

# Chapter 7

## Nanotubes/Polymethyl Methacrylate Composite Resins as Denture Base Materials

Russell Wang, Ramazan Kayacan and Cenker Küçükeşmen

### 7.1 Introduction

Denture base acrylics resins are subjected to many different types of stresses. Intra-orally, repeated masticatory forces lead to fatigue phenomena. Several studies have investigated the incidence and types of fracture of dentures [1–3]. Darbar et al. [4] reported that 33% of the repairs carried out were caused by debonded/detached teeth and 29% were repairs to midline fractures more commonly seen in upper complete dentures. The midline fracture in a denture is often a result of flexural fatigue. Impact failures usually occur due to sudden blow to the denture by accidental dropping.

The fracture resistance of denture base polymers has been investigated [5–9]. Reinforcement of denture base material has been a subject of interest to the dental material community. Polymethyl methacrylate (PMMA) resin is the principal material of dental prosthesis. To improve the properties of PMMA, many have incorporated an ample variety of additive materials into the polymer, including glass fibers, long carbon fibers, and metal wires [10–13], although success has been limited [14, 15]. Flexural strength of PMMA resin such as Lucitone-199 (Dentsply Trubyte, New York, PA, USA), which is a high-impact resin with butadiene and styrene additives, has been evaluated by several investigators, showing conflicting values but generally without significant material strengthening [1, 8, 16, 17].

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R. Wang (✉)

Department of Comprehensive Care, Case Western Reserve University, School of Dental Medicine, 10900 Euclid Ave. Cleveland, OH 44106-4905, USA  
e-mail: rxw26@case.edu

R. Kayacan

Department of Mechanical Engineering, Süleyman Demirel University, Isparta, Turkey

C. Küçükeşmen

Department of Prosthodontics, Süleyman Demirel University School of Dentistry, Isparta, Turkey

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Carbon nanotubes (CNTs) have shown extremely high mechanical properties with reported strengths 10–100 times higher than the strongest steel at a fraction of the weight [18, 19]. They have light weight. There are two main types of CNTs that can have high structural perfection: single-walled nanotubes (SWCNTs) consisting of a single graphite sheet seamlessly wrapped into a cylindrical tube, and multi-walled carbon nanotubes (MWCNTs) composed of an array of such nanotubes concentrically nested like rings of a tree trunk. In addition to the exceptional mechanical properties associated with CNTs (elastic modulus of 1 TPa, diamond: 1.2 TPa), they also possess superior thermal and electric properties [20, 21].

The incorporation of carbon fibers into a matrix not only confers strength and elasticity to the material but also greatly enhances toughness [22]. Research on nanotube composites has concentrated on polymer–CNT-based materials, wherein they exhibit mechanical properties that are superior to conventional polymer-based composites due to their considerably higher intrinsic strengths and moduli, and the fact that the stress transfer efficiency can be ten times higher than that of traditional additives [23]. It is generally believed that most MWCNTs have a “Russian doll” structure in which each constituent tubule is only bonded to its neighbors by weak van der Waals forces. This immediately raises a problem when one is considering incorporating CNTs into matrices [24].

The second concern is the even dispersion of CNTs into a polymer matrix. Methods of using sonic dismembrators, such as chemical modification, have been proposed by Fiedler et al. [20, 25, 26]. Sui and Wagner [27] observed unusually large deformation in PMMA electrospun fibers under tension when MWCNTs or SWCNTs were included as a second phase in the fibers. The addition of CNTs caused a striking, visible transformation in the deformation mode of PMMA fibers. In pure PMMA fibers, sparse and unstable polymer necking occurs under increasing tension, leading to failure at relatively small strains. However, the presence of either SWCNTs or MWCNTs causes the failure strain to reach comparatively enormous values. According to Marrs, MWCNTs are believed to effectively bridge cracks and reduce the extent of plastic deformation experienced by a PMMA matrix. MWCNTs can successfully reinforce the craze by strengthening the fibrils and bridging the recesses or submicron voids to prevent their coalescence, thus enhancing the fatigue performance of the material.

