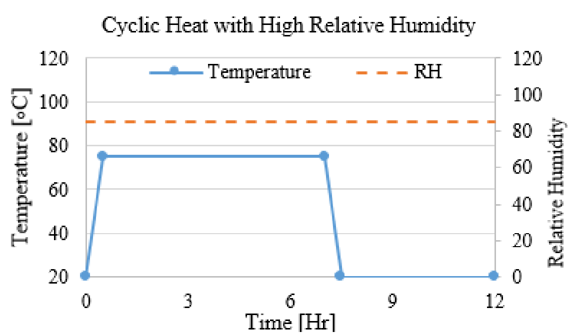


FIGURE 2. CYCLIC TEMPERATURE AND CONSTANT HUMIDITY PROFILE

5. MEASUREMENT OF GLASS TRANSITION TEMPERATURE T_g

Dynamic mechanical analysis (DMA) experiments were carried out in a TA Instruments Q800. Force and displacement calibration were done according to manufacturer recommendations. The complex Young's modulus E* of the cured AF163 epoxy film was measured using a DMA over the temperature range 20 to 170°C in an air environment with sinusoidal displacement. Measurements were at a fixed frequency of 1 Hz with a temperature ramp rate of 1°C/min. figure 5, demonstrate of T_g analysis with DME of ID 1 process parameters. Three samples for each of 32 different autoclave cure conditions were tested. The glass transition temperature was calculated as the peak of the tangent delta signal. Sample size was 10mm in width and 20 mm in length. As shown in figure 4, the fixture directly gripped the specimen approximately 5 mm from the end of the specimen length.

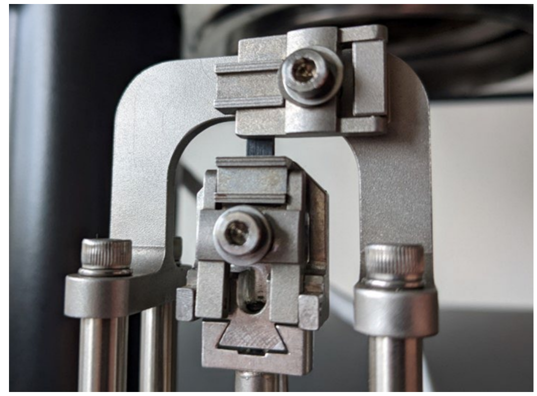


FIGURE 3. DYNAMIC MECHANICAL ANALYSIS (DMA)

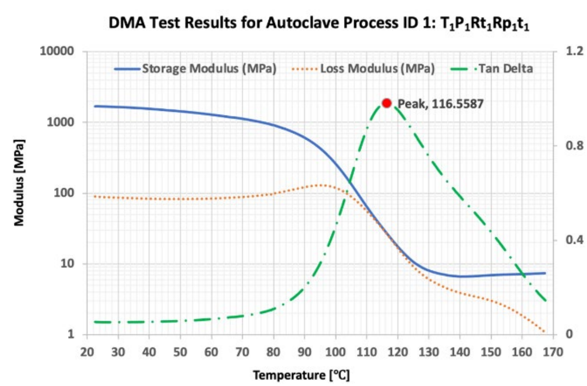


FIGURE 5. ILLUSTRATION OF TG ANALYSIS WITH DMA

5.1 Relationship Between Glass Transition Temperature T_g and Degree Of Cure α

The glass transition temperature T_g is one of the most important parameters of an epoxy system. T_g determines the temperature boundary of significant changes in the enthalpic, viscoelastic, dilatometric etc. The relationship between T_g and conversion degree in the curing reaction (α) of thermosetting materials is a central concept in analysing cure phenomena. The relevance of T_g to properties of material is based on the following a) processing of a reactive material must be performed at temperatures above the value of T_g of uncured material T_{g0}, b) the value of T_g at full conversion of a thermoset $T_{g\infty}$ is related to the upper temperature limit of the glass-converted material as shown in Figure 6. The relationship between T_g and $\boldsymbol{\alpha}$ determines when vitrification occurs during cure, for which T_g equals the cure temperature T_c. At a given T_c, the reaction of a thermosetting material proceeds generally at a rate dictated by chemical kinetics if T_g is less than T_c. However, if T_g is higher than T_c, the reaction rate is controlled by diffusion. The empirical DiBenedetto equation [14] gives relationship between T_g and a for a wide variety of thermosets,

$$\frac{T_g - T_{g0}}{T_{g\infty} - T_{g0}} = \frac{\lambda\alpha}{1-(1-\lambda)\alpha} \qquad (1)$$

