TRANSPARENT STRUCTURAL COMPOSITES FOR SPACE APPLICATIONS

Zhanhu Guo[1], Hyo-Chan Kim[1], Yong-Bin Park[2], H. Thomas Hahn[1] and [Tsuyoshi Saotome[1]]: saotome@naa.att.ne.jp

[1]UCLA Mechanical and Aerospace Engineering Department
Multifunctional Composites Laboratory
[2]High-Performance Materials Institute (HPMI)
Department of Industrial and Manufacturing Engineering
FAMU-FSU College of Engineering

Abstract

Polycarbonate (PC) is widely used in aerospace applications, such as airplane windows and space suit helmets due to its superior mechanical properties, impact resistance, thermal properties and transparency. These properties are expected to improve by incorporating carbon nanoparticle filler materials, such as graphite nano platelet (GNP), while maintaining its optical transparency. It is expected that reinforced PC nanocomposite will make inroads into space applications as primary structures.

This paper presents a process that combines solution casting and compression molding to obtain neat PC, dog-bone specimens, which is considered the first important step to get GNP-loaded PC specimens. The pure PC specimens fabricated are characterized by air bubbles. The causes of the air bubbles are discussed, and possible solutions are proposed. We also propose a plan to evaluate the effect of the GNP loading in PC on mechanical properties and optical transparency of PC/GNP composites.

1 Introduction

In a manned space activity, a big shift from government to private companies is occurring all over the world, and companies such as Virgin Galactic, Space Exploration, and Bigelow Aerospace are having an important role to form a big space tourism industry at the moment. When space tourism industry is maturing like the current aviation industry, sub-orbital space vehicles with transparent walls or orbital resort with transparent primary structure (Fig.1) are expected to attract a number of customers by providing an immense view of the earth and space.

Our motivation is to develop GNP or carbon nanotube (CNT)-loaded PC, which can ultimately be used in space in such applications.

Fig. 1. An application of PC Nanocomposites to future orbital resort "the Crystal Space Palace"

Since the discovery of the CNT by Dr. Iijima [1] in 1991 and first synthesis of the CNT-loaded polymer nanocomposite (PNC) by Ajayan et al. [2] in 1994, several ways to make PNC have been proposed, such as solution processing, melt processing, in-situ polymerization and others [3]. A solution processing has been tried to get CNT-loaded transparent thin films [4] and succeeded in improving its elongation. Our attempt is to expand it to make a thin plate specimen.

Our research consists of three parts.

- ✓ Making dog-bone shape PC specimen (pure PC and GNP-loaded PC) by compression molding. The specimen is not a film but a thin plate. The basic flow is shown in Fig.2.
- ✓ Measuring tensile strength, Young's modulus, and transparency with different GNP loading.

- ✓ Comparing the results among several GNP loading and evaluating the effect of loading. Eventually we will expand the evaluation to CNT.

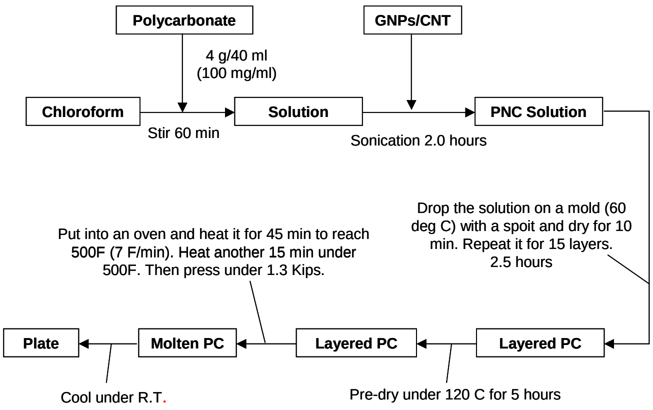

Fig. 2.Flowchart of making dog-bone shape PC specimens

This paper focuses on fabrication of void- and defect-free neat PC specimens as a preliminary step toward processing of PC/GNP composites.

2 Methods

2.1 Mold Design

The design of the mold used for our compression molding is shown in Fig.3. The mold is specifically designed to make dog-bone shape specimens defined in ASTM D1708-02a.

