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Effect of High Impact Loading on Nanoclay Reinforced Polypropylene

Gebremeskel SA and Bhatnagar N*

Department of Mechanical Engineering, Indian Institute of Technology Delhi, Hauz Khas, New Delhi, India

Abstract

Except for few attempts to show the high impact load effect, the difference in level of contribution of nanoclay on polymers at their quasi-static and dynamic mechanical behaviors is a lacuna in the available literature. Quasi-static and dynamic responses of a high impact copolymer polypropylene (PP) in its neat and nanoclay-filled forms are presented in this study. The study helped in percentage quantification of differences in nanoclay effect, imposed by the very nature of loadings. The PP-nanoclay nanocomposite (PP+5 wt%nc) is prepared by melt compounding of 5 wt% nanoclay (nc) with PP using twin screw extruder. Tensile specimens for UTM (Universal Testing Machine) were prepared using injection molding machine. Dynamic loading specimens were made for SHPB (Split Hopkinson Pressure Bar) using an extruded sheet. The experiments were performed at strain rates of 10^{-2} s^{-1} and $2.2*10^{4}$ s^{-1} for quasi-static and dynamic loadings respectively. As a result, the contribution of 5 wt% nanoclay on PP at quasi-static loading is shown to be 2.6%, 10.8% and 13% on Yield stress, Young's modulus and toughness, respectively. However, significantly different results are observed after dynamic loading experiments. Intense improving contributions of 506% and 53% in impact modulus and impact toughness respectively are observed. While, minor reduction (3.5%) in plateau (Yield) stress is experienced in the case of dynamic impact loading.

Keywords: Impact; Nanocomposite; Polypropylene; High strain rate; Quasi-static

Introduction

The matter of rate dependency of many soft material systems, like polymers, is becoming a great concern for researchers on the area of materials science and engineering, nowadays. Soft materials, generally, demand to be individually characterized for all levels of loading rates. Varying the loading rates ranging from quasi-static loadings up to 'ultrahigh strain rate' impacts, Ramesh, may lead the materials to behave differently [1]. In this context, PP and its nanocomposite are studied at both quasi-static and 'very high strain rate' loadings. Zwick/Roell UTM machine for quasi-static test and an in-house developed SHPB (using Ti6Al4V bars), Gebremeskel, were used for this particular study [2]. The experimentation was ultimately to nominate possible candidate of matrix material for the development of a FRPC (fiber reinforced polymer composite) armour system, under a research project at IIT Delhi. Some literatures of importance related to the effect of loading rate on mechanical properties of neat PP and nanoclay reinforced PP are swotted. Majority of the studies reported that nanoclay improves most of the mechanical responses of PP at any level of strain rate. However, the effect of strain rate on the extent of improvement using the same nanoclay content on the same material was not addressed on the available literature.

Using tensile test (quasi-static loading rates) and flexural (low strain rate loadings), Saminathan studied EWF (essential work of fracture) of PP-nanoclay composite [3]. The nanoclay filled PP was reported to have 25% improvement in specific EWF compared to neat PP. Similarly, 9.3% improvement in tensile strength and 27.4% improvement in tensile modulus of H030SG PP as a result of 5 wt% of Cloisite20A nanoclay were reported by Sharma [4]. Depending on the types of processes and experiments used, particular types of PP and the clay particles the improvements reported in different studies are not one and the same. Shariatpanahi, for example, revealed improvements of 15% and 22% in tensile modulus and impact modulus, respectively, using INSTRON and low strain rate impact testers [5].

Quasi-static study performed by Cauvin considered 2 to 7 wt% of nanoclay and increase in modulus and yield stress of PP nanocomposite with an increase in nanoclay mass fraction was addressed [6]. On the other hand, Kim had similar experimentation and noted that the aspect ratio of the clay particles at higher weight fractions reduced due to bending and overlapping effects [7]. This in turn reduced the enhancement level in modulus of PP nanocomposite.