The effects of CNTs reinforcement on mechanical properties of denture base materials have not been explored. The purpose of this study was to evaluate the effect of CNT reinforcement on mechanical properties of a commonly used PMMA denture base material. The null hypothesis is that the addition of CNTs to a PMMA polymer matrix will improve its mechanical properties.

## 7.2 Materials and Methods

The specimens were fabricated using the denture base resin, Lucitone-199® original shade (Dentsply International Inc., New York, PA, USA). The MWCNTs, as received from the manufacturer (Designed Nanotubes, LLC., Austin, TX, USA),

were added to the measured acrylic monomer at 0.50, 1, and 2 wt% of total weight in a glass beaker. The liquid monomer was then under ultrasonic mixing for 20 min (Model UP400S, Hielscher Ultrasonics GmbH, Teltow, Germany). Following the manufacturer's instructions, the powder to liquid ratio of 21 g (32 cc)/10 ml and a mixing time of 20 s were used. Liquid monomer with and without MWCNTs was added to the powder and mixed for 20 s to assure wetting of all powder particles. The mixture was covered for 9 min at room temperature and allowed to reach packing consistency. The mixture was packed using conventional denture flasks (Hanau Type, Whip-Mix Corporation, Louisville, KY, USA). Samples were molded as rectangular beams prepared in standard denture flasks, using a template measuring  $70 \times 40 \times 3$  mm. The flasks were tightened by spring clamp and samples were cured in a water bath for a period of 9 h at 160 °F, followed by a cooling time of 30 min in water at 60–80 °F. The flasks were bench-cooled for 30 min and submerged in cool water for 15 min before deflasking. The specimens were removed from the flasks and cleaned from stone particles. After deflasking, each mold was prepared to the size of  $70 \times 10 \times 3$  mm for a three-point flexural test. The specimens were sequentially polished with SiC papers (600, 800, and 1000 grit) to achieve smooth surfaces.

### Mechanical Tests

The flexural strength and flexural modulus were determined by using the three-point bending test as specified by the ISO specification 20795-1:2008. Four groups were prepared at 0.0, 0.5, 1, and 2 wt% of MWCNTs with seven samples per group. The specimens were tested without thermocycles using a universal testing machine (Sintech Renew 1121, Instron Engineering Corp., Canton, MA, USA). Before each test, the specimen thickness and width were measured with a digital micrometer and recorded. A standard three-point bending jig was attached to the machine controlled by a computer. The testing parameters were set at the rate of 0.5 mm/min and load was recorded by a 1000-lb load cell. The same flexural test was performed for another set of four groups of same MWNCT fractions, which underwent 5000 thermocycles from 20 to 80 °C at 1-min intervals using a thermocycle machine (Model Haake EK30, thermo Electron Corporation, Feldkirchen, Germany).

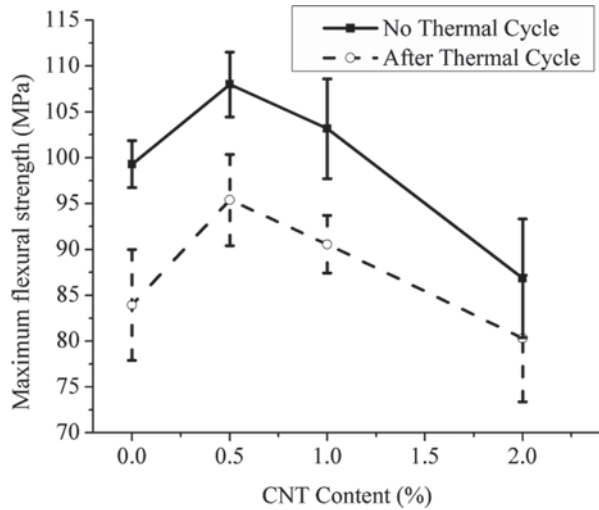
The flexural strength  $S$  was calculated using the following formula:

$$S = 3FL / 2bd^2$$

where  $S$  is the flexural strength in MPa,  $F$  is the failure load,  $L$  is the span of specimen between supports (50 mm),  $b$  is the width of each specimen, and  $d$  is the thickness of each specimen.

