Chapter-8

Summary and Conclusion

In this research work, we have reported on how to fabricate composites of polycarbonates-multiwall carbon nanotube (PC-MWCNT) and add protective coatings of multiwall carbon nanotube (MWCNT) layers on glass. Realizing the significance of newly discovered materials of carbon nanotubes having highly attractive mechanical properties, there was a need to exploit these in modifying easily available and workable low cost engineering materials like polycarbonates (PC) and glass. We achieve this goal by fabricating their composites and coatings and then characterizing these materials to ascertain if new modified materials qualify as enhanced materials for their role in dynamic impact resistance as well as in static load resistance. Such study required the use of state of the art experiments involving Split Hopkinson Pressure Bar (SHPB) set ups for dynamic loading and nano indenters for static loading. Further, this required an analysis of the samples by using modern characterization techniques like Scanning Electron Microscopy coupled with traditional tools like Infrar-Red and Raman spectroscopy. In the end, it also required an understanding of what goes into enhancement of the strength of composites at the microscopic level. We addressed all these issues.

We fabricated two types of materials- one as a composite material and the other as a coated specimen. In both cases, we used as synthesized- MWCNTs as a filler material to be used in minor composition. MWCNTs possess excellent mechanical properties. They are highly elastic and their Young's modulus is nearly 1TPa. Due to these properties, they can be proposed as filler materials to impart strength in other light weight and weak bulk materials.

For fabricating the composite, we chose the base matrix polycarbonate (PC) which is a polymer containing carbonate group and is of great commercial interest because PC being soft and light weight, can be easily worked upon, molded and
thermoformed. So our main goal was to modify PC by embedding modern materials like MWCNTs (which have become available only recently) in it. Similarly, glass based specimen were also modified by coating them with different amounts of MWCNTs layers. These coatings could serve as excellent materials to resist impact loads and protect the glass surfaces. We used simple chemical methods to fabricate composites and coated samples. We also used certain characterization techniques to validate the composition of these composites. The properties of the composite and coated materials were extensively studied by methods employed for mechanical characterization.

Different compositions of MWCNTs were used for both PC composite and glass surfaces. For dynamic load testing, Split Hopkinson Pressure Bar (SHPB) was used for both types of specimen which indicated dynamic load response of the specimen in the form of stress-strain diagrams. Study of these diagrams indicated the variation in resistance to external load with change in compositions of MWCNTs in both types of samples.

The composite specimens were also tested for static load response by using Nano-indentation technique. These experiments indicated on how the composition of MWCNTs affects the modulus and hardness of the composite specimen. Generalized expressions were also obtained to correlate dynamic and static properties with MWCNTs’ compositions in PC.

Finally, a model for pure PC and MWCNT-PC composite was generated to study how the mechanical properties were affected due to interaction of molecules. In the following, we present a summary of each of the Chapters for a quick overview.

In Chapter-2, we describe the methodology behind MWCNT-PC fabrication. Before proceeding with fabrication of composites, MWCNTs were characterized using Fourier Transform Infra Red (FTIR) and Raman spectroscopy techniques. The peaks at 1576/cm and 1176/cm in FTIR spectrum (Fig.2.1) were indicative signatures of MWCNTs and the peaks at 1338/cm and 1563/cm in Raman
spectrum (Fig.2.3) were indicative of the D and G peaks, respectively which are characteristics of MWCNTs. For composite preparation, we took the base material as PC. A simple technique of solution blending was adopted for preparing composites of MWCNTs and PC. MWCNTs were ultrasonically dispersed in a suitable solvent and then mixed with PC beads in the same solvent. Different compositions of MWCNTs in PC were obtained. We obtained compositions of 0.1, 0.5, 0.75, 1.0, 2.0, 5.0 and 10.0 wt % of MWCNTs in PC. These solutions of different compositions were then stirred to form a uniform dispersed solution which were then poured in separate glass petri dishes for drying. After drying, we obtained thin films of nearly 0.2mm thickness and each film was molded into a disc shaped specimen of diameter 10mm and height 5mm by pressing the films in a compression molding machine. This fabrication technique can be used to obtain compositions of various other compositions and shapes. As we had to test the specimen using SHPB so we fabricated disc shaped specimen.