Nevertheless, the high surface area to volume ratio of nanoparticles is imperative as most properties of materials, like chemical, physical, mechanical and thermal properties, depend on surface interactions, Hussain [8]. On this regard, the van der Waals force nature of surface properties of PP molecules, Richard, helps to achieve better properties in its nanocomposite form [9]. The enhanced dispersion of fillers with no need of surface treatment, in case of PP, was also reported. Additional evidence was given by Sun stating that reinforcing polyimide (PI) and polypropylene (PP) with nano SiO2, the later was observed to have less crack areas [10]. Better interfacial interaction of PP-clay than Polyethylene (PE)-clay accounted for higher toughness of PP was evident.

An experimental study performed at higher strain rates, Boumbimba, used classical SHPB to characterize PP-organoclay nanocomposite [11]. Reportedly, the yield stress was shown to increase with increase in strain rate. Experiments were performed at dynamic strain rates ranging within orders of 10^{2} to 10^{3} s^{-1}. Investigation of PP-nanocomposite at 6 wt% organoclay and strain rate of $2.4*10^{3}$ s^{-1}, Wang , also depicted 19.20% and 26.90% improvements in yield stress and Young's modulus, respectively, than neat PP [12]. Lin , studied the effect of nano $CaCO_3$ particles on PP and performed annealing of the nanocomposite at 150°C for 2 hours which was found to improve modulus, yield strength and toughness [13].

***Corresponding author:** Bhatnagar N, Department of Mechanical Engineering, Indian Institute of Technology Delhi, Hauz Khas, New Delhi, India, Tel: 911126591139; E-mail: nareshb@mech.iitd.ac.in

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In addition to the ability of polymers to absorb and mitigate impact energy [14] the new, clay induced, mechanism of plastic deformation [15] boosts toughness of the nanocomposites. As per the report, the 'crazing and vein-type' failure mode of neat PP was changed to 'microvoid-coalescence fibrillation' mode, hence improved toughness of PP-nanoclay nanocomposite.

Presumably, the high impact loading was to affect the mechanical responses of polymers and could alter the contribution level of nanoreinforcements. Mechanical properties of polymers and polymer matrix composites obtained at quasi-static experiments are no more to be adopted for dynamic designs. Such softer materials behave differently at impact loading rates. In this study, PP and its nanoclay reinforced nanocomposite are experimented at both quasi-static and 'very high strain rate' impact. Thus, the effect of high strain rate loading on the contribution level of nanoclay to the mechanical responses of PP is revealed, where the existing literature lacks.

Materials and Methods

Polypropylene is considered in this study as one potential matrix material. It is deliberated in the development of FRPC (Fibre Reinforced Polymer Composite) armor material system in IIT Delhi research work. Particular PP 'Impact Co-polymer, REPOL C015EG' supplied from 'Reliance Industries Limited, India', was used. As per ASTM D-1238, the MFI (Melt Flow Index) of this typical PP is 1.5 g/10 min at 230°C. The nanoclay powder used as a filler, shown in Figure 1a, is an MMT (montmorillonite) designated as Cloisite® 15A. It was supplied from 'Southern Clay Products Inc., USA'.

Two material systems, Neat PP and PP+5 wt%nc, are well-thought-out in this study. The granules of Neat PP (Figure 1b) were melt mixed with 5% by weight of nanoclay fillers from which granules of PP+5 wt%nc (Figure 1c) were prepared using twin screw extruder machine (Figure 1d). Then an injection moulding machine (Figure 1e) was used to prepare tensile test specimens (Figure 1f) out of both material systems. Similarly, sheets of the two material batches were produced using microcellular extruder machine (Figure 1g). Out of the extruded sheet rolls like in Figure 1h, specimens for SHPB experiments (Figure 1i) were cut and prepared.