Based on the load–displacement ( $F$ – $\Delta$ ) curves of a three-point bending test, mechanical properties were determined. Stiffness was calculated as the slope of the load–displacement curve obtained by linear regression. The yield point was calculated as the intersection of the load–displacement curve with a line having a slope that was 95% that of the stiffness (Fig. 7.1). The load and displacement at the yield point were taken as the yield load ( $F_y$ ) and the yield displacement ( $\Delta_y$ ). The elastic work was calculated as the area under the load–displacement curve within the elastic range using the trapezoidal rule.

**Fig. 7.1** Maximum flexural strength versus CNT content (0, 0.5, 1.0, 2.0 wt% of MWCNTs). CNT carbon nanotubes



The moment of inertia about the bending axis ( $I$ ) and the thickness ( $t$ ) of each specimen was used to calculate yield stress, yield strain, resilience, maximum flexural strength, and flexural modulus according to Akkus et al. [28]:

$$\text{Yield stress} = (F_Y Lt)/(8I)$$

$$\text{Yield strain} = (12\Delta_Y t)/(2L^2)$$

$$\text{Resilience} = 0.5 (\text{Yield stress} \times \text{Yield strain})$$

$$\text{Maximum flexural strength} = (F_U Lt)/(8I)$$

$$\text{Flexural modulus} = \text{Stiffness } L^3/(48I)$$

$$\text{Flexural rigidity} = \text{Flexural modulus} \times I$$

where  $L$  is the span length between the two outer support points and  $F_U$  is the ultimate load.

**Microhardness Test**

The hardness test was performed for each sample in the dry condition. Fractured specimens from the flexural strength test were used for the Knoop hardness test. Each sample was tested six times with a Knoop hardness tester (Lecco® Corp. M-400 St. Joseph, MI, USA) using a 200 g load and 20 s dwell time. Microhardness number for each specimen was based on the average of six repeat measurements from the same sample. The Knoop hardness number (KHN) is the ratio of the load applied to the area of the indentation calculated from the following formula:  $KHN = L/2 \times [C_p]$ . In this equation,  $L$  is the load applied in kilogram force,  $[$  is the

length of the long diagonal of the indentation in millimeters, and  $C_p$  is a constant relating  $l$  to the projected area of the indentation.

### Statistical Method

After data collection, mean values of the surface microhardness, maximum flexural strength, resilience, bending modulus, yield stress, and yield strain for each group were analyzed with two-way analysis of variance (ANOVA) to determine the main significant effects of CNT percentage, thermocycle process, and their interactions. Furthermore, the Tukey's honest significant difference (HSD) post hoc (multiple comparisons) analysis was applied to determine the significant effects. All statistical analysis was performed with statistical software (IBM SPSS Statistics 19, Statistical Package for Social Science; SPSS Inc, Chicago, IL, USA).

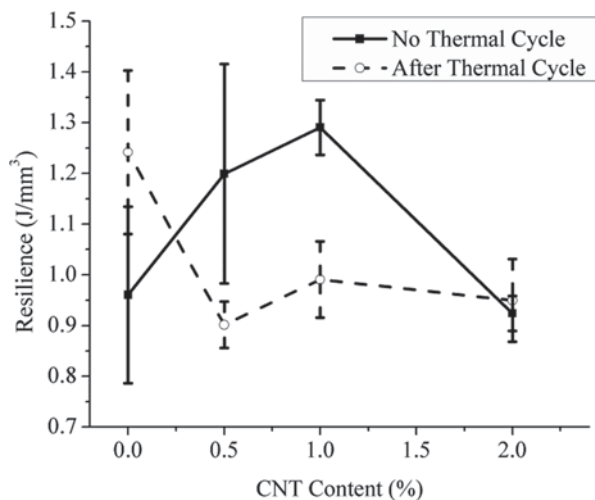
## 7.3 Results

Figures 7.1, 7.2, 7.3, 7.4, 7.5 summarize the mechanical behaviors of MWCNT/PMMA composites of maximal flexural strength, resilience, bending modulus, yield stress, and yield strain with and without thermocycling (TC). Tables 7.1, 7.2, 7.3, 7.4, 7.5, 7.6 are the summary of means and overall means of the results of each mechanical property and microhardness evaluated in this study.

