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Significant Effect of Microwave Curing on Tensile Strength of Carbon Fiber Composites

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Abstract

The traditional process for curing carbon fiber (CF) composites has been the autoclave system. A review of recent research indicates curing CF composites in a microwave oven has the potential for reducing processing time. The problem statement of the research study was that the impact of a microwave curing process on tensile strength (maximum tensile stress) of selected CF composite specimens was unknown. The research study describes the statistical procedure and analysis of data to answer the specific question for the experimental trials: What is the significant effect on the tensile strength of cured CF composite samples due to the variables of the autoclave and microwave curing process cycle time and temperature? ASTM International standard test method designation *D 5083 – 02* was used for testing tensile strength of reinforced carbon fiber plastics using straight-sided specimens. Data was obtained for evaluating the effects of process cycle time and temperature on tensile strength of the CF composite specimens. The result was that curing time of the autoclave system and microwave process had significant effects on the tensile strength of CF composite specimens. The CF composite specimens from the microwave process showed lower tensile strength than the autoclave specimens due to greater void content.

Introduction

In 2005, U.S. EPA fuel economy regulations were increased to 27.5 mpg for passenger vehicles (Environmental Protection Agency [EPA], 2005). "Of critical importance will be the extent to which more than 200 million light vehicles on U.S. highways . . . become more fuel efficient as vehicle buyers choose the lower fuel costs of lighter or hybrid vehicles" (Greenspan, 2005).

One approach being followed by OEM automakers is to design vehicles that consume less fuel by using new materials like carbon fiber (CF) composites (Aronson, 1999). In aerospace, corporate jets are being produced using lightweight material to reduce weight and increase range (Sutton, 1998; Dornheim & Meacham, 2005).
The traditional method for curing CF composite material is the autoclave system with cycle time ranging as long as 8 – 10 hours and produces known tensile strength (Ashley, 1997, Dornheim & Meacham, 2005). The manufacturing process of carbon-fiber composites has the highest potential for the reduction of cycle time (Feher & Thumm, 2004). A faster processing method is the microwave system. According to Feher and Thumm, the microwave system is a better system with the potential benefit of reducing processing time (2004). The potential benefits of using the microwave system for curing CF composites include the reduction of processing time, energy consumption and lower operating costs.

The effects on the tensile strength (maximum tensile stress) of cured CF composite material are unknown when using a microwave curing system. Tensile strength is defined as the maximum tensile stress of reinforced thermosetting plastics (ASTM, 2002). Therefore, the problem statement of this research study was that the impact of the microwave curing process on tensile strength of selected CF composites was unknown. Experimental trials were completed to determine the effect.

Review of Literature

Conventional fabrication of carbon fiber composites is a slow, labor intensive process (Morey, 2007). The conventional process for curing carbon fiber composites utilizes an autoclave. Autoclave composite manufacturing begins by heating resin until it is liquefied. In the curing process, the liquefied resin impregnates a fabric form called a prepreg matrix (prepreg).

A specific number of prepregs are then stacked, or laid-up, to the required thickness. The laid-up prepregs are placed onto tooling that will form it into the desired shape. A plastic membrane is applied as a cover over the prepreg matrix and an adhesive is used to seal it to the tooling. The sealed prepreg and tooling are placed into the chamber of the autoclave for curing. A vacuum is applied to evacuate air from the plastic membrane forcing the prepreg matrix against the tooling walls to form its final shape.

Morey (2007) points out that a more innovative approach is the resin transfer molding (RTM) process. In RTM processing, a single three-dimensional fabric preform is woven in the shape of the finished part and placed in a mold. A vacuum is induced prior to injecting the resin into the mold to draw the resin through all of the spaces and voids of the preform. The final part can then be cured in the mold itself, or it can be transferred into an autoclave for final curing.