The necessary dimensions of tensile test specimens were made as per the standard for thermoplastic and thermosetting plastics, ASTM D 638. While the specimens for SHPB tests were made following the classical rules of the experiment and using calibration results. Square specimens of 6.5 mm×6.5 mm were prepared for both material systems. Thickness of the specimens of 350 μm and 310 μm were taken for Neat PP and PP+5 wt%nc, respectively, to achieve as close strain rates as possible. The 'quasi-static loading experiments' were done using a UTM machine shown in Figure 2a. However, the 'very high strain rate experiments' were performed using SHPB setup developed in-house (Figure 2b) [2].

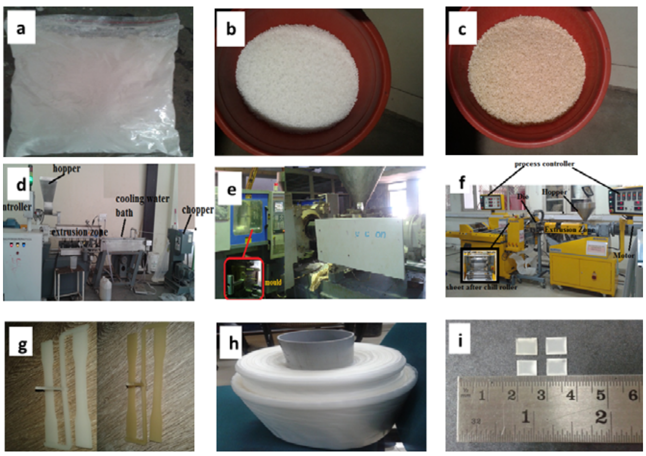

Figure 1: Materials, process methods and specimens (a) nanoclay powder (b) neat PP granules (c) PP+5wt%nc granules (d) twin screw extruder machine (e) injection moulding machine (f) sheet extruder machine (g) tensile test specimens (h) extruded sheet roll (i) SHPB test specimens

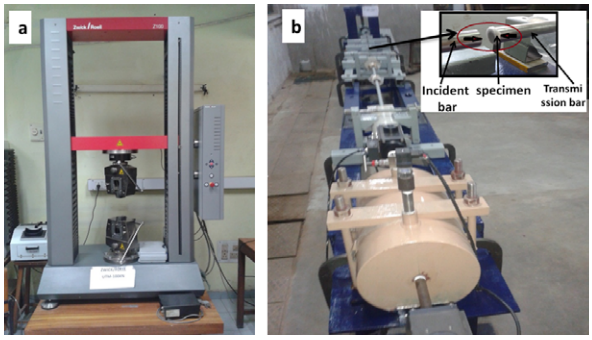

Figure 2: Experimental apparatuses (a) Zwick/Roell UTM machine, @IITD-AMD (b) SHPB, @IITD-MED

Three main mechanical properties, Plateau (Yield) Stress, Modulus of elasticity and Toughness, were investigated to compare the effect of loading type on the contribution level of nanoclay fillers in PP. Five specimens were tested for each material batch during both quasi-static and 'very high strain rate' loading experiments. Thus, an average true stress-true strain plots are generated. Modulus of elasticity 'E' is calculated as slope of the linear elastic region of the graph using eq. 1.

$$E = \frac{\Delta\sigma}{\Delta\varepsilon} \tag{1}$$

Where $\Delta\sigma$ (change in stress) and $\Delta\varepsilon$ (change in strain) are taken from any two points with in the elastic deformation region of the true stress-true strain graph.

In quasi-static loading experiment, the initial maxima of the graphs are considered as Yield stresses 'σ_y', as done for linear polymers including PP. In case of 'very high strain rate' experiments, the possible plateau stress after the start of yielding is considered as Yield stress. However, toughness 'u' is calculated as the area under the true stress-true strain curve using eq. 2.

$$u = \int_0^{\varepsilon_f} \sigma \, d\varepsilon \tag{2}$$

For the very high strain rate experiments on SHPB, certain classical theories and governing equations should be implemented to find mechanical responses of a material. The voltage pulses from the DAQ (data acquisition) system were changed to useful data of strain rate $\dot{\varepsilon}(t)$, strain $\varepsilon(t)$ and stress $\sigma(t)$ using the following equations.