$\lambda = \Delta C_{p\infty}/\Delta C_{p0}$(Structure-dependent parameter with value between 0-1. λ value for AF163-2K is 0.422 (from manufacture's specification). The T_g value of uncured epoxy film $T_{g0} = 28$ [°C] is obtained from tan delta curve of DMA test results. For T_g value at full conversion of an epoxy film ($T_{g\infty}$) series of DMA tests are carried out over cure temperature (T_c) range from 82.22 to 148.89 [°C] holding all other cure parameters constant. Highest value of T_g on the T_c vs T_g graph is considered as $T_{g\infty}$ = 154.44 [°C] as shown in Figure 7.

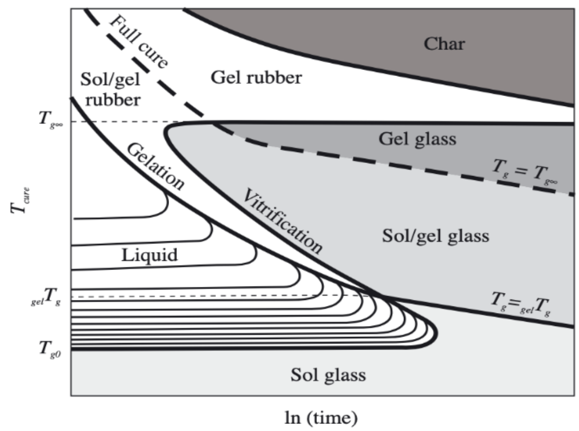


FIGURE 6. ISOTHERMAL TIME-TEMPERATURE-TRANSFORMATION (TTT) CURE DIAGRAM FOR AN EPOXY SYSTEM, SHOWING THREE CRITICAL VALUES OF TEMPERATURE (T_{G0}, ${}_{GEL}T_G$, AND $T_{G\infty}$) [22]

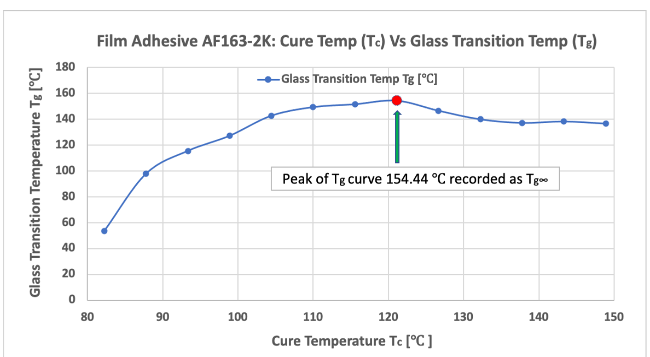


FIGURE 7. CURE TEMP T_C VS GLASS TRANSITION TEMPERATURE T_G FOR AF163-2K EPOXY FILM ADHESIVE

6. RESULTS AND DISCUSSION

In this section experimental results are presented and discussed. Figure 10 shows static strength and glass transition temperature (T_g) for all autoclave bonding process for ambient and after environmental loading.

6.1 Correlation Between Glass Transition Temperature and Static Strength

Figure 8 adds DMA test data of the glass transitions temperature T_g of the epoxy film adhesive to the corresponding joint strength for the same 32 combinations of autoclave variables. The trendline of the glass transition temperature T_g follows that of the joint static strength very closely. On the one hand, the highest glass transition temperature of T_g = 306.2 °C was attained by the combination of higher cure temperature T_2 = 126.67 °C, higher cure pressure P_2 = 0.689 MPa, lower cure time t_1 = 40 min, lower temperature ramp rate R_{t1} = 1.11 °C/min, and higher pressure ramp rate R_{p2} = 0.103 MPa/min. On the other hand, the lowest glass transition temperature T_g =197.2 °C corresponded to a lower cure temperature, cure time, and pressure (T_1 = 93.33 °C, t_1 = 40 min, P_1 = 0.345 MPa), and higher temperature and pressure ramp rates (R_{t2} = 3.89 °C/min, R_{p2} = 0.103 MPa/min). The glass transition temperature of Epoxy film adhesive (AF163-2k) is significantly affected by the cure temperature level.