The aerospace industry has been the leader in utilizing an advanced RTM process for mass producing carbon fiber components (Ashley, 1997). The traditional curing process of RTM is the autoclave system, however, innovative methods are being applied to reduce cost (Morey, 2007). As the entire RTM preform assembly is heated, the resin is injected, then cured under high pressure. The preparation, lay-up and de-molding is a highly manual process. A vacuum assisted process (VAP) can reduce the manual labor process (Feher and Filsinger, 2005).

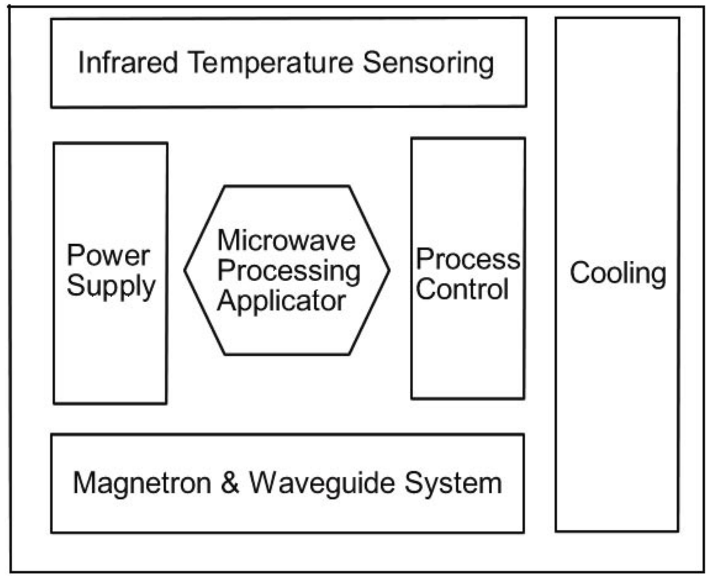

Figure 1. ***– Components of a microwave system.***

In an autoclave system, carbon fiber matrix or preform woven material is placed in a vacuum bag and mold. The autoclave processing method utilizes a vacuum and pressure level. Unlike an autoclave, the curing process in a microwave oven is accomplished by the distribution of magnetic-fields from microwave energy (Orbzut, 2006).

The basic components of a typical microwave system are shown in Figure 1. Power is supplied to the magnetron creating an electromagnetic (EM) field that is distributed by the waveguide system into the applicator. Temperature sensors are located at the magnetron generator and air inlets to monitor the temperature and prevent overheating. To prevent damage to the magnetron caused by reflective microwaves, industrial microwaves use a circulator to deflect electromagnetic waves into an absorber (Chan & Reader, 2000).

There are two basic components to propagating electromagnetic waves: (a) electric field (*E*), volts per meter (V/m), and (b) magnetic field (*H*), amperes per meter (A/m) (Ulaby, 2004). Two electrostatic charges *E* spaced apart in a vacuum are known as permittivity in free space, ε_0. Two current loops *H* spaced apart in a vacuum are described as permeability in free space, μ_0. The primary component to heating material in a microwave is the electric field of the electromagnetic wave (Akhtar, Feher & Thumm, 2006; Orbzut, 2006). If the space between the electrostatic charged particles is filled with dielectric material, the mechanical force between charges is increased by a factor known as relative permittivity of the material, ε' (Chan & Reader, 2000).

Although the electric field strength (*V*/*d*, volts per meter distance) over a rectangular conductive material is relatively constant (Ulaby, 2004), the geometric discontinuity of the edges and sharp corners increases the electric field strength, causing surface charges to accumulate (Meredith, 1998). This accumulation of electrostatic charges in the corners causes certain material to arc during microwave heating. This is one reason why material at the corners and edges heats faster (Pearce, 2005). Figure 2 shows the geometric discontinuity.

Using a microwave oven in the manufacturing process of carbon-fiber composites has the highest potential for reduction of cycle time and cost. As stated by Feher and Thumm (2004), "The highest potential for cost reduction is to be found [in] the manufacturing process which implies substantial long-time and high-energy consumption, as well as a low degree of automation" (p. 73). Potential benefits of using the microwave technology for fabrication of carbon fiber composites can include "high heating rates – reduction of processing time; savings on energy consumption; [and] 'clean' heating technology" (Feher & Thumm, 2004, p. 73).

