Heat and Time Effect on Properties of Polycarbonate Composites

Chalermchai Chaitongrat[*1], Somsak Siwadamrongpong[2]

[1, 2] Department of Mechanical Engineering, Faculty of Engineering, Suranaree University of Technology, Nakhonratchasima, Thailand 30000

* Corresponding Author: Tel: 044 224 236, Fax: 044 224 613

E-mail: somsaksi@sut.ac.th

Abstract

Composite of polycarbonate is widely used in various applications as parts and products, such as parts in electronics industries. It is know that heat usually affects in deterioration trend to polycarbonate composite properties. The deterioration may be resulted from changing of its structure and yield to shorter life time. However, mechanism of heat effect to life time of part or product is still unclear. Therefore, aim of this study was to study effect of temperature and time to properties of polycarbonate composite. Polycarbonate composite specimen was annealed at 75 90 105 120 degree Celsius for 1/4 3/4 4 8 12 and 24 hours. It was found that increasing of treatment time and temperature results to changing of the polycarbonate composite from ductile to brittle material. From thermal analysis, it was found that treatment time and temperature seem to be not affecting to the polycarbonate structure and composition change. It was proposed that heat may result the interdiffusion of polymer matrix and lead to more order of its structure.

Keywords: Polycarbonate, Composite Material, Heat & Time effect, Elongation, Glass transition Temperature

1. Introduction

It is well known that Polycarbonate (PC) is polymer that has good thermal resistance, high strength and good wear resistance (Fang and Ming, 2007). PC composite is widely used in various industries as parts and products, such as parts in electronics industries. Generally, heat will affect the alignment of the chain and crystallinity of PC composite which led to changes in physical properties. Consequently, it is significantly affect to performance and lifetime of parts. However, mechanism of heat effect to lifetime of part or product made from PC is still unclear.

The purpose of this study was to study effects of temperature and time to properties of PC composite specimen.

2. Experiment

2.1 Experimental plan

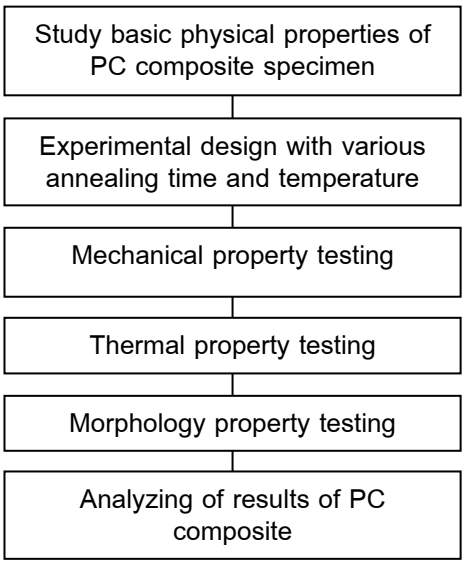


Fig. 1 Operating plan

Figure 1 shows the experimental overview of this study. The specimen was annealed at various temperature and time. The physical, mechanical, thermal and morphology properties of the specimen were investigated. The effect of temperature and time to PC composite was analyzed and mechanism of property change will be discussed.

2.2 Polycarbonate composite specimen

This study used samples formed by injection followed to American Society for Testing and Materials ASTM D638 (ASTM, 2004), dumbbell shaped specimen. The specimen of specified geometry and size shows in figure 2, length 220 ± 0.4 mm, width 12.5 ±0.4 mm, thickness 3.2 ± 0.4 mm.

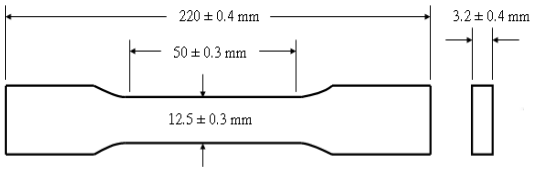


Fig. 2 PC composite specimen

2.3 Thermal annealing

Specimens were annealing at various temperature and time using oven Memmert Model UFE 600. The annealing temperatures and time are at 75 90 105 and 120°C for 1/4 3/4 4 8 12 and 24 hours, respectively, as shown in table 1.