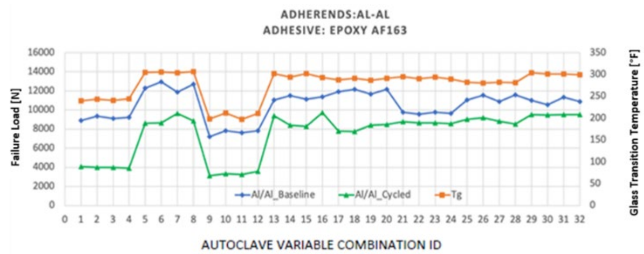


FIGURE 8. JOINT STRENGTH AND ADHESIVE GLASS TRANSITION TEMPERATURE DATA (FOR VARIOUS AUTOCLAVE VARIABLE COMBINATIONS FOR BASELINE TEST JOINTS AND AFTER HEAT CYCLING)

Raising the cure temperature T would increase the adhesive glass transition temperature T_g until it reaches its maximum when the Epoxy adhesive (AF163-2k) is fully cured. Beyond that, continued increase of the cure temperature or cure time would cause thermal degradation of polymeric chains of the adhesive, and accordingly reduce the strength epoxy film adhesive joints.

7. ANOVA ANALYSIS OF GLASS TRANSITION TEMPERATURE (Tg)

Based on ANOVA data in Table 4, the cure temperature (factor C), Cure time (factor E), interaction CE along with other factors and interactions found to be significant as P-value is less than 0.05 for 95% confidence. Normal plot (Fig. 9) shows the significance of factors and interactions.

Table 4 . ANOVA DATA FOR GLASS TRANSITION TEMPERATURE T_g

Source	Sum of Squares	DoF	Mean Square	F-value	p-value	VIF
Model	104928	31	3384.8	3769.85	0	
Cure Pressure [A]	13	1	12.8	14.2	0	1
Press. Ramp Rate [B]	4	1	4.2	4.64	0.035	1
Cure Temperature [C]	47972	1	47971.6	53429.1	0	1
Temp. Ramp Rate [D]	2741	1	2741.5	3053.33	0	1
Cure time [E]	19751	1	19751.1	21998.04	0	1
AB	2	1	2.5	2.75	0.102	1
AC	417	1	417.5	464.95	0	1
AD	3	1	2.7	2.97	0.09	1
AE	78	1	77.7	86.59	0	1
BC	5	1	4.7	5.22	0.026	1
BD	4	1	3.6	4.02	0.049	1
BE	1	1	1.4	1.51	0.224	1
CD	3344	1	3344.2	3724.7	0	1
CE	26740	1	26739.7	29781.77	0	1
DE	3174	1	3174.1	3535.22	0	1
ABC	0	1	0	0	0.984	1
ABD	0	1	0.2	0.27	0.608	1
ABE	0	1	0	0.02	0.898	1
ACD	63	1	63.4	70.6	0	1
ACE	67	1	66.7	74.23	0	1
ADE	4	1	3.6	4.02	0.049	1
BCD	0	1	0.3	0.39	0.535	1
BCE	2	1	1.9	2.08	0.154	1
BDE	1	1	0.5	0.6	0.44	1
CDE	257	1	257.4	286.74	0	1
Error	57	64				
Total	104986	95				
	R2		0.9995			
	Adjusted R2		0.9992			
	Predicted R2		0.9988			

The R-Squared shows accuracy of the model in predicting the response values or the measurement of the amount of variation around the mean; R-Squared value closer to 1.0 is desirable. In this analysis, the value is equal to 0.9995, which is good. Additionally, the difference between Predicted R^2 of 0.9988and Adjusted R^2 of 0.9992is less than 0.0004 which is favourable. In addition, Variance Inflation Factor (VIF) with value of 1 indicates an ideal correlation between the regression coefficients or how much the variance around the estimated coefficient is inflated by the lack of orthogonality, in which all the model terms are orthogonal to all other factors in this study. VIFs values more than 10 would raise alarm that present coefficients are poorly estimated because of multi-co-linearity.