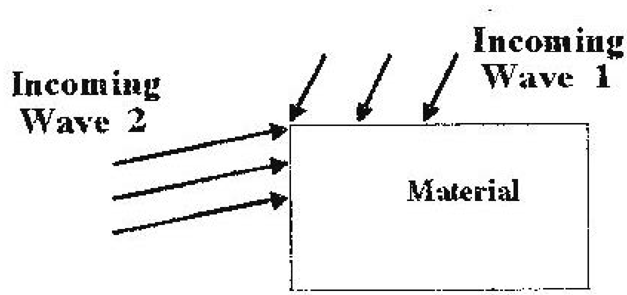

Figure 2. ***– Electromagnetic waves and geometric discontinuity.***

Method

The selection of the statistic, including the error rate, the statistical procedure and analysis of data was used to answer the specific question for the experimental research study: What is the effect of the autoclave and microwave curing process cycle times and temperatures on the tensile strength of cured CF composite samples? By completing experimental trials of CF composite samples using the commercial microwave curing system, analytical data was obtained for evaluating the effects of cycle time and temperature on tensile strength of the CF composite specimens.

Statement of Hypothesis

There were two process variables used as independent variables (IV) and one dependent variable (DV): The type of curing process (autoclave / microwave) was an IV with two factors: (a) cycle time, Factor *A*, was an IV, (b) temperature, Factor *B*, was an IV; and CF tensile strength (maximum tensile stress) was a DV. Factor *A*, cycle time, had two levels: (a) 180 minutes for the autoclave and (b) 30 minutes for the microwave. Factor *B* had two levels: (a) 250 ºF temperature and (b) 356 ºF temperature. The following states the hypothesis:

Null hypothesis 1 (H_{01}). $\mu A_1 = \mu A_2$, states that there is no difference in the mean measurement of autoclave curing process cycle time, 180 minutes, and microwave curing process cycle time, 30 minutes, on the effects of CF maximum tensile stress.

Null hypothesis 2 (H_{02}). $\mu B_1 = \mu B_2$, states that there is no difference in the mean measurement of microwave curing process temperatures, 250 & 356 degrees Fahrenheit, on the effects of CF maximum tensile stress.

The independent variable subjects were categorical factors with two levels for Factor *A*, cycle time, and two levels for Factor *B*, temperature. The dependent variable subject was an interval factor and random in nature, being representative of the population. The main effects were identified.

Curing time Factor *A* Level 1 was 180 minutes for the autoclave and Factor *A* Level 2 was 30 minutes for the microwave. Temperature was Factor *B* Level 1 for 250 degrees Fahrenheit and Factor *B* Level 2 for 356 degrees Fahrenheit. The autoclave had only Factor *B* Level 1 for 250 degrees Fahrenheit. The microwave had Factor *B* Level 1 for 250 degrees Fahrenheit and Factor *B* Level 2 for 356 degrees Fahrenheit.

Statement of Procedures

The research study used two different systems to cure the CF composite materials: autoclave and 2.45-GHz microwave oven. The equipment used for the autoclave study was the Hercules autoclave. The equipment used for the microwave study was the Amana 3 kW commercial 2.45 GHz microwave oven, as shown in Figure 3 (Amana, 2001). A digital temperature data recorder with fiber-optic probe was used for temperature sensing. Two universal tension test machines were used in the research study. Each machine was calibrated prior to testing. Experimental trials were completed using the CF prepreg variable temperature matrix product code VTM®264/CF0300 provided by Advanced Composites Group, Inc. It is a 2 X 2 woven CF pre-impregnated resin and precursor matrix.