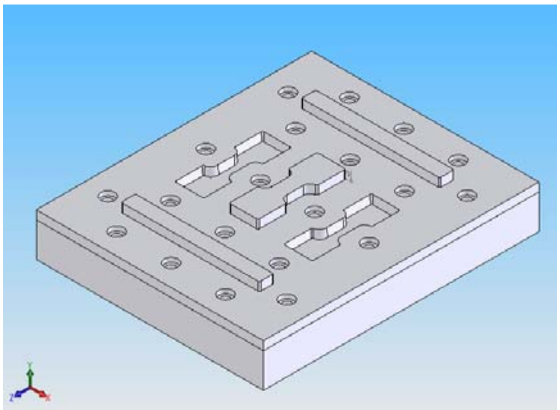

Fig. 3. Design of the dog-bone shape mold

2-2 Solution Processing/Hot Casting

Chloroform (EMD CX1054-6) was used to dissolve PC (GE Plastics LEXAN 103-112) in our case. As our future plan, nitric acid treated GNP (see Section 3) will be mixed into the PC solution and sonicated for 2 hours to have a good dispersion.

Thicker solution is recommended to minimize the solvent-induced crystallization [4], so we dissolved 100 mg PC into 1 mL chloroform. Then we set the mold temperature to 60°C, poured a small amount of the solution in it, dried it out to make a thin film, poured another solution directly on top of the film, then repeated the process till solid PC fill the mold.

2-3 Pre-dry

PC usually contains around 0.15-0.20 weight % of moisture in it, which causes hydrolysis under high compression molding temperature, generating bubbles from water vapor and CO_2 gas (Fig.4) [6]. Hydrolysis also turns the specimen into yellow or brown due to bisphenol A monomer or carbonate oligomer occurring in the process. Since appropriate sample condition for mechanical property and transparency measurement is vital for our experiment, both bubbles (harm the mechanical property) and yellow/brown color (harm the transparency) have to be eliminated.

To avoid the hydrolysis, a process called "pre-dry" is highly recommended to eliminate the moisture from PC specimen before doing compression molding [6]. Pre-dry requires you to dry PC or GNP-loaded PC for 4-5 hours at 120°C (Fig.5). We go through the process after hot casting mentioned in Section 2-2.

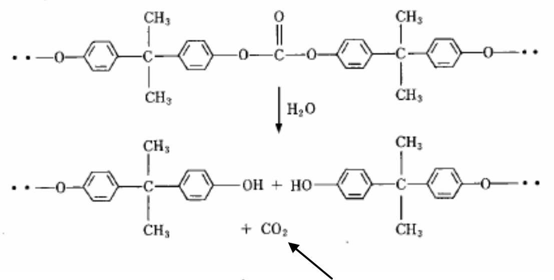

Fig.4. CO_2 gas from the PC hydrolysis [6]

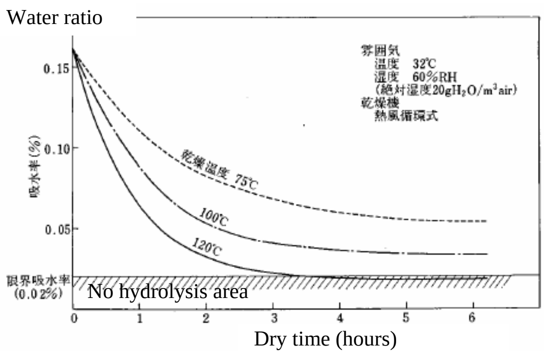

Fig.5. Moisture curve of PC [6]

2-4 Compression Molding

The specimen going through the solution processing, hot casting and pre-dry is ready for compression molding. The press condition is shown in Fig.6 [6]. Note that the press temperature is below that of PC decomposition detailed in Section 2-5. The hot press machine (Tetrahedron) is shown in Fig.7.

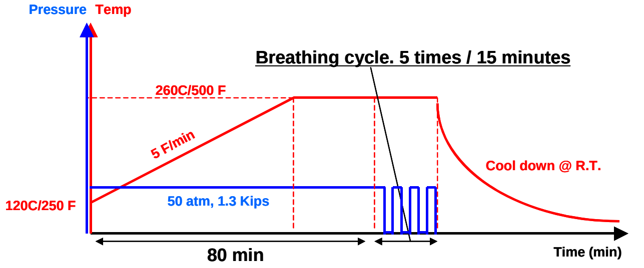

Fig.6. Hot press condition for PC

Fig.7. Hot press machine

2-5 Thermo Gravimetric Analysis of PC

Decomposition temperature of PC is measured using thermogravimetric analysis to confirm that the compression molding does not cause any decomposition. Fig. 8 shows that the decomposition occurs at 400°C (750°F), which is above the compression molding temperature of 260°C (500°F).

