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The Effects of Post Curing Process on the Physical Properties of Glass Fiber/Phenol –Formaldehyde Molded Composites

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ABSTRACT

The effect of post curing on two important physical properties of a Glass/Phenol-Formaldehyde (Novolac) composite is investigated. Samples are molded and post cured in different procedures. The Their Tg are measured using DMA test. The weight loss of sampled during post curing are determined as well. Result suggest that the Post curing has a significant effect on TG of thermoset resin and increases it considerably. This ,in turn, will raise thermal resistance and dimensional stability of the composite.

KEYWORDS: Post Curing, Glass / phenol –formaldehyde composite,Novolacresin , Tg,Tg_0

1. INTRODUCTION

For thermo set polymers, Which are used to manufacture various ports in different applications, Achieving a particular cross linking density for the polymer structure is very important and The molding or shaping system is designed to be able to control this density and stop the cross linking process in the desirable degree. However, in some ports such as hard matrixes in glass reinforces composites, achieving the highest possible cross linking density is required to maximize the strength of the matrix and consequent strength of the composite.

The most important ways for increasing the cross linking density of Novolac resin as a matrix, are increasing the molding temperature, molding time and post curing the part. Post curing is a process in which the molded part is exposed to extra heat after molding, in an atmospheric pressured chamber, for a particular time and designed heating procedure. In Polymer industries, post curing is a normal process for novolac resin parts and composites which is done after molding for various reasons.

one of the most important effects of post curing on structure of the novolac resin is to raise its Tg .Tg is the temperature in which the chains of polymer receive the enough energy to move and change their shape and position. As a result, polymer mass in Tg starts to soften and convert to a very high viscosity liquid. For thermoset plastics, Tg exist, but cross bonds do not allow chains to move. Therefore, the polymer mass does loose its dimensions.

In temperatures beneath Tg, The physical and mechanical properties of the polymer is on their highest .In addition, it's dimensional stability is very good. By contrast, the mechanical properties and dimensional stability of polymer is deteriorated in the temperatures above Tg.

For Novolac made parts or composites as a thermoset polymer mass, although the polymer maintain its dimensions and does not fail, but witnesses a considerable fall in its mechanical properties. In thermoset polymers, Tg is a function of the density of cross links in the network structure and the effect of any factor that increase the density of these links, will raise the Tg.

In the past, it was deemed that the Tg of thermoset polymers like Novolac is not important and does not have a great influence on the properties of resin as a matrix in a composite. However, the recent research has shown that The Tg of such matrixes have a considerable influence on its mechanical properties. Thus, determination of the Tg for thermoset composite matrixes is an important factor for designing the parts made from these composites that will be exposed to intensive Static and dynamic forces in their service conditions. It is also is used for predicting the behavior of the matrix in high temperatures.

Landi and Merserou(1) have reported the temperature of 51 degrees as the Tg of un cross linked Novolacresin using DSC test. During molding, the Tg of Novolac is in the range of 160 to 188 centigrade degrees which is almost equal to the temperature of molding. After molding, The Tg temperature of the cures Novolac can be increased to 290 degrees. There are two important tests for determining the Tg temperature of cured Novolacresin, which are DSC and TMA tests. In addition, the DSC result normally shows crucial information about the degree of cross linking process during Curing of the resin.

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In TMA test, the dimensional change of the samples shape is considered. In this test, the slope of the coefficient of thermal expansion curve of the mass is monitored. In Tg span of temperature, this slope changes and continue its increase with more rapid rate. One normal method for determination of Tg is distinguishing the temperature in which the sign of the Tanδ is reversed. There is another important point on the curve that the changes of thermal expansion coefficient show a slight non linear behavior. This temperature is known as TG0 or the initial glass transition temperture.Tg0 is normally 35 degrees less than TG and is the highest safe temperature for the sample that can tolerate and doesn't fail.

Another test for determination of the Tg is DMA test. In DMA, the vibration resonance as a function of temperature is monitored in order to determine Tg, Module and Tanδ .Test is used by Landi and Merserou for studying the thermal behavior of Novolacresin. From their result, it can be understood that determining TG using DMA and DSC curves are difficult. But by considering TG_0 as an indicator of TG and correcting it to TG, the Tg temperature can be determined.

concerning the above information, the effects of Post curing process on physical properties of the Novolac molded part can be summarized as following:

1- Increasing the Tg temperature of the cured resin in order to maximize its thermal strength.

2- Shrink the dimensions and volume of the part. This effect can have harmful consequences for the mechanical properties of the part, because it makes some remained and locked stresses in the mass of the matrix, especially in the parts that have variation s in thickness or in the sharp shaped areas of the part.

3- Extracting the small molecules which are trapped in the mass of matrix during shaping processes like hand lay up or during the curing process. These small molecules are H2O and NH3 which comes from Hexamine. Some molecules from solvents which are used in shaping processes also can remain in the mass and needs to be taken out in post curing.

Designing the post curing process

There are many methods which are used in the literature and patents for post curing process. They can be categorized to 3 main patterns: Post curing in constant temperature, post curing by step increasing temperatures and post curing in gradual increasing temperature.

The Maximum temperature which is reported in post curing of Novolac composite matrixes is 280 C and rarely more temperature is used for this purpose.

The duration of post curing is designed based on the thickness of the thickest part of the part, In that the desired temperature can reach the most intimate chains and increase their Tg.

In Hybrid composites in which the layers of glass and carbon fiber reinforced composites are used, post curing may course remained stress due to the different thermal expansion coefficient of these two fibers.