Figure 3. ***2.45-GHz commercial microwave oven and temperature data recorder.***

Figure 4 shows the slab of prepreg matrix prior to curing. Preparation of the specimen was based on the Advanced Composites Group ([ACG], 2006) and Thomas and Kardos (1994) method for material preparation. There were eight layers in the slab to achieve a minimum 2 mm (0.079 in) thickness. Each corner of the slab was trimmed, as shown in Figure 4.

The ASTM International Standard known as *D 5083 – 02* standard test method for testing tensile strength of reinforced composite plastic was used as the reference guideline for preparing test specimens. The preferred specimen size was an overall length: > 250 mm (9.843 in); width: 25 mm +/- .5 mm (0.984 in +/- .020 in); and, thickness: between 2 mm and 14 mm (0.079 in and 0.551 in) (ASTM, 2002). After curing the slab, each specimen was cut into the correct rectangular dimension size, as shown in Figure 5.

For the research experimental trials, there were three trial runs with 30 CF composite specimens from each type of curing process, for a total of 90 specimens: (a) one trial run from the autoclave at 250 ºF, (b) one trial run from the microwave at 250 ºF, (c) and one trial run from the microwave process at 356 ºF.

Figure 4. ***– Trimmed slab with minimum 2 mm (0.079 in) thickness.***

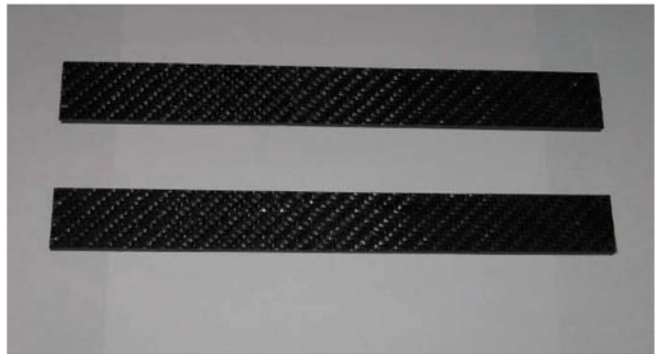

Figure 5. ***Cured carbon fiber composite specimens cut into 2.58 cm width and 25.4 cm length.***

The research procedure used (a) pre-heat (ramp-up) cycle and (b) cure (soak) cycle. The ramp-up time for the autoclave was 2 hours and for the microwave the time was 8 minutes. The cure cycle time in the autoclave was 1 hour and in the microwave the time was 22 minutes. For this research study, the amount of time to complete the combined pre-heat and cure cycles equaled the total curing cycle time.

Table 1 shows the microwave power setting and cycle time. Based on preliminary trials of required power output to fully cure CF composite specimens, the microwave power settings used to reach and sustain 250 ºF were as follows: (a) Setting 5 for 8 minutes, (b) Setting 0 for 4 minutes, (c) Setting 1 for 13 minutes, and (d) Setting 2 for 9 minutes. The power settings necessary to reach and sustain 356 ºF were: (a) Setting 5 for 9 minutes, (b) Setting 3 for 5 minutes, (c) Setting 1 for 6 minutes, and (d) Setting 2 for 10 minutes. The total power cycle time was 30 minutes for both temperatures.

Power settings of the microwave oven were pre-set by the manufacturer. Power Setting 5 is 50% of the available power of 3 kW, or 1.5 kW. Power Setting 3 is 30% (.9 kW), Setting 2 is 20% (.6 kW), and Setting 1 is 10% (.3 kW). Calibration of the microwave oven was achieved by using known data values for (a) specific heat and rate of temperature rise of water, (b) specific heat for the temperature rise and power density of water, and (c) time to boil water. Standard rules of thumb were followed for achieving specific heat and power density, 600 W – 800 W: (a) 4 minutes to boil 1 cup of water, (b) 6 minutes to

Table 1.
Microwave Power Settings and Duration

Temp./power	Curing cycle				
250 ºF	Stage	1	2	3	4
Power setting		5	0	1	2
Watts (kW)		1.5	0	0.3	0.6
Duration (min.)		8	4	13	9
356 ºF	Stage	1	2	3	4
Power setting		5	3	1	2
Watts (kW)		1.5	0.9	0.3	0.6
Duration (min.)		9	5	6	10

boil 2 cups of water, and (c) 10 minutes to boil 4 cups of water (Meredith, 1998; Dodson, 2001).