$$\dot{\varepsilon}(t) = -\frac{2C_b}{L_s}\varepsilon_r(t) \tag{3}$$

$$\varepsilon(t) = -\frac{2C_b}{L_s}\int_0^t \varepsilon_r(t)\,dt \tag{4}$$

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$$\sigma(t) = \frac{E_b A_b}{A_s} \varepsilon_t(t) \quad (5)$$

Where, C_b is the elastic wave velocity in the bars, L_s is the sample thickness and A_S and A_B are the sample and bar cross-sectional areas respectively. ε_r and ε_t are reflected and transmitted strains measured from strain gages on the bars of the SHPB setup, respectively. The reflected strain is measured from the incident bar while the transmitted strain is measured from the transmission bar. The orientation of incident and transmission bars between which a specimen is sandwiched and impacted is shown in Figure 2b.

But, the integrals in eq. 2 and eq. 4 need to be approximated using suitable method. Thus, Trapezoidal rule of integrals is chosen as it can be used for numerical integrations including functions with weaker smoothness conditions resulting in faster convergence. Hence, eq. 2 is approximated to eq. 6.

$$u = \frac{1}{2}\sum_{i=0}^{n}\left(\sigma_i + \sigma_{i+1}\right)*\left(\varepsilon_{i+1} - \varepsilon_i\right) \quad (6)$$

Where, the subscripts 'i' and 'i+1' indicate the successive coordinate values of stress and strain throughout the curve. Similarly, eq. 4 is approximated to eq. 7 as;

$$\varepsilon(t) = -\frac{2C_b}{L_s} * \left[\left(\varepsilon_{r(n-1)} * t_{n-1}\right) + \frac{1}{2} * \left\{\varepsilon_r\left(t_{n-1}\right) + \varepsilon_r\left(t_n\right)\right\} * \left(t_n - t_{n-1}\right)\right] \quad (7)$$

Where the subscript 'n' indicates the reflected strain 'ε_r' corresponding to the n^{th} time 't'.

Results and Discussions

The effect of nanoclay reinforcement on mechanical properties of PP and other thermoplastic polymers is becoming an established fact as reported by many researchers, based on quasi-static and low strain rate loadings. However, no substantial study is reported on how 'very high strain rate', let alone 'ultrahigh strain rate', impact loadings alter the contribution level of nanoclay on the mechanical responses of PP. This study shows interesting results on how the level of effect of nanoclay become significantly different when the material is subjected to high impact loads than is in the case of quasi-static loadings.

Before the loading experiments were carried out the nanoclay distribution in PP+5 wt%nc was evaluated using SEM imaging by comparing with the Neat PP. Hence, the bulk morphology of Neat PP (Figure 3a) abetted to conclude that an almost exfoliated distribution of nanoclay, as shown in the bulk morphology of PP+5 wt%nc (Figure 3b), was achieved. Thus, both the material systems were tested at quasi-static and dynamic loading rates. The quasi-static strain rate was set to 10^{-2} s^{-1} while dynamic (very high strain rate) impact of $2.2*10^4$ s^{-1} was achieved. Details of the results are reported and discussed in the following subsections.

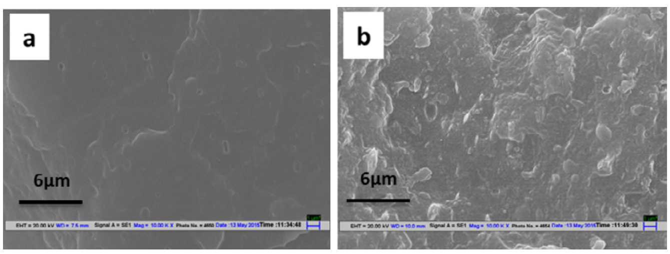

Figure 3: SEM images of specimens before loading (Mag. 10kX) (a) Neat PP (b) PP+5wt%nc.