Table. 1 The temperature and duration time used for annealing PC composite specimen

Temperature, T_b (°C)	Duration, t_{ag} (hr)
75	1/4, 3/4, 4, 8, 12, 24
90	1/4, 3/4, 4, 8, 12, 24
105	1/4, 3/4, 4, 8, 12, 24
120	1/4, 3/4, 4, 8, 12, 24

2.4 Mechanical property testing

Mechanical properties were tested by dumbbell shaped PC composite specimen that shown in figure 2. The testing device was Instron Universal Testing Machine 5560 with load cell 5 kN and crosshead speed of 50 mm/min (Constant rate of extension) at room temperature 25°C (Medhat; et. al. 2010) as shown in Figure 3. The testing was conformed to standard test methods for properties in tension of the plastic (ASTM: D638-03). 3 specimens were test for each condition. Maximum tensile stress and elongation of the PC composite specimens were analyzed.

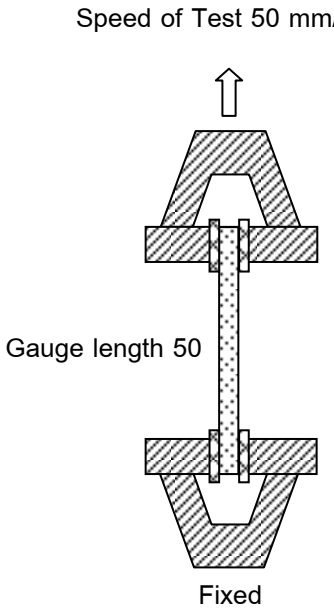


Fig. 3 Schematic diagram of tensile test for mechanical properties of PC specimen

2.5 Differential Scanning Calorimeter (DSC)

The thermal properties were determined using a Mettler Toledo 822^e differential scanning calorimeter (DSC). The annealed PC composite samples was cut into a small beads about 8 mg, tested at a scan rate of 20°C/min from the second heating in the range of 50-330°C under nitrogen atmosphere (Yiging and David, 2005). Glass transition temperature (T_g) of the specimens were analyzed.

2.6 Thermal Gravimetric Analysis (TGA)

Thermal gravimetric analysis (TGA) was performed with Mettler Toledo TGA / SDTA 851°. The annealed PC composite samples about 14 mg were tested at heat rate of 20°C/min and the temperature range from 50-850°C under a nitrogen atmosphere (Yiging and David, 2005). The components in the annealed specimens were analyzed.

2.7 Morphological characterization

The replica of fracture surface of PC composite specimens was performed with Scanning Electron Microscope (SEM) JSM-6400 (JEOL, Tokyo, Japan) with an acceleration voltage of 10 kV. The sample was cooled in liquid nitrogen and then breaks it rapidly. The fracture surfaces were vacuum coated with gold for SEM testing (Ning; et. al. 2008).

3. Results and discussion

3.1 Mechanical properties

Figure 4 shows the stress-strain curves of the PC composite specimen annealed at 120°C for 1/4 3/4 4 8 12 and 24 hours compared with the specimen that does not annealed as the reference.

Figure 4 demonstrate that the reference specimen indicates the unique characteristics of flexible polymer material. It was found that the annealed PC composite

shows significantly low elongation with little higher maximum tensile stress compared