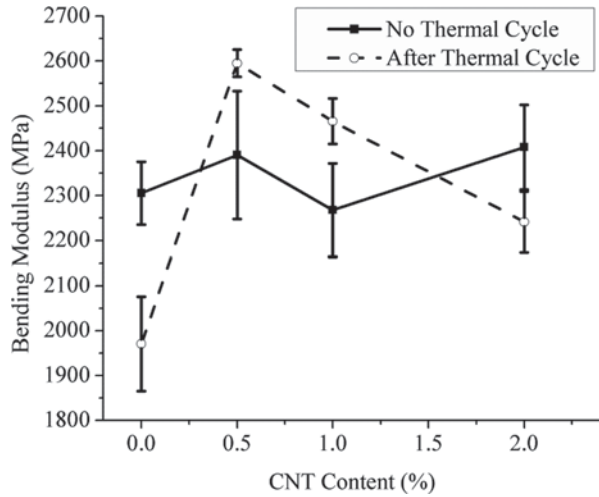
### Maximum Flexural Strength

When all the tested groups were compared, there was no significant interaction between CNT level and TC process on the PMMA's maximum flexural strength ( $P=0.22$ ). However, the fraction of MWCNT significantly affected the PMMA's maximum flexural strength ( $P<0.0001$ ). When 0.5 wt% of MWCNT was used, a clear significant improvement of maximum flexural strength (101.8 MPa vs.

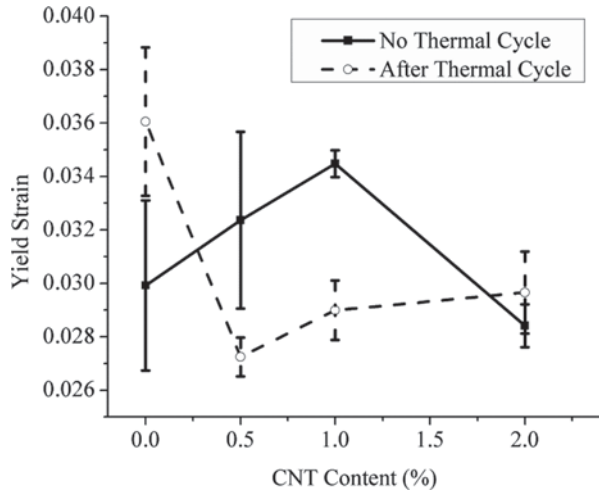
**Fig. 7.2** Resilience versus CNT content (0, 0.5, 1.0, 2.0 wt% MWCNTs). CNT carbon nanotubes



**Fig. 7.3** Bending modulus versus CNT content (0, 0.5, 1.0, 2.0 wt% MWCNTs). CNT carbon nanotubes



**Fig. 7.4** Yield strain versus CNT content (0, 0.5, 1.0, 2.0 wt% of MWCNTs). CNT carbon nanotubes

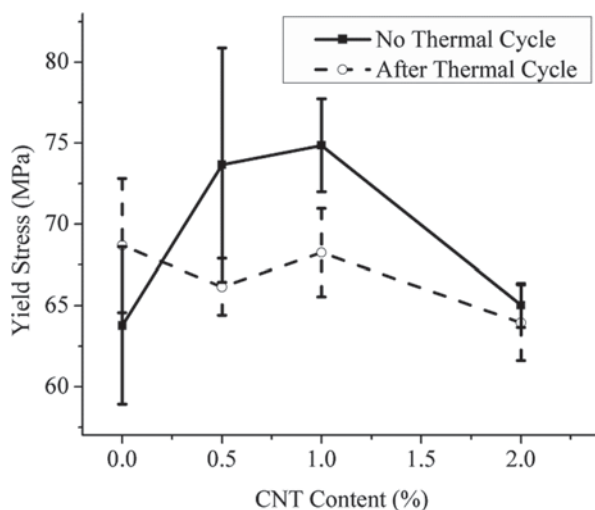


91.5 MPa,  $P < 0.0001$ ) was found in comparison with that of the control group (a 9.3% increase). Contrarily, the increase in the percentage of CNT to 2.0% actually weakened the maximum flexural strength (84.1 MPa vs. 91.5 MPa,  $P = 0.002$ ). The TC process also significantly decreased the PMMA's maximum flexural strength ( $P < 0.0001$ ). In general, 10–13% of flexural strength was lost among the groups after 5000 thermocycles.