For the composite structures which contain metal parts, the post curing may also cause residual stresses. Furthermore, the post curing may change the properties of aluminum parts.

Experimental section

MATERIALS AND METHODS

Composite samples The Novolac resin of IP-500, purchased from Resitan company of Iran was used as the matrix for the composites. E-glass grade Glass mat was used as the reinforcement.

The reinforcement fabric was dipped in the resin solved in Ethanol and dried in oven in 60 C for one hour the sheets were molded in a compression molding press and cured in the mold for one hour in 160 C.

The samples was prepared in the dimensions of 16*8* Cm *7mm for TMA and mass loss and with the dimensions of 2*8* Cm *2mm for DMA tests. The DMA test machine used for the test was a DMA model 983 made by Du Pont company.

Post Curing process

For the post curing of samples, three patterns were used. A set of samples was post cured in step increasing temperatures for equal time, they were post cured in 75 C (2 hours), 95 C (2 hours), 130 C (2 hours), 160 C (2 hours), 200 C (2 hours) for the total time of 10 hours.

The other two set of samples were post cured in two temperatures of 160 and 200 C in different times. After post curing, DMA spectrum of the samples were prepared with the heating rate of 10 C/Min .

RESULTS AND DISCUSSION

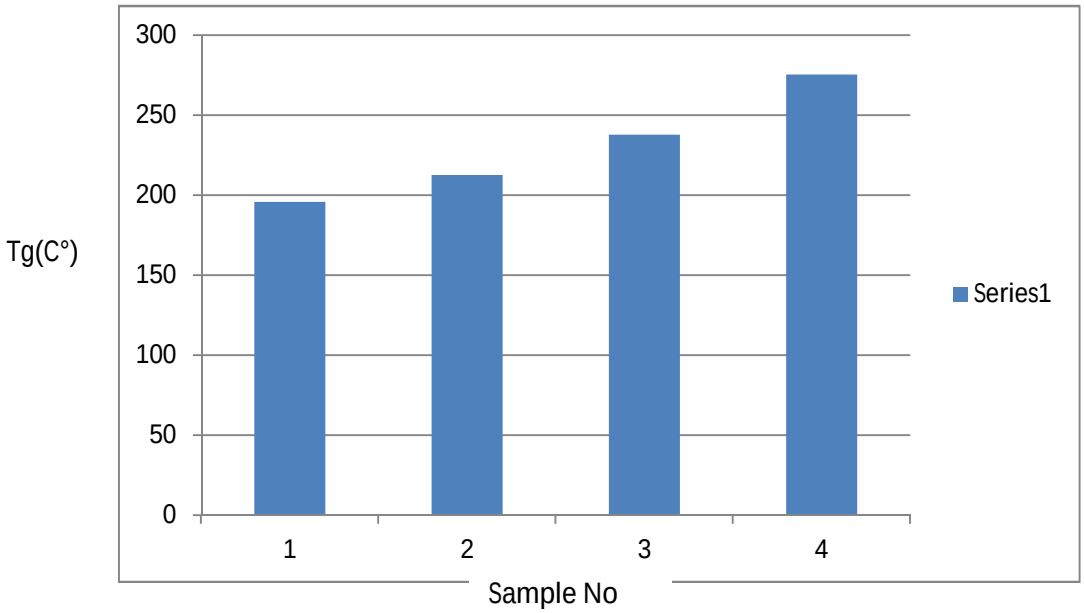
1- The effect of post curing process on the increase of Tg in composites with Novolac cured matrix

DMA curves were used for measuring the Tg of post cured samples comparing to not post cured samples. All samples were taken from one molded sheet to make sure that the degree of cross linking of all is the same. The results are given in the table 1:

Table 1. The Tg of samples with different post curing history:

Sample no	Composite structure	The reinforcement mass percentage in the sample	The post curing procedure	Tg temperature besed on DMA curve(C)
1	Novolac/E-Glass matt	51.549	Not post cured	196.1
2	Novolac/E-Glass matt	51.211	2 hours,200 C	212.7
3	Novolac/E-Glass matt	49.61	10 hours,200 C	237.9
4	Novolac/E-Glass matt	49.111	75,95,130,160,200C,2 hours each,10 hours totally	275.8

Bar Chart 1- The Tg of samples with different post curing history:



2- The effect of post curing on mass loss of composites with Novolac cured matrix

In these series of tests, samples were weighted before and after post curing .the results are indicated in table2:

Table 2. The mass loss of samples with different post curing history:

Sample no	Composite structure	The reinforcement mass percentage in the sample	The post curing procedure	Tg temperature besed on DMA curve(C)
1	Novolac/E-Glass matt	51.549	2 hours,160 C	1.895
2	Novolac/E-Glass matt	51.911	2 hours,200 C	2.123
3	Novolac/E-Glass matt	50.546	5 hours,200 C	2.745
4	Novolac/E-Glass matt	49.973	10 hours,200 C	3.222
5	Novolac/E-Glass matt	49.973	24 hours,200 C	3.694
6	Novolac/E-Glass matt	49.973	2 hours,230 C	3.152
7	Novolac/E-Glass matt	49.973	75,95,130,160,200C,2 hours each,10 hours totally	3.368

CONCLUSION

The results clearly show that the post curing process has an significant effect on the Tg of Novolac resins and increase it dramatically. This is because of the increase of the cross links density in the network structure of the thermoset resin.

The results suggest that post curing process improve the physical properties of the cured resin and extracts the small molecules which are trapped in the composite layers. This, in turn, decreases the possible fail of the composite in high temperatures because of the voids and bubbles which can be created by these substances.

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