Baking at T_b = 120°C

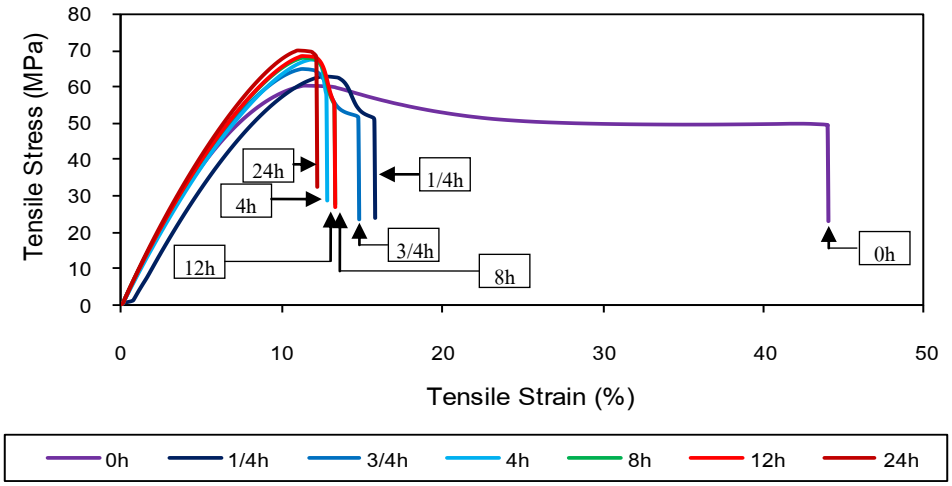


Fig. 4 Stress-strain curve of PC specimen at annealing temperature T_b=120°C, t_{ag}=0-24 hr.

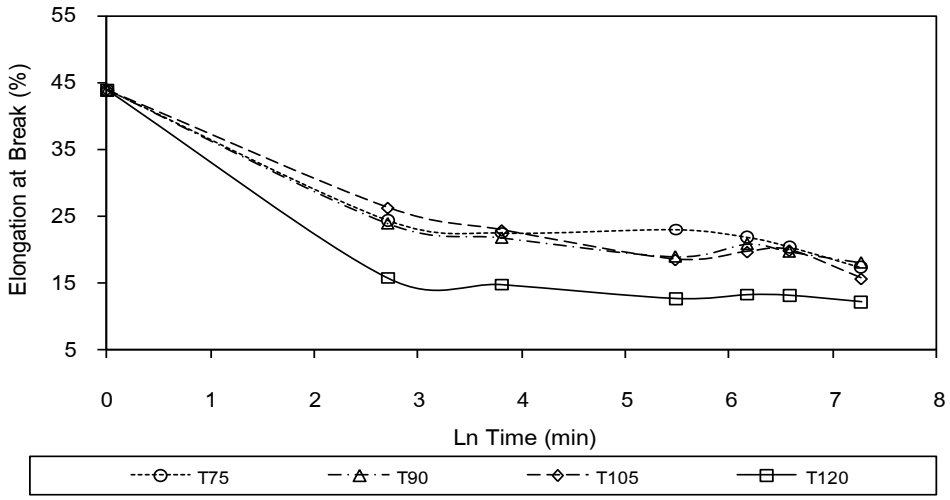


Fig. 5 Relation between elongation and duration of annealing temperature T_b=75-120°C, t_{ag}=0-24

