Composites: Part A 90 (2016) 457–469

Contents lists available at ScienceDirect

Composites: Part A

journal homepage: www.elsevier.com/locate/compositesa

Effect of bubble based degradation on the physical properties of Single Wall Carbon Nanotube/Epoxy Resin composite and new approach in bubbles reduction

Seyyed Alireza Hashemi [a,*], Seyyed Mojtaba Mousavi [b]

[a] Department of Mechanical Engineering, Najafabad Branch, Islamic Azad University, Najafabad, Iran
[b] School of Chemical and Petroleum Engineering, Shiraz University, Shiraz 71345-1789, Iran

ARTICLE INFO

Article history:
Received 10 June 2016
Received in revised form 27 July 2016
Accepted 16 August 2016
Available online 17 August 2016

Keywords:
Degradation
Void
Bubble
Composite
Single Wall Carbon Nanotube

ABSTRACT

In this study the effect of bubble based degradation on the physical and structural properties of Single Wall Carbon Nanotube (SWCNT)/epoxy resin composite samples were investigated and new method based on vacuum shock was presented. For this purpose, with two different methods samples with and without bubble based degradation were fabricated and effect of degradation on the value of electrical conductivity and the amount of Electromagnetic (EM) waves absorption were investigated. Which vacuum shock technique can improve above mentioned properties about 58319.594 and 63.921 percentage for sample without degradation in comparison with destroyed sample due to the bubbles based voids effect. Moreover, the main factors in the bubbles formation and migration during the manufacturing process and their behavior in the matrix with the help of optical and SEM images were examined and their effect on structural properties of composite samples with Micro Raman Spectroscopy was evaluated.

1. Introduction

Bubbles and their resulting voids are among most destructive factors during the fabrication of composite materials that can lead to significant decrease in overall properties and huge destruction in composite samples structure due to increase in gas concentration of bubbles. Creation of bubbles depend on the balance between their inner and outer pressure. Also bubbles growth and formation may occur due to diffusion of air or by agglomeration with other bubbles. Moreover, among the most effective factors that can affect bubbles mobility, we can refer to bubbles size, viscosity of polymeric matrix, fluid surface tension and fillers network configuration. Despite of that, simultaneous usage of vacuum and vibration with low frequency about 10–30 Hz can highly reduce the overall amount of bubbles and their size in the matrix [1]. In addition, many factors such as temperature, time, pressure, heating transfer and curing process conditions are involved in bubbles reduction [2,3]. As well as above mentioned conditions, molecularly disperse of bubbles into the polymeric matrix can lead to reduction in overall amount of bubbles in the whole structure [3]. In addition, some production situations such as operating time, concentration and flow rate of the surfactant and superficial gas velocity can affect bubbles size and air hold up in the suspension. Despite of that, superficial velocities and surfactant concentration can lead to generation of medium-sized bubbles and increase in air hold up values [4]. Besides, surface reaction and gas transfer rate from liquid to bubbles interface are two main reasons for bubbles growth during photo electrochemical and electrochemical conversions respectively. In spite of that, bubbles growth rate for photo electrochemical conversion and electrochemical conversion are due to small effective solid surface engagement and big effective solid surface participating [5].

Moreover, bubbles have a strong desire to combine with each other and create larger bubbles that can cause huge destruction due to increase in their inner pressure after firs curing step of composite samples [6]. In addition, bubbles can grow due to the diffusion enhanced by the Thomson-Freundlich effect and pressure oscillation [7]. In aerospace and other industries, bubbles and their resulting voids content would be better not to be more than 1 and 5 wt% respectively [8,9]. Furthermore, the presence of bubbles and their resulting voids in a large numbers and with large diameters as well as increase in volume fraction, can act as a stress concentration areas and lead to reduce in the mechanical properties of composite samples include tensile, flexural and shear strength and also bending, tension and fracture toughness [3,10–13]. Despite of that,

* Corresponding author.
E-mail addresses: s.a.hashemi0@gmail.com (S.A. Hashemi), kempo.smm@gmail.com (S.M. Mousavi).