**Resilience**

There was significant interaction between CNT level and thermocycle process on the PMMA's resilience ( $P = 4.2e-06$ ). Without the thermocycle, there was an improvement of resilience when MWCNTs were added to PMMA at 0.5 and 1 wt% level ( $P = 0.017$ ). There was a reduction in resilience when CNT levels increased

**Fig. 7.5** Yield stress versus CNT content (0, 0.5, 1.0, 2.0 wt% of MWCNTs). CNT carbon nanotubes



**Table 7.1** Summary of means and overall means of maximum flexural strength (MPa)

Flexural strength	Thermocycle (no)	Thermocycle (yes)	Mean (overall)
MWCNT=0%	99.29857	83.62571	91.46214
MWCNT=0.5%	108.18571	95.39	101.787855
MWCNT=1.0%	103.16857	90.44429	96.80643
MWCNT=2.0%	87.97143	80.29429	84.13286
Mean (overall)	99.65607	87.4385725	—

MWCNT multi-walled carbon nanotubes

**Table 7.2** Summary of means and overall means of resilience ( $10^{-3}/\text{mm}^2$ )

Resilience	Thermocycle (no)	Thermocycle (yes)	Mean (overall)
CNT=0%	1.003	1.2525714	1.1277857
CNT=0.5%	1.1548571	0.9012857	1.0280714
CNT=1.0%	1.2905714	0.9811429	1.13585715
CNT=2.0%	0.9717143	0.9495714	0.96064285
Mean (overall)	1.1050357	1.02114285	—

CNT carbon nanotubes

**Table 7.3** Summary of means and overall means of bending modulus (MPa)

Bending modulus	Thermocycle (no)	Thermocycle (yes)	Mean (overall)
CNT=0%	2291.714	1950.571	2121.1425
CNT=0.5%	2379.571	2595	2487.2855
CNT=1.0%	2267.429	2487.429	2377.429
CNT=2.0%	2379.286	2241.286	2310.286
Mean (overall)	2329.5	2318.5715	—

CNT carbon nanotubes

**Table 7.4** Summary of means and overall means of yield stress (MPa)

Yield stress	Thermocycle (no)	Thermocycle (yes)	Mean (overall)
CNT=0%	64.98714	68.66429	66.825715
CNT=0.5%	72.05	66.15	69.1
CNT=1.0%	74.86571	68.15429	71.51
CNT=2.0%	66.06143	63.94714	65.004285
Mean (overall)	69.49107	66.72893	–

CNT carbon nanotubes

**Table 7.5** Summary of means and overall means of yield strain (mm/mm)

Yield strain	Thermocycle (no)	Thermocycle (yes)	Mean (overall)
CNT=0%	0.03061429	0.0364	0.033507145
CNT=0.5%	0.03181429	0.02724286	0.029528575
CNT=1.0%	0.03447143	0.02877143	0.03162143
CNT=2.0%	0.02932857	0.02964286	0.029485715
Mean (overall)	0.031557145	0.030514288	–

CNT carbon nanotubes

**Table 7.6** Summary of means and overall means of microhardness (kg/mm<sup>2</sup>)

Microhardness	Thermocycle (no)	Thermocycle (yes)	Mean (overall)
CNT=0%	16.56667	16.07381	16.32024
CNT=0.5%	15.89524	15.27619	15.585715
CNT=1.0%	15.36667	14.50238	14.934525
CNT=2.0%	11.35238	11.33333	11.342855
Mean (overall)	14.79524	14.2964275	–

CNT carbon nanotubes

from 1.0 to 2.0% ( $P=0.008$ ). The thermocycle also significantly reduced the resilience; a further study is needed to improve the dispersion of MWCNTs into the commercial denture base system at 0.5 and 1 wt% groups ( $P=1.5e-06$ ).