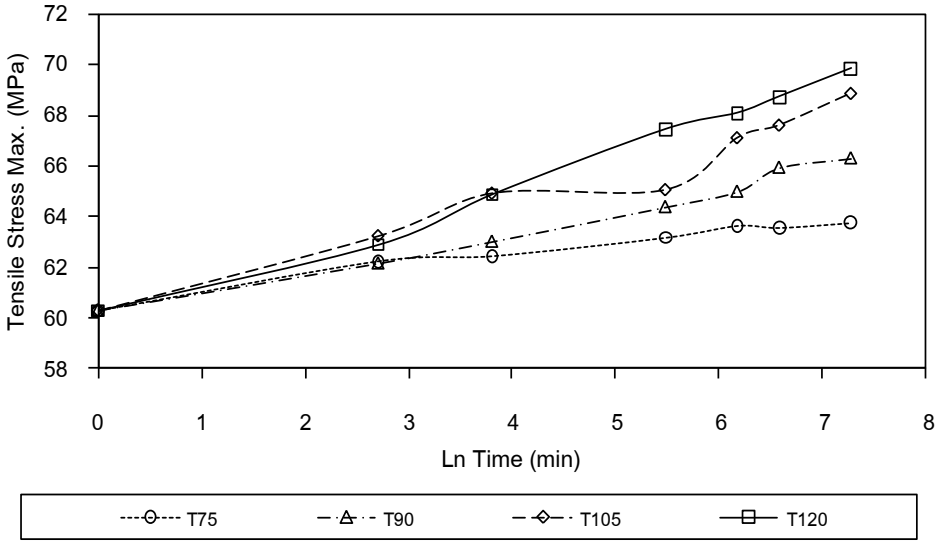


Fig. 6 Relation between maximum stress and duration of annealing T_b=75-120°C, t_{ag}=0-24 hr.

to the reference specimen. It implies that heat might affect mechanical properties of PC composite even the shortest annealed time for 1/4 hours. The same result trend can also be found in the rest annealed temperature. Figure 5 shows that Elongation was significantly dropped by heat from annealing and slightly decreased with longer annealing time. Figure 6 indicates that maximum tensile stress increased with increasing annealing time at all temperatures. The heat effects result to changing of the polycarbonate composite from ductile to brittle material.

From these results, it could be discussed that an increasing of annealing time and temperature yields the higher of maximum tensile stress and significant decrease of elongation (Medhat; et. al. 2010). On the other word, an increasing of annealing time and temperature results to changing of the polycarbonate composite from ductile to brittle material. However, the slope (Young's modulus) of stress-strain curve, shown in Figure 4, of each specimen could be considered the same. This may result from changes in either polymer chain type and length to be more strengthening or from the degradation of the components in the PC composite sample. These assumptions will be tested and discussed later.

3.2 Thermal properties from DSC

Figure 7 shows the heat flow from DSC of PC composite specimens with annealing temperature at 120°C for 4 8 12 and 24 hours compared with the reference specimen.

Figure 7 shows that the positions of the change of endothermic slope during heating up were located at about 146°C (Bo; et. al. 2006). The point of slope changing represents the important characteristics of polymer so call Glass Transition Temperature, T_g. Table 2 shows T_g of the specimen annealed at 120°C. It was found that T_g of specimens do not show significantly change under influence of annealing time. This result implies that the PC composite structure chain should be considered as no significant change or transform.

Fig. 7 DSC tested results of PC specimen at annealing temperature T_b=120°C, t_{ag}=0-24 hr.

Table. 2 T_g of specimens of various annealing time tested by DSC

Duration baking; t_{ag}, hr.	Temperature; T_g, °C
0	146.37
4	145.59
8	146.22
12	145.89
24	146.24
Mean	146.06

3.3 Thermal properties from TGA

Figure 8 shows the TGA curves of weight loss as elevated temperature. From the results,

the weight lost during temperature below 400°C can be considered mainly due to the decomposition of moisture. The weight loss in temperature range 480-540°C is considered as the decomposition of compound 1, equivalent to the weight loss of about 74 percent. Then, the weight loss in the temperature range 610-710°C is considered due to the decomposition of compound 2 equivalent to the weight loss of about 25 percent as shown in table 3.

From the results of the decomposition of 2 compounds, it can be analyzed and concluded that the first compound is polycarbonate (Bok and Charles, 2004) and the second compound is considered as Carbon Black (TA Instruments, n.d.). Figure 8 also shows the weight changes to the temperature of the PC specimen annealed at 120°C for 0 4 8 12 and 24 hours. It was found that the temperature range and percent weight change of all specimens could be considered the same. This result indicates that composition and compound type in all specimens do not change by heat from annealing temperature and time.