Selection of Statistic / Error Rate

The primary statistical procedure used was two-way analysis of variance (ANOVA). The general linear model (GLM) in SPSS 14.0 was used for analyzing data. The SPSS 14.0 software program includes an additional column labeled *Sig*. for significance (Norusis, 2005). Several assumptions were made for the two-way ANOVA statistical procedure in the study: (a) the dependent variable was measured on an interval scale; (b) samples were randomly selected from the population and randomly assigned to groups; (c) there was homogeneity of variance; and, (d) the error rate selected was .05 Type I error, ($\alpha = .05$).

Results

The descriptive statistics for the dependent variable are shown in Table 2. The mean value for maximum tensile stress for the autoclave curing time and temperature was 110.5 ksi.

The mean value for the microwave curing time at the two temperatures of 250 ºF and 356 ºF were the values of 67.2 ksi and 57.6 ksi, respectively. The standard deviation for the autoclave system mean measurement result was 3.82. The microwave process standard deviations were 3.14 at 250 ºF and 7.33 for the higher temperature of 356 ºF.

Statistics Analysis

Using the two-way ANOVA based on the SPSS 14.0 univariate GLM, the test of the between-subjects effects was run for the main effects as shown in Table 3. Curing time was statistically significant, $F(1, 87) = 1079.88$, $p = .00$. Temperature was statistically significant, $F(1, 87) = 53.08$, $p = .00$. Using a test of significance reference table from Mendenhall and Reinmuth (1978, p. 711, the F-critical value was 6.3 at 1 degree of freedom and alpha equals .05, which was the 95% confidence level. The F-observed value for curing time of 1079.88 with 1 degree of freedom, and temperature of 53.08 with 1 degree of freedom were greater than the F-critical value, 6.3. Based on analysis of the F-statistic values, curing time and temperature were significant at the 95% confidence level.

Table 2. *Descriptive Statistics for the Dependent Variable: Maximum Stress*

Curing time	Temperature	Mean (ksi)	SD	*N*
Level 1 (Autoclave)	Level 1 (250 ºF)	110.5	3.82	30
	Total	110.5	3.82	30
Level 2 (Microwave)	Level 1 (250 ºF)	67.2	3.14	30
	Level 2 (356 ºF)	57.6	7.33	30
	Total	62.4	7.38	60

Note. Dependent variable: maximum stress.

Table 3. *Test of Between-Subjects Effects for Main Effects*

Source	Type III sum of squares	df	Mean square	F	Sig.
Corrected Model	47498.4[a]	2	23749.2	914.92	.00
Intercept	423785.5	1	423785.5	16326.07	.00
Curing Time	28031.1	1	28031.1	1079.88	.00
Temperature	1377.9	1	1377.9	53.08	.00
Error	2258.3	87	23.0		
Total	603437.9	90			
Corrected Total	49756.7	89			

Note. Dependent variable: maximum stress.

Results from Hypothesis

The determination for rejection of a specific hypothesis was based on the significance level for the respective factor. When the F-observed value is greater than the F-critical value for a given variable, the difference between the variables is stated to be significant and the null hypothesis is rejected.

The difference in the mean measurement of curing time was statistically significant, $F(1, 87) = 1079.88$, $p = .00$. Therefore, the first null hypothesis of H_{01}: $\mu A_1 = \mu A_2$ was rejected. The alternative hypothesis of H_{A1}: $\mu A_1 \neq \mu A_2$, states that there is a difference in the mean measurement of curing time on the effects of CF maximum tensile stress. The mean measurement results for tensile strength of CF specimens from the autoclave system were larger values than the results from the microwave process.