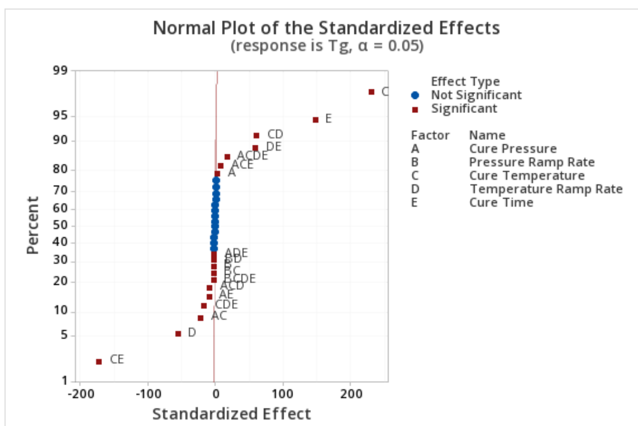


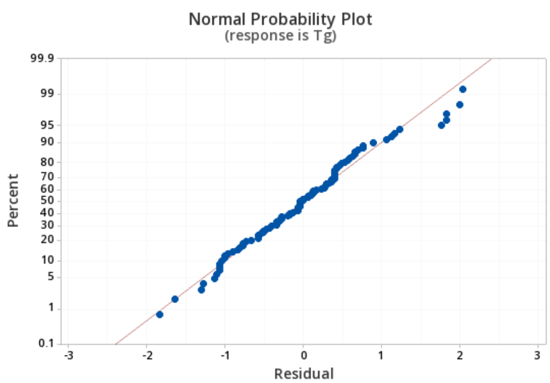
FIGURE 9. NORMAL PLOT FOR GLASS TRANSITION TEMPERATURE TG [°F]

An empirical model is presented in coded format to predict joint static strength based on the statistical analysis of test data. Response equation based on coded factors allows the prediction of response by assigning +1/–1 level to each independent process variable. In this study, the coded response is given by

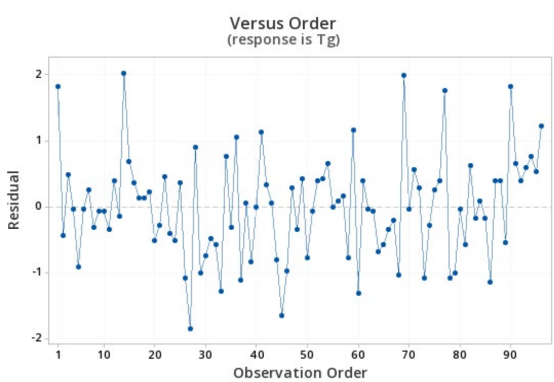
$$
\begin{aligned}
T_g\ (^\circ C) =\ & 276.688 + 0.3645{*}A - 0.2082{*}B + 22.3541{*}C \\
& - 5.3439{*}D + 14.3436{*}E + 0.1603{*}AB - 2.0853{*}AC \\
& - 0.1666{*}AD - 0.8999{*}AE - 0.2209{*}BC - 0.1939{*}BD \\
& - 0.1189{*}BE + 5.9022{*}CD - 16.6895{*}\ CE + 5.7501{*}DE \\
& - 0.0020{*}ABC - {*}0.0499{*}ABD - 0.0124{*}ABE - 0.8126{*}ABE \\
& + 0.8332{*}ACD - 0.1939{*}ADE - 0.060{*}\ BCD - 0.1395{*}BCE \\
& + 0.0751{*}BDE - 1.6376{*}CDE - 0.0251{*}ABCD \\
& + 0.0166{*}ABCE + 0.0895{*}ABDE + 1.6939{*}ACDE \\
& - 0.2334{*}BCDE + 0.1897{*}ABCDE \qquad \text{......(2)}
\end{aligned}
$$

Equation (2) gives the statistical prediction of the T_g (°C) of AF163-2k epoxy film adhesive that have not been heat cycled.