### Bending Modulus

There was significant interaction between CNT level and thermocycle process on the PMMA's bending modulus ( $P<0.0001$ ). In the absence of TC, there was no overall significant effect of CNT levels on the PMMA's bending modulus ( $P=0.14$ ). However, with the thermocycle process there is an overall significant effect of CNT levels on the PMMA's bending modulus ( $P<0.0001$ ). When CNT level of 0.5% was used, a clear significant improvement of bending modulus (2595 MPa vs. 1950.6 MPa,  $P<0.0001$ ) was found in comparison with the strength of the control samples (0% CNT). Contrarily, the increase in the percentage of CNT to 2.0% actually weakened the bending modulus in comparison with the CNT percentage of 0.5%. On the other hand, at the same CNT levels, the TC process led to an improvement of bending modulus with the existence of CNT (0.5–1.0%), but a weakening of bending modulus in the control samples (0% CNT) and in the sample with the 2.0% CNT level.



### Yield Stress

There was significant interaction between CNT level and thermocycle process on the PMMA's yield stress ( $P=0.007$ ). Where without the thermocycle process there was an improvement of yield stress when CNT levels going from 0 to 1.0% (74.9 MPa vs. 64.9 MPa,  $P=0.008$ ), and there was a weakening of yield stress when CNT levels going from 1.0 to 2.0% (74.8 MPa vs. 66.1 MPa,  $P=0.021$ ). With the thermocycle process, there was an overall weakening effect of CNT levels ( $P=0.013$ ) on yield stress. The smallest yield stress (63.9 MPa) was found in the samples with 2.0% CNT.

### Yield Strain

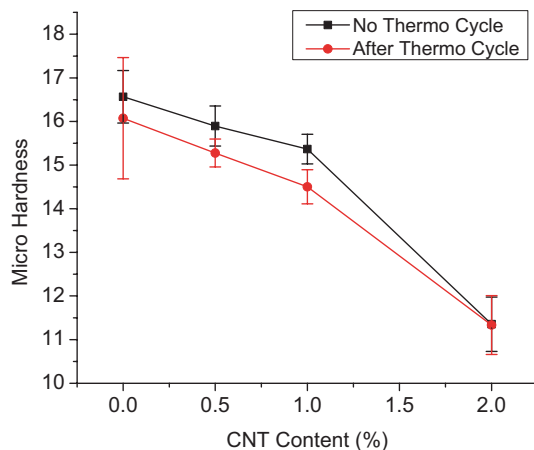
There was significant interaction between CNT level and thermocycle process on the PMMA's yield strain ( $P<0.0001$ ). Without the thermocycle process there was an overall significant effect of CNT levels ( $P=0.012$ ) on yield strain, and there was a weakening of yield strain when CNT levels going from 1.0 to 2.0% (0.34 mm/mm vs. 0.29 mm/mm,  $P=0.009$ ). With the thermocycle process, there was an overall weakening effect of CNT levels ( $P<0.0001$ ) on yield strain, the smallest yield strain (0.027 mm/mm) was found in the samples with 0.5% CNT.

### Surface Microhardness

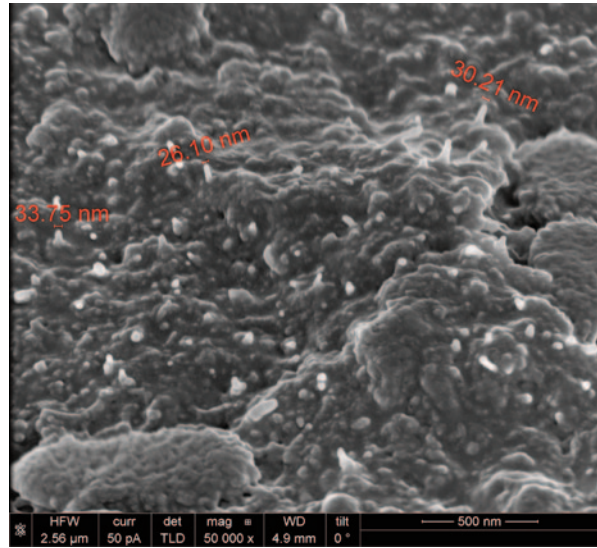
There was no significant interaction between CNT level and thermocycle process on the PMMA's surface microhardness ( $P=0.42$ ). Increasing CNT level significantly decreased the PMMA's surface microhardness ( $P<0.0001$ ). The mean surface microhardness of the control samples (0% MWCNT) was found stronger (16.32), whereas the mean surface microhardness of samples with 2.0% CNT was found actually weaker (11.34). The thermocycle process also significantly decreased the PMMA's surface microhardness ( $P=0.0086$ ) (Fig. 7.6).