The difference in the mean measurement of temperature was statistically significant, $F(1, 87) = 53.08$, $p = .00$. The second null hypothesis of H_{02}: $\mu B_1 = \mu B_2$ was rejected. The alternative hypothesis of H_{A2}: $\mu B_1 \neq \mu B_2$, states that there is a difference in the mean measurement of temperature on the effects of CF maximum tensile stress. The tensile strength results from the microwave process at 250 ºF were larger values than the results at 356 ºF.

Results of Fiber Matrix Density

Table 4 shows the fiber matrix density of autoclave specimens. The results showed variation in the thickness and fiber matrix density of CF specimens from the three different trials. The mean value for thickness of the CF specimens from the autoclave system was .22 cm (2.23 mm) and fiber matrix density was 1.96 g/cm^3. The mean value for thickness of the CF specimens from the microwave process at 250 ºF was .28 cm (2.77 mm) and fiber matrix density was 1.30 g/cm^3. The mean value for thickness of the CF specimens from the microwave process at 356 ºF was .30 cm (3.01 mm) and fiber matrix density was 1.12 g/cm^3.

Figure 6 shows the comparison of the measurement for maximum stress for CF composite specimens from the microwave process at the temperatures of 250 ºF and 356 ºF. When comparing the mean measurement data result from the microwave at 250 ºF with the mean measurement data result of the microwave at the higher temperature of 356 ºF, shows a higher maximum tensile strength in favor of the lower temperature setting.

Table 4. *Fiber Matrix Density of Specimens*

	Autoclave (250 ºF)		Microwave (250 ºF)		Microwave (356 ºF)	
No.	Thickness	Density	Thickness	Density	Thickness	Density
	(cm)	(g/cm^3)	(cm)	(g/cm^3)	(cm)	(g/cm^3)
1	.22	1.96	.27	1.45	.30	1.00
2	.22	2.19	.28	1.32	.30	1.05
3	.22	2.00	.28	1.29	.29	1.26
4	.23	1.78	.28	1.28	. 30	1.12
5	.22	1.89	.28	1.16	.30	1.15
Mean	.22	1.96	.28	1.30	.30	1.12

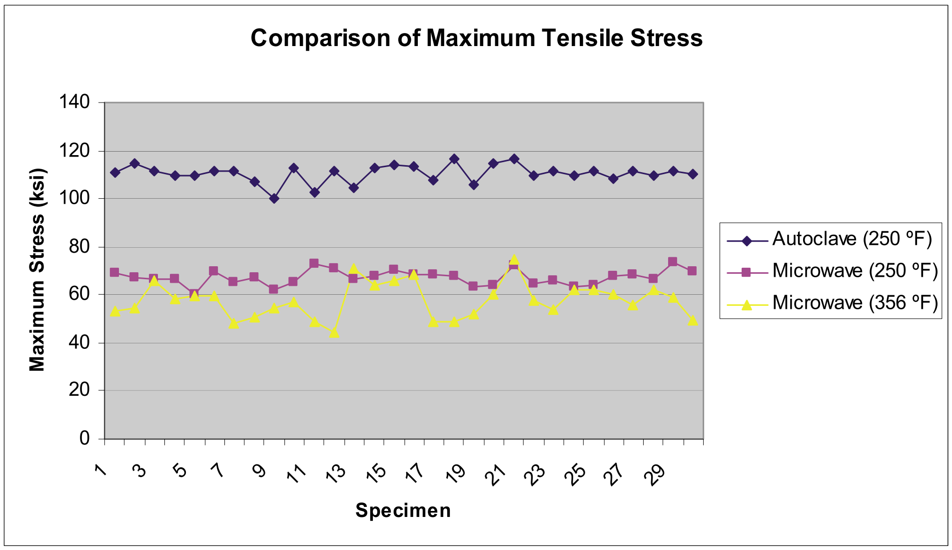

Figure 6. ***Comparison of maximum tensile stress. Actual data measurements are plotted.***