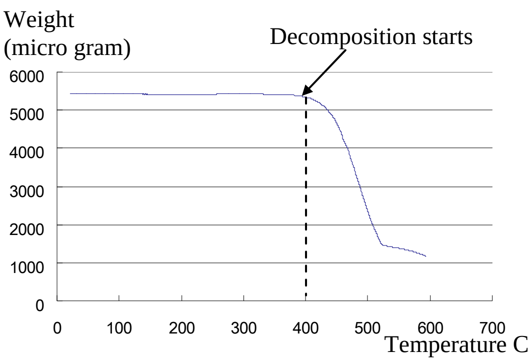

Fig.8. Decomposition temperature of PC

3 Results and Discussions

3-1 Pure PC Specimen

A pure PC dog-bone shape specimen is shown in Fig. 9.

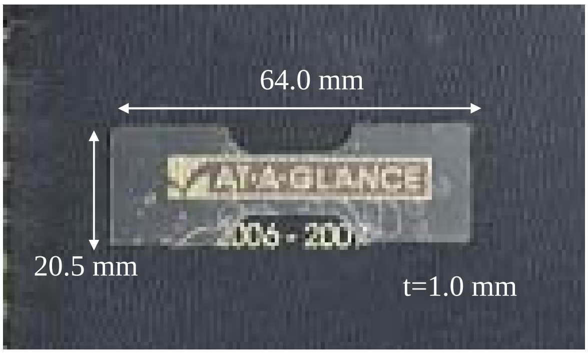

Fig.9. Pure PC dog-bone shape specimen

In spite of the pre-dry process mentioned in Section 2-3, the PC specimen still contains bubbles, which might be caused by the hot casting process mentioned in Section 2-2. In the process, we could observe that the thin PC films began to crack after laid up for 3 to 4 layers and started to have a gap containing air in it.

To avoid this crack issue, we can hot cast thin films first, stack them in the mold under dry condition, and compression mold. The current challenge is that, it is really inefficient to cut out plenty of dog-bone shaped thin films and lay them up. Instead, we will try to fabricate multiple circular films (4.0” in diameter) by hot casting in petri-dishes, lay them up and compression mold in a newly-designed circular mold (see Fig. 10), and cut out the dog-bone specimen using water-jet cutting after compression molding. Other advantages of the idea are:

- ✓ We can cut out the area which has no bubbles or we can also cut out any desirable shapes.
- ✓ We can cut out a few samples having uniform quality from one circular plate.
- ✓ It is easier to control the gap tolerance between male and female mold for circular mold than dog-bone shape mold so that there is no leakage of the polymer.
- ✓ It is much easier to design, machine, and maintain the circular mold than dog-bone shape mold. In case the mold is worn out, the circular mold can also be much more easily replaced than dog-bone shape mold which is expensive to machine.

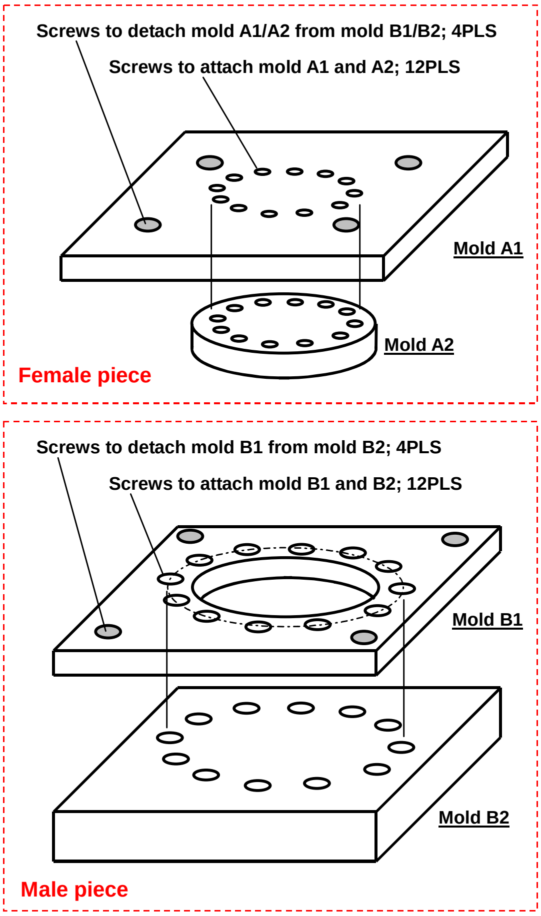

Fig.10. Design of the cylindrical mold

We can also use PC powders instead of films for compression molding. As is explained in chapter 3-2, we could successfully make a dog-bone shape PMMA specimen from its fine powder. The advantage of the concept is it is much more time-efficient to make samples than laying up films. The challenge is that, for our PC/GNP or PC/CNT polymer nano composite;