In Chapter-3, we present the high strain rate loading procedure adopted on composite samples fabricated by us. Before testing composites for impact and static load testing, these were characterized using Raman spectroscopy and Scanning Electron Microscopy (SEM). The pre-impact images (Fig.3.9,3.10,3.11) of the composites showed a regular topography of the surface. After ascertaining the quality of the composites by SEM and evaluating the composition through difference in Raman peak intensity (Fig.2.4), mechanical characterization under high strain rate loading was done using SHPB. Such type of loading is generally encountered during explosions. This instrument consists of two horizontal cylindrical bars and the specimen of disc shape is sandwiched between them. One of the bars which is called the input bar is subjected to high pressure on one of its ends which causes the bar to move in the other direction (Fig. 3.1) and squeeze the sandwiched specimen with the output bar. The wave motion during this process is picked up by the strain gauges installed on both input and output bar and generates stress vs strain data for the complete process. All the composite
and coated glass samples were crushed at the end of this process, however the stress vs strain data varied for all these specimens. After analyzing the data for all the composites comprising different MWCNTs composition we found out that for a strain rate of nearly 2500/s, the stress resistance for composites comprising 0.1wt% to 2wt% MWCNTs enhanced consistently in comparison to pure PC. For a composition of 0.5wt% and 1wt% this enhancement was about 20% higher in comparison to the resistance offered by pure PC specimen for minor(10% to 20%) deformations. For 2wt% composites this strength did not change, however beyond 2wt% there was a fall in strength and at 5wt% it reached nearly the same value as that of pure PC. The strength for these minor compositions(less than 2wt%) was nearly 100 to 105MPa. The major reason suggested for the fall in strength at higher compositions was aggregation of MWCNTs to form localized bunches. Similarly, specimen comprising of functionalized-MWCNTs as reinforcement in MWCNT-PC composites were also tested and the resistance offered by these composites was compared with composites comprising of as synthesized-MWCNTs. It was observed and suggested, that although functionalized MWCNT fillers have stronger bonding with PC but they did not enhance the stress resistance under impact loads because of their shorter length and most of the impact load is taken up along the length of MWCNTs.

Apart from this, another important aspect was studied, which indicated that maximum and elastic deformation of the composites increased with increase in strain rate. This pattern was analyzed for samples of pure PC and with 0.1wt%, 0.5wt% and 1wt% MWCNT-PC compositions. All these samples indicated same behavior of increase in both elastic and maximum strain. This information provides important information in the context that for specific strain rate, the maximum deformation that the specimen undergoes can also be estimated. In case, the specimen has to be used in combination or as a part of an assembly with some other units, then the positioning and location can be decided owing to the maximum and elastic deformation which that specimen has to undergo. Post
impact SEM images (Fig.3.12-3.16) indicated irregularity and presence of MWCNTs bound with the base material.

Our SHPB experiments on composites revealed that minor compositions (0.5 wt% to 2 wt%) of MWCNTs in PC enhanced the strength so that PC can be used at high strain rates specifically for defense and aviation related applications.

Apart from composites, we also fabricated coated samples. This sample preparation process has been discussed in Chapter-4. We took the base material as boro-silicate glass of the same disc shape as 10mm diameter and 5mm thickness. After properly cleaning the glass surface, different amounts of dispersed MWCNTs were poured over their surface and dried. This resulted in forming a coat of variable thickness on the glass surface. We obtained different samples, with MWCNTs coating thickness in the range of 12 μm to 95 μm and the estimated number of layers on each sample varied between 3 and 19. This method can be used to fabricate coated samples of variable thickness and layers that comprise of random orientation of MWCNTs.

In Chapter-5, we discuss the high strain rate loading on coated glass samples. These impact tests were similar to the ones performed on composites earlier. The coatings on glass surface measured nearly 0.1 mg to 0.8 mg of MWCNTs which meant a thickness in the range of 12 μm to 95 μm. The results obtained from the impact studies revealed that for a strain rate of nearly 2500/s, minor coatings (0.1 mg to 0.2 mg) of MWCNTs enhanced the maximum stress resistance in comparison to pure glass surfaces. In comparison to pure glass, the samples which were coated with very small amounts of 0.1 mg and 0.2 mg MWCNTs exhibited 50% to 70% increased stress resistance. However, for thicker coatings this resistance reduced and remained nearly constant in comparison to 0.2 mg of coatings. This stress resistance was still 30% higher in comparison to the stress resistance offered by pure glass surface. For all practical purposes and calculations the thickness becomes more relevant instead of the coating amount.
Depending upon the thickness of coating required in resisting the impact, various types of sensors and absorbers can be designed. Slipping among the MWCNTs is suggested to be the main reason for the lowering of resistance at larger coating deposits.