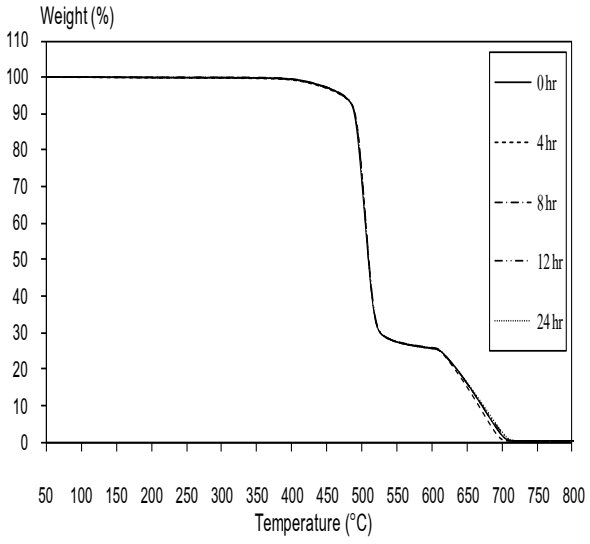


Fig. 8 TGA curve of PC specimen at Tb=120°C, tag=0-24 hr.

Table. 3 Decomposition percentages by TGA

Duration, hr.	Composite 1, %	Composite 2, %
0	74.15	25.30
4	74.27	25.37
8	73.97	25.50
12	74.16	25.42
24	74.30	25.37
Mean	74.17	25.39

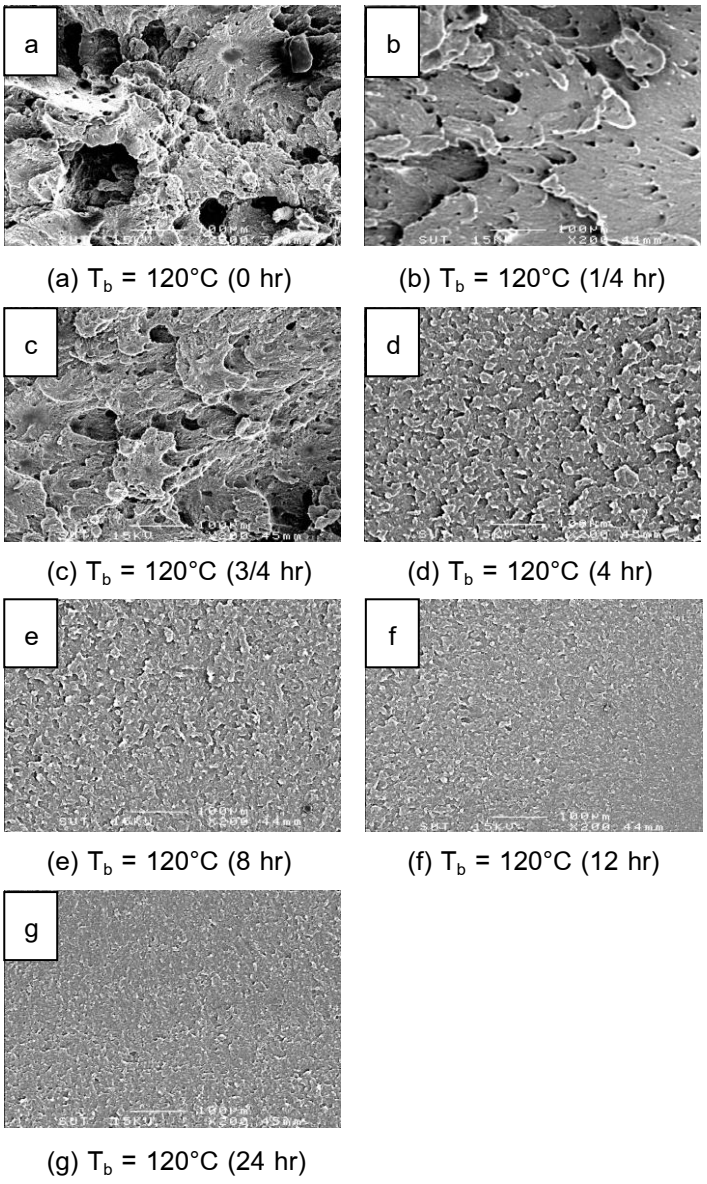
3.4 Morphological study

Figure 9 shows the SEM photomicrograph of fracture surface of PC composite specimen at annealing temperature of 120°C for 1/4 3/4 4 8 12 and 24 hours at 200x magnifications.