http://dx.doi.org/10.1016/j.compositesa.2016.08.015
1359-835X/

among this mechanical properties, voids can seriously affect shear and compression strength [14]. Moreover, cure cycle can highly affect the mechanical properties of composite samples. By means of ILSS tests, for void content about 1 wt% the ILSS decreased due to the voids volume fraction [15]. Also increase in the overall amount of temperatures and pressures during the cure cycle can affect the bubbles content and size distribution. In addition, increase in the magnitude of applied pressure to the composite samples and also removal of the gaps inside the mold can lead to significant decrease in the overall amount of bubbles content and their average diameter [16]. Besides, cure cycles can affect both bubbles content and mechanical properties of composite samples. Furthermore, increase in the pressure can lead to decrease in the amount of bubbles and their resulting voids content in the matrix [13]. In a work by Li et al. [3] they have investigated the formation mechanism of voids and also their effect on the mechanical properties of composite samples. According to their study, the amount of bubbles volume fraction in the composite samples decreased due to increase in the magnitude of cure cycle pressure. Furthermore, increase in the amount of cure cycle temperature had no effect on the amount of bubbles inside the matrix context, but it can affect the location of the bubbles. In addition, cycle temperature can affect the amount of bubble based voids, dimensional structure and spatial distribution of voids in the matrix. Besides, elongation factor of bubble based voids that is based on their length and average diameter will increase due to voids size and volume. Despite the fact that most of the voids were the result of air entrapment and wrinkles. Furthermore, higher pressure in regions can lead to the migration of resin as well as bubbles in the polymeric matrix. In a work that had been conducted by Nie et al. [2], they have investigated the bubbles behavior in the polymeric matrix and also bubbles formation reasons. Then they have compared experimental and theoretical results with each other. Some experimental and production methods indicates that bubbles can shrink and also disappear due to certain conditions. In this situation, bubbles will disperse into the polymeric matrix molecularly and the stress concentration due to these bubbles will not exist any longer. Moreover, the gas diffusion is very important in bubbles formation. Gas molecules are migrating from high concentration zones toward lower ones and also the bubbles volume fraction can change due to the migration of gas molecules. In addition, by increase in the temperature and pressure and decrease in the viscosity of polymeric matrix, the amount and average diameter of bubbles and their resulting voids will decrease [2]. Furthermore, bubbles and their resulting voids can be formed at the bond interface due to incomplete flowing of the matrix and gas remnant from the neighbor polymer may fill empty spaces to form gas bubbles [2,17,18]. In another works by DeValve et al. [19] and Frishfelds et al. [20], they have simulated the bubbles formation in the liquid composite and also their motion through non-crimp fabrics respectively. Based on their study, bubbles can highly affect and distribute the structural shape of composite samples. Moreover, while bubbles reaching the resin flow front, their mobility will increase significantly. The highest bubbles mobility occur while the bubbles are near to the resin flow fronts. When the flow cell has a moving flow front, the bubbles mobility was found to be greater than saturated flow cells. This is due to the dominance of the surface tension at flow front while bubbles approaching to the flow front. Also decrease in the thickness of the resin substrate can lead to increase in the migration speed of the resin flow and thus the migration speed of bubbles in the flow front [21]. In addition, natural fillers in the composite structure can increase the speed of degradation in the composite structure [22].

In this study for better investigation about the impact of bubbles and their resulting voids on the physical properties of composite samples such as electrical conductivity, Electromagnetic (EM) waves absorption and composite structural shape, different production methods were used for fabrication of composite samples with and without bubbles and their resulting voids in the whole composite structure. Then composite samples containing Single Wall Carbon Nanotubes (SWCNTs) with and without voids were fabricated and compared with each other and effect of bubble based voids on the above mentioned properties of composite samples were examined. Then the behavior of bubbles before and after fully curing of composite samples were examined and samples structure were evaluated with SEM and optical images and Micro Raman Spectroscopy. Bubbles and their resulting voids are among most destructive factors in the fabrication of composite samples, this study was conducted for better investigation about bubbles behavior and their creation reasons in order to present a multi-step method based on the vacuum shock technique for removal of bubbles and their resulting voids from the composite samples.

2. Experimental section

2.1. Materials and instruments

In this study SWCNTs was Supplied by US Research Nanomaterial. Average external and inner diameter and length of these SWCNTs are about 1–2 nm, 0.8–1.6 nm and 5–30 μm respectively. The purity of these SWCNTs is more than >96%. Moreover, for better dispersion of these SWCNTs in the polymeric matrix, —COOH (Carboxyl) functional group were applied on their surface by acidic method. Other specification of these SWCNTs can be seen in Table 1.

In addition, NPEL-128 (Nan Ya-128) epoxy resin and Acetone were used as polymeric matrix and dispersant agent respectively. The epoxy resin and Acetone dispersant agent were supplied by Nan Ya Plastics Corporation and Merck respectively. Moreover, for measuring the amount of Electromagnetic waves absorption and electrical conductivity, Vector Network Analyzer (manufactured by ROHDE & SCHWARZ) by frequency range between 10 MHz and 20 GHz and IV-CV parameter analyzer (model K361, manufactured by Keithy) were used. Also for examination of composite samples Field Emission Scanning Electron Microscope (FE-SEM) (model HITACHI S-4160) analysis, 1000× Digital Microscope (manufactured by Rohs) and Micro Raman Spectroscopy (model SENTERRA (2009) manufactured by BRUKER CO) were used.

2.2. Composite samples production methods

In this study, for the production of composite samples with and without degradation that was caused by bubbles and their resulted

Table 1
Specification of SWCNTs that had been used in this study.

SWCNTs specification		Measurement instruments
OD	1–2 nm	From HRTEM and Raman Spectroscopy
ID	0.8–1.6 nm	From HRTEM and Raman Spectroscopy
Average diameter	1.1 nm	From Raman Spectroscopy
Length	5–30 μm	TEM
Tap density	0.14 g/cm^3	–
True density	~2.1 g/cm^3	–
SSA	>380 m^2/g	BET
Ash	<1.5 wt%	HRTEM and TGA
Electric conductivity	>100 S/cm	–
Thermal conductivity	50–200 W/m K	–
Color	Black	–
I_g/I_d	>9	Raman Spectroscopy