From Fig. 10 (A) can be observed that residuals follow a normal distribution, as a straight line is fitted through the normal plot of residuals. Fig. 10 (B) is a plot of the residuals versus the experimental run order, checks lurking variables that may have influenced the response during the analysis. Random scatter must be obvious; blocking and randomization of testing would minimize the probability of trends that may invalidate the analysis.



(A)



(B)

FIGURE 10. TG [°C] FOR AF163-2K EPOXY FILM ADHESIVE (A) NORMAL PLOT OF RESIDUALS (B) RESIDUAL VS. RUN

Fig. 11 shows single-variable effect on glass transition temperature T_g [°C]. Lines with positive slopes indicates that increasing that factor would increase joint static strength; the opposite is true as well. The value of the slope is a measure of how significant the factor is [21].

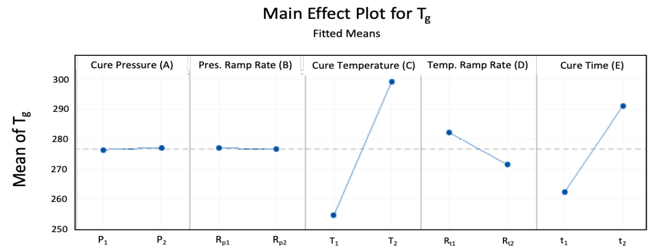


FIGURE 11. SINGLE VARIABLE EFFECT ON GLASS TRANSITION TEMPERATURE (T_g)

8. CONCLUSIONS

The effect of cure process variables on Static performance and glass transition temperature of autoclave cured epoxy film adhesive is investigated. The experimental study offers the following observations and conclusions.

High cure temperature (T_2 =126.67 °C) have positive effect on static strength and T_g as compared low cure temperature (T_1= 93.33 °C) when combined with cure time (t_1). But opposite effect was observed for higher cure time (t_2) process combined with high cure temperature. It's because with increase in cure temperature T_g continue to rise to almost linearly to reach vitrification.

It shows lower temperature ramp rate (R_{t1}) have positive effect on the static strength as compared with high cure temperature (R_t). R_{t1} ensures longer cure process and more mobility of monomers to increase cross linking and degree of cure ($\propto$).

High cure pressure (P_1) results into higher static strength, T_g when compared to low cure pressure (P_2). High cure pressure results in formation of intermolecular hydrogen bonding between hydroxy groups and carbonyl groups also increased the interaction of the polymer chains which improve the cross-linking densities and the mechanical properties of the cured adhesive.

Longer cure time (t_2 = 100 min) combined with lower cure temperature (T_1= 93.33 °C) increased the static strength of test joints. The static strength was lower for the cure variable combination of t_2, T_2, R_{t1} (longer cure time, higher cure temperature, and lower temperature ramp rate, respectively). The thermal degradation or oxidative crosslinking (above the $T_{g\infty}$ of the adhesive) is responsible for the decrease of strength with increasing curing time.

When below the T_{cure} at which the $T_{g\infty}$ is achieved, the static strength and degree of cure ($\propto$) of the adhesive increases as the cure temperature increases. When above the T_{cure} at which the $T_{g\infty}$ is achieved, there is an opposite behavior, i.e., both the strength and stiffness decrease as the cure temperature increases.

T_g and the mechanical properties have a similar behaviour. It was found that the T_g, static strength, and degree of cure ($\propto$) vary as a function of the cure temperature of the epoxy adhesive. When cured below the T_{cure} at which the $T_{g\infty}$ is achieved, the T_g, static strength, and degree of cure ($\propto$) increase as the cure temperature increases. When cured above T_{cure} (at which $T_{g\infty}$ is achieved), both the T_g, static strength, as well as the degree of cure $\propto$ are decreased by increasing the cure temperature T_{cure}.

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