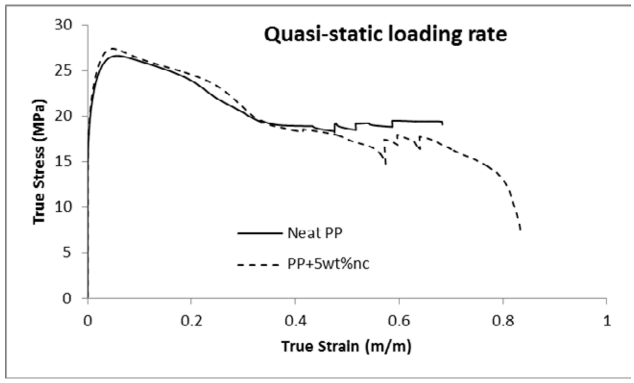

Figure 4: True Stress-True Strain graph of Neat PP and PP+5wt% nc at quasi-static rate of loading.

Contribution of nanoclay at quasi-static rate of loading

The true stress-true strain curve for the Neat PP shown in Figure 4 depicts that an elasto-plastic deformation started from about 20 MPa and an initial maximum (yield strength) 'σ_y' is found to be 26.7 MPa. Beyond the point of yield strength, Neat PP shows complete plastic deformation phenomena with smooth fall in stress. Some fluctuations in stress trend are observed within the strain range of 0.4 m/m to 0.6 m/m which is an indication of transition from end of pure plastic deformation to the beginning of constant load strain or strain hardening. The strain hardening region started from strain value of around 0.6 m/m and lasted up to the breaking strain value of around 0.7 m/m.

Considering the elastic deformation region of the curve, modulus of elasticity 'E' of Neat PP at quasi-static rate of loading is found to be 0.83 GPa. Moreover, the energy absorbed per unit volume or toughness 'u' is calculated as the area under the true stress-true strain curve using trapezoidal rule of integration and come out to be 14.56 MJ/m^3.

The responses of PP+5 wt%nc at quasi-static rate of loading, on the other hand, can be learned from its true stress-true strain curve shown in the same figure (Figure 4). Relatively steeper slope of the elastic deformation region resulting in modulus of elasticity 'E' of 0.92 GPa is achieved. The initial maximum of the curve (yield strength) 'σ_y' of 27.4 MPa, at which the elasto-plastic nature ends and pure plastic deformation starts, is ensued. The uniform plastic deformation continued up to strain value of around 0.5 m/m and a fluctuating region is observed within the strain range of 0.5 m/m to 0.7 m/m. This indicates a combination of some part of plastic deformation and some part of early initiated strain hardening beyond which pure strain hardening with fall trend of stress is observed and breakage occurred at around 0.83 m/m of strain. Relatively higher energy absorbed per unit volume (toughness) 'u' of 16.46 MJ/m^3 is also found after calculating the area under the curve.

The main mechanical responses of both material systems at quasi-static loading rate (10^{-2} s^{-1}) are presented in Table 1. As the nanoclay induces newer fracture mechanism to the physical nature of PP, more delayed failure in the case of PP+5 wt%nc than in Neat PP is observed. This failure delaying mechanism is the process of surface disintegration between the filler particles and the matrix. Hence, reinforcing PP with 5% by weight of nanoclay particles resulted in improving contributions of 2.6%, 10.8% and 13% on Yield stress, Young's modulus and toughness, respectively.

Contribution of nanoclay at very high rate of impact loading

The effect of high impact loading on contribution level of the nanoclay reinforcement is clearly reflected in Figure 5. The true stress-true strain curves of both Neat PP and PP+5 wt%nc at this 'very high rate of impact loading' show significantly different results than that of 'quasi-static rate of loading'. By noting the curve for Neat PP in Figure 5, an elastic region with relatively low impact modulus of 0.111 GPa persisted up to an elastic strain of about 0.6 m/m. However, its plateau (yield) stress of 347.80 MPa started at strain value of about 0.75 m/m and lasted up to 1.1 m/m. This shows a relatively narrow range of strain for the strength of Neat PP to sustain the impact loading. Consequently, the impact toughness (energy absorbed per unit volume) is calculated to be 198.10 MJ/m^3.