Chapter-6 elaborates the static strength testing on composites. For static strength evaluations, we used a Hysitron T1 950 Tribol Indentor which evaluates elastic modulus and hardness of the specimen. This instrument consists of a very fine and hard tip which is forced to form an indent into the sample during loading process. After the indentation is caused, the tip moves away and reaches its initial position during the unloading process. Resistance offered to the indentation by the specimen is an indirect measure of its properties. The shape of the indent depends upon the type of indenter used. In our case, we used a Berkovich tip which is a standard three sided pyramidal probe having an included angle of 142.30° and radius of curvature about 150nm. This type of a tip resulted in a pyramidal shaped indent. The area and height of the indent caused gives a measure of hardness and elastic modulus directly.

We observed that for minor compositions of MWCNT-PC composites there was improved elastic modulus and hardness by nearly 50% in comparison to pure PC. Hardness increased consistently till 5wt% MWCNTs composition but beyond that it began to saturate and remained almost constant till 10wt% MWCNT composition. On the other hand, elastic modulus was found to increase consistently even for higher compositions(5% and 10%). The mechanism of increase in hardness was possibly due to stronger short range interactions between PC molecules mediated by MWCNTs and this mediation continued upto a critical composition of MWCNTs. As far as increase in elastic modulus was concerned, we suggest that the highly elastic nature of MWCNTs takes over at higher compositions causing reduction in hardness. Moreover, for higher compositions the elastic deformation becomes very difficult to achieve but once it is reached the composite will transit to plastic
deformation with ease. A competition between the two is covered by the saturation region from 5wt% to 10wt% MWCNT-PC composites.

In Chapter-7, we analyze all the results obtained in earlier Chapters on the basis of fitting most of the experimental data of stress-strain-composition to suitable polynomials both for dynamical and static loading to figure out important critical stresses and concentrations of interest that maximize the enhancement. Thereafter, we also report the results of a theoretical model for composites. Our proposed simple model was based on the assumption of a potential between constituent PC-PC or PC-MWCNT or MWCNT-MWCNT molecules interacting via their constituent atoms which take form of 6-12 Lennard Jones potential. We obtained an expression for total energy of the composite at various inter-particle separation distances and accordingly a minimum energy configuration was also obtained. The minimum energy configuration gave us shape of the potential function whose second derivative with respect to inter-particle distance gave us Young’s modulus. Thus we were able to model density, Young’s modulus and the energy of the composites with respect to their configuration. It was noted that this model was able to qualitatively interpret the properties of bulk PC and its composites. It was observed that for 5wt% MWCNT-PC composite, the cohesive energy increased by nearly 60% and Young’s modulus increased by a factor of two. Our aim was to understand in a simple way if the strength of the composite material gets modified in the manner the experimental observations were made. This calculation and model thus provided enough insight into the problem. There is scope for refinement of the proposed model and procedure to optimize the observed static and dynamic loading results for various mechanical applications.

In short, the work reported in this thesis highlights significant enhancement in dynamic as well as static mechanical properties of MWCNT-PC composites. The concentration of MWCNTs plays a key role in maximizing the advantages of composites. It has been found that less than 2wt% of MWCNT is adequate to maximize the dynamic characteristics whereas this concentration can be raised.
upto 10% to maximize the static characteristics of the MWCNT-PC composite. Therefore, the optimum composition of MWCNT in MWCNT-PC composite which leads to maximum enhancement in both static and dynamic mechanical properties for small deformations comes out to be nearly 2%. For glass coated samples also minor coatings of nearly 25µm proved effective in enhancing the dynamic strength significantly by 70% in comparison to pure glass surfaces. The coating thickness can be controlled to resist the pressure or impact load for protecting the base specimen. The experimental results on composites are also expressible in simple expressions and simple stress-strain relations give the numerical values at any given concentration of MWCNT in PC. A simple LJ potential model has also been successfully suggested to provide an insight into the enhancement effects of mechanical properties of static properties.

It is hoped that all the experimental work reported here will motivate the development of applications related to pressure sensing by using these composites which are low cost and simple to produce. It is also hoped that coated glass will lead to successful development of an energy absorbing device. It is also hoped that more theoretical work will be generated in the future to provide insight into the dynamic strength enhancement effects.