Figure 7.7 is the micrograph of a fracture surface of a 0.5 wt% MWCNT/PMMA sample. Even dispersion of MWCNTs on the fractured surface is evident. The rods with bright appearance range from 25 to 35 nm in diameter. The figure also shows

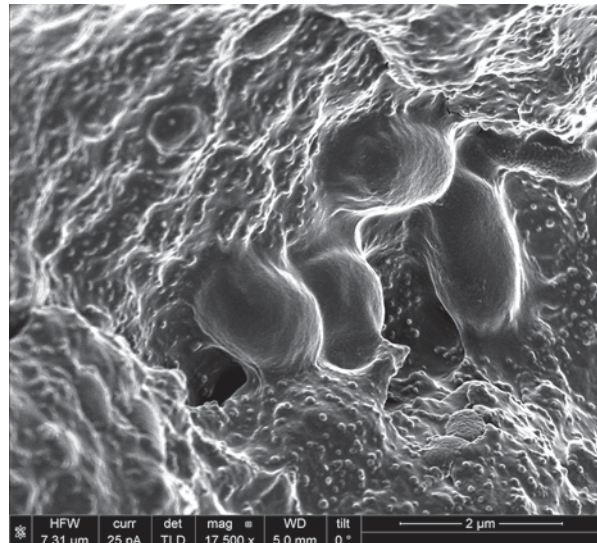
**Fig. 7.6** Microhardness (Knoop hardness number) versus CNT content (0, 0.5, 1.0, 2.0 wt% of MWCNTs). CNT carbon nanotubes



**Fig. 7.7** Micrograph (50,000 $\times$ ) of fractured surface of 0.5% CNT/PMMA. *Bright rods* are CNTs with 25–35 nm diameter



**Fig. 7.8** Micrograph (17,500 $\times$ ) of fractured surface of 1% CNT/PMMA. *Bright dots* are CNTs and *dark voids* are porosities



that the bright and protruded MWCNTs are perpendicular to the surface with clean cleavage fracture, which is indicative of the lack of adhesion of MWCNTs to the PMMA matrix..

Figure 7.8 presents voids at fracture surface. The amount of voids likely was introduced into the samples during powder/liquid mixing and packing the material into a denture flask. The amount of voids might affect the mechanical strength of a sample.

**Fig. 7.9** Micrograph (25,000 $\times$ ) of fractured surface of a 2 wt% MWCNT/PMMA sample. Center of the image shows an agglomeration MWCNTs and *arrows* are single fibrils MWCNTs

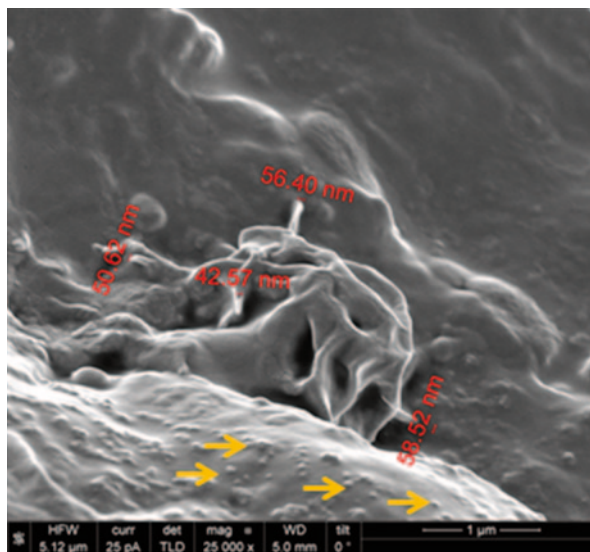


Figure 7.9 is the representation of fractured surface of a 2 wt% MWCNT/PMMA sample. The center of the figure shows an agglomeration of MWCNTs with polymerized monomer. This characteristic is noticed on the other fractured surface of 2 wt% MWCNT/PMMA samples. The arrows are the small single MWCNTs embedded in PMMA matrix.