Discussion

There are several impacts of the microwave curing process on maximum tensile stress of CF composite specimens. One impact of the microwave curing process was that a 30 - minute curing cycle time has been validated to fully cure CF composite specimens compared to 180 minutes in the autoclave system. Another impact was that comparing the results of just the microwave process showed that using a 250 ºF temperature in the microwave curing process provided better tensile strength results than a 356 ºF temperature. Lastly, fiber matrix density is lower from the microwave curing process compared with the results of autoclave. The following discussion reviews the conclusions about the results of the experimental trials.

Conclusions

First, the results showed that the maximum tensile stress was higher for CF composite specimens from the autoclave system compared to the CF composite specimens from the microwave process. The results showed the mean measurement of the maximum stress for the autoclave CF composite specimens was 110.5 ksi. The mean measurement of the maximum stress for the microwave CF composite specimens at the same temperature was 67.2 ksi. The difference in mean measurement of maximum tensile stress was 43.3 ksi.

Second, the results showed that the mean measurement difference of temperatures was significant. The microwave process at the higher maximum temperature of 356 ºF had the lowest mean measurement for maximum tensile stress of 57.6 ksi. The microwave process at the lower temperature of 250 ºF had a mean measurement of maximum tensile stress of 67.2 ksi, which is a difference of 9.6 ksi. The standard deviation for the lower microwave process temperature specimens was 3.14 compared to the standard deviation for higher microwave process temperature specimens at 7.33. A smaller standard deviation indicates that the data distribution is narrow. A narrower distribution of data suggests that the lower temperature microwave process has a higher probability of producing specimens with higher tensile strength, when compared to specimens from the microwave process at a higher temperature.

Third, arcing occurred on the outer edges and corners of the CF prepreg slab in the microwave process. Arcing caused the loss of vacuum by burning a hole in the vacuum bag. In this study, electrostatic charges accumulated at the corners and exposed edges of the CF prepreg slab which resulted in arcing (Meredith, 1998). As a result of this occurrence, it was necessary to eliminate the resin absorbing cloth and vacuum bag from the microwave curing process procedure. Figure 7 shows the autoclave and microwave specimens. Lack of vacuum in the microwave process allowed greater expansion of moisture and evaporation of resin. The result was larger voids in the microwave CF composite specimens, Larger voids increased the thickness and reduced the fiber matrix density and tensile strength of the microwave CF composite specimens.

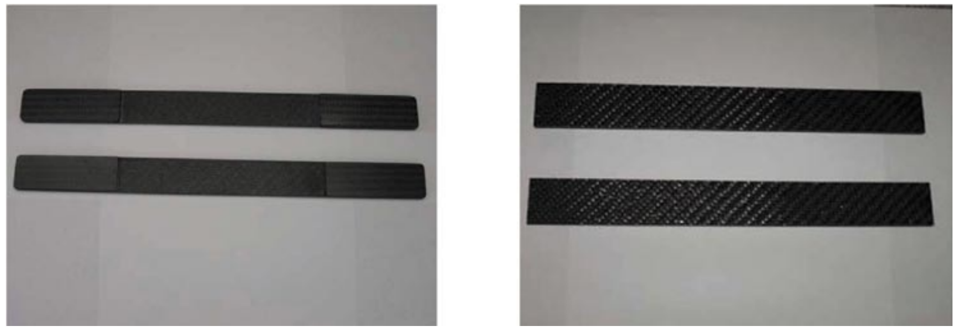

Figure 7. ***Specimens' avg. thickness - left: autoclave (2.23 mm); right: microwave (2.77 mm).***

Lastly, curing time for the autoclave system was 180 minutes compared to 30 minutes in the microwave process. Although the maximum tensile stress results were lower, the microwave curing process was 83% faster than the autoclave system.

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