- ✓ PC is very hard to be ground with Cryo-grinding due to its high toughness. We can try spray method, spray the PC or GNP-loaded PC solution into a hot air flow and dry it as powders.
- ✓ There might be disconnections of GNP-loaded PC networks over the boundary between powders whereas thin films have continuous networks of GNP-loaded PC towards tensile orientation; i.e. not necessarily to the thickness direction. Whether we can make strong networks over the powder boundary by compression mold is yet to be studied.

3-2 Pure PMMA Specimen

We made a dog-bone shape PMMA specimen from PMMA powders. The powder could be obtained with Cryo-grinding, the basic idea of which is to cool down the PMMA pellets to –200°C with liquid nitrogen and grind them while being brittle. PMMA, LDPE, and polypropylene could be well ground, but it was very hard to grind PC in our experience.

Fig.11. Microtech UTM100 cryo-grinder

Once ground, the PMMA powder less than 100 μm was separated using a sieve, and compression molded under the pressure and temperature condition shown in Fig. 12. 5 hour-pre-dry under 80°C was also incorporated.

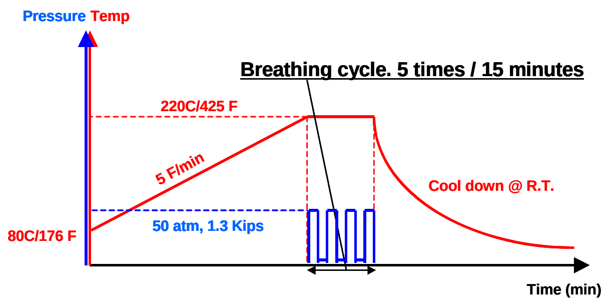

Fig.12. Hot press condition for PMMA

We confirmed that the obtained PMMA specimen had no moisture or air induced bubbles in it (Fig. 13). The left side surface of the specimen was polished to take out the surface roughness and enhance its transparency.

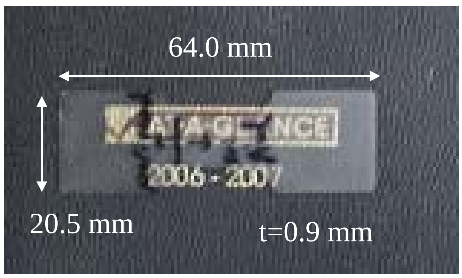

Fig.13. Pure dog-bone shape PMMA specimen

4 Conclusions

A solution processing, hot casting, pre-dry and compression molding was implemented to get a pure PC dog-bone shape specimen which contains bubbles in it. By comparing the PC and PMMA samples, we speculated that what caused the bubbles was our hot casting, and discussed about our future plan to get an appropriate pure PC specimen which will lead to GNP-loaded or CNT-loaded PC specimen.

Our future plan is to machine a new cylindrical mold (Fig.10.) and try the film lay-up method to make pure PC specimen first. We will also try the powder method if needed.

Good dispersion of CNT or GNP can be achieved either by incorporating surfactant or surface functionalization [3]. Once we get a pure PC specimen, we will use nitric acid (EMD NX0409P-5) to introduce Carboxylic functional group onto the GNP (Asbury Carbons grade 3775) surface [5] and make GNP-loaded specimen.

Then we will evaluate the effect of the GNP's loading to tensile strength, Young's modulus, and transparency. We will also check the dispersion level of the GNP using optical microscope and SEM.

Eventually we will expand the whole ideas to CNT and evaluate its effects as well.

References

[1] Iijima, S. *Nature (London) 1991, 354, 56-8*

[2] Ajayan P.M. Stehpan O., Colliex C., and Trauth D. *Science 1994, 265, 1212-14*

[3] Moniruzzaman M., and Winey K.I. *"Review. Polymer nanocomposites containing carbon nanotubes".*

[4] Chin Y.-C., Chu A., Pham G.T., and Park Y.-B. *"Design and fabrication of transparent, impact resistant polycarbonate/carbon nanotube composites".*

[5] Marshall M.W., Popa-Nita S., and Shapter J.G. *"Measurement of functionalized carbon nanotube carboxylic acid groups using a simple chemical proess.*

[6] Honma S. *"Polycarbonate Polymer Handbook"*