## 7.4 Discussion

The introduction of MWCNT–polymer composites has been advocated to usher in fresh opportunities for engineering new materials with high-performance capabilities. Some researchers believe that the nanoscale dimensions and extraordinary properties of MWCNTs directly address the submicron damage mechanisms (such as crazing) that culminate during the fatigue failure of most polymers. The large traditional glass or graphite fibers commonly used in composite materials do little to address these microscopic mechanisms of cyclic deformation; thus, MWCNTs may potentially offer a new promise for improving the performance of polymer systems, such as PMMA, where the previous successes of traditional fibers were limited. To test this theory, we added small amounts (0–2 wt%) of MWCNTs to Luciton-199, a PMMA denture base polymer, to examine any effect on mechanical properties of MWCNT/PMMA composites. We also tested the effect of thermocycles on those properties of the composite.

We demonstrate that while MWCNT reinforcement improves the mechanical properties of the composite structure mainly by elevating the yield point without affecting the modulus or fracture strength, yield point is the region where the elastic

deformation ends and plastic/damage-related mechanisms initiate. Elevation of this point indicates that MWCNTs would delay the damage initiation and may improve the longevity of the denture base material under impact forces or static loadings.

Without thermocycles, the values of our control groups, such as flexural strength, flexural modulus, and microhardness number of Lucitone-199®, are in the same range of the ones reported in the literature [6, 14, 16]. Our results showed that adding 0.5–1 wt% of MWCNTs to Lucitone-199® enhanced the flexural strength (4–11%), resilience (15–30%), and bending modulus (27–33%) performance of PMMA. However, the addition of 2 wt% of MWCNTs to PMMA had no effect or even decreased the performance of PMMA. This unfavorable result for 2 wt% group may attribute to some improper dispersion of MWCNTs into PMMA monomer. For example, more agglomerations were found on the fracture surfaces in 2 wt% group. Agglomerations of MWCNTs could nucleate pores and other nonhomogeneous regions in the resulting nanocomposite. While individual nanotubes and perhaps even clumps of nanotubes can reinforce polymer matrices, clumps of such nanotubes also could have a detrimental role. An elevated number of MWCNT agglomerations likely contributed to the premature failure of the 2 wt% MWCNT specimens.

The mode of thermocycles in this work may differ from other studies; the results show a general trend that TC decreases in mechanical properties of the MWCNT/PMMA composites. The microhardness values have an inverse relationship to the increase of MWCNTs in PMMA. Large standard deviations in some test groups are believed to related to the amount of voids (Fig. 7.9) in the samples resulting from polymer and monomer mixing during denture base packing.

Clean adhesive fracture rather than cohesive fracture exists at the interface of MWCNTs and PMMA matrices on all samples. This indicates that there is no chemical bonding between MWCNTs and PMMA. Future improvement by adding functional groups on MWCNTs may be desirable to enhance chemical bonding between Lucitone-199® and MWCNTs.

## 7.5 Conclusions

The addition of MWCNTs at 0.5 and 1 wt% level to PMMA improves the mechanical properties of Lucitone-199®. Furthermore, the effectiveness of MWCNT reinforcement is dependent on the concentration of MWCNTs and the dispersion of the MWCNTs into PMMA matrix. Subsequent efforts need to be focused on dispersing MWCNTs into the commercial denture base system (both liquid and powder phases), and thereby allow testing of the effect of MWCNTs on denture base materials as it is prepared in clinics or dental laboratories. Additionally, surface treating or functionalizing the surface of the MWCNTs could improve dispersion and bonding within the denture base materials, which should lead to material performance enhancements that exceed those presented here. The results of this study show that further efforts to develop a clinically relevant material formulation with appropriate mixing techniques are justified given the benefits of nanotube reinforcement of the

present polymer matrix. Furthermore, the results obtained in this study have broader implications for other applications in dentistry and the medical industry.

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