Figure 9a shows that the fracture surface of the reference specimens was rough with many tear lines and branching. This type of failure indicates the failure of ductile material that conforms to the results of mechanical property testing. Figure 9b for the specimen at 1/4 hours annealing. Although the fracture surface also shows some matrix tearing and some branching, the failure surface also indicates more percentage of flat and smooth surfaces.

Figure 9c-9g also illustrate the same trend of failure surface as mention above, an increasing of more percentage of flat and

smooth surfaces as increasing of annealing time. It could be discussed and concluded that the microstructure of the specimen becomes finer and more homogenous as increasing of annealing time. This phenomena also found in another works related to polycarbonate (Medhat; et. al. 2010). The mechanism of this change could be considered due to thermal energy from annealing which causes the increase in mobility of the molecules (G. S. Sekhon; et. al. 2008).



(a) T_b = 120°C (0 hr) (b) T_b = 120°C (1/4 hr)

(c) T_b = 120°C (3/4 hr) (d) T_b = 120°C (4 hr)

(e) T_b = 120°C (8 hr) (f) T_b = 120°C (12 hr)

(g) T_b = 120°C (24 hr)

Fig. 9 The SEM photographs on surface of PC specimen (a-g) at baking T_b=120°C, t_{ag}=0-24 hr.

4. Conclusion

Effect of temperature and time to the PC composite specimen was carried out at various temperature and time. It could be discussed that an increasing of annealing time and temperature yields the changing of the polycarbonate composite from ductile to brittle material that reflects in higher of maximum tensile stress and significant decrease of elongation (Medhat; et. al. 2010).

The tests of thermal properties were using DSC and TGA techniques. No significant change of T_g, weight loss and

temperature range of weight loss imply that the main structure chain and composition of polycarbonate composite are not changes during annealing.

From the SEM results, it was found that increasing of treatment time yields the finer and more homogenous of PC composite microstructure. It could be considered that thermal energy from annealing causes the increase in mobility of the molecules. The increase in mobility of the polymer chains and carbon black will allow them to interdiffuse and thus form a stronger bond (G. S. Sekhon; et. al. 2008). From the elongation was significantly dropped by heat from annealing caused the degradation of the polycarbonate composite result to changing from ductile to brittle material.

References

[1] Annual Book of ASTM Standards. (2004). Section Eight Plastics Volume 08.01 Plastics. International Standards Worldwide. (I) D256-D2324.

[2] Bo Yin, Yin Zhao, Wei Yang, Min-min Pan, Ming-bo Yang. (2006). Polycarbonate/liquid crystalline polymer blend: Crystallization of polycarbonate. Polymer. 47, 8237-8240

[3] Bok Nam jang, Charles A. Wikie. (2004). A TGA/FTIR and mass spectral study on the thermal degradation of bisphenol A polycarbonate. Polymer Degradation and Stability. 86, 419-430

[4] G.S. Sekhon, Sunil Kumar, Charanjit Kaur, N.K. Verma, S.K. Chakarvarti. (2008). Effect of thermal annealing on pore density, pore size and pore homogeneity of polycarbonate NTFs. Radiation Measurements. 43, 1357-1359

[5] I. Groves, T. Lever, and N. Hawkins. (2008). Thermal Analysis Application Brief Determination of Carbon Black Pigment in Nylon 66 by TGA. Thermal Analysis & Rheology. Number TA-122

[6] Medhat M. Hassan, Nagwa A. Badway, Azza M. Gamal, Mona Y. Elnaggar, El-Sayed A. Hegazy. (2010). Effect of carbon black on the properties of irradiated recycled polyamide/rubber waste composites. Nuclear Instruments and Medthods in Physics Research, B 268,2527-2534