On the other hand, the true stress-true strain curve for PP+5 wt%nc in the same Figure 5 show greatly improved effects of the nanoclay reinforcement. Highly steep curve of the elastic region with impact modulus of 0.673 GPa is achieved. The relatively rigid nature of this material system, imposed by the fillers, causes the elastic range to end at a lower strain value of around 0.2 m/m. Though the reduction is not significant, lower plateau (yield) stress value of 335.52 MPa was recorded in PP+5 wt%nc. The material system is shown to persist for wider range of strain (0.35-1.0 m/m) with this value of plateau stress. Calculating the impact toughness, therefore, PP+5 wt%nc achieved remarkable value of 303.40 MJ/m^3. Observing both the curves of high strain rate impact, the impact modulus, strain and impact toughness values are filler dominated. While, the strength values (plateau stresses), showing no significant difference, are matrix dominated.

Main responses of both Neat PP and PP+5 wt%nc at a 'very high rate of impact loading' ($2.2*10^4$ s^{-1}) are presented in Table 2. Except the less significant reduction in plateau stress by 3.5%, PP+5 wt%nc remarkably outperformed Neat PP in both impact modulus and impact toughness. Thus, highly prominent improvements of 506% and 53% in impact modulus and impact toughness, respectively, are achieved.

Material system	Yield Stress (MPa)	Young's Modulus (GPa)	Toughness (MJ/m^3)
Neat PP	26.7	0.83	14.56
PP+5wt%nc	27.4	0.92	16.46

Table 1: Mechanical responses at quasi-static rate of loading.

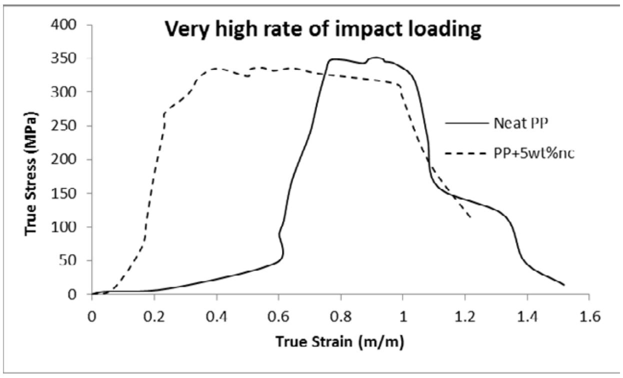

Figure 5: True stress-True strain graph of Neat PP and PP+5wt%nc at very high rate of impact loading

Material system	Plateau (Yield) Stress (MPa)	Impact Modulus (GPa)	Impact Toughness (MJ/m^3)
Neat PP	347.80	0.111	198.10
PP+5wt%nc	335.52	0.673	303.4

Table 2: Mechanical responses at dynamic rate (very high strain rate) of loading .

One should note that how loading rate could affect the behavior of rate dependent material systems, from the results discussed above. This study, particularly, revealed that the enhancement level of mechanical responses of PP due to the 5% by weight of nanoclay particle fillers is extremely changed at 'very high rate of impact loading'. Thus, rate dependent material systems primarily need to be characterized at specific level of loading rates for corresponding design purposes.

Conclusion

The study to develop appropriate matrix material system for FRPC (Fiber Reinforced Polymer composite) body armour at IIT Delhi reveals results of polypropylene (PP) based material systems. Effect of high strain rate impact loading on the mechanical responses of Neat PP and PP+5 wt%nc (PP compounded with 5% by weight of nanoclay) was deliberate. In addition, this study revealed that the contribution level of 5 wt% nanoclay on the mechanical behaviours of PP is significantly affected by loading rate. Hence, the percentage improvements of responses of PP+5 wt%nc over Neat PP at quasi-static loading rate and at 'very high strain rate' loading were found vastly different to each other. Considering the main mechanical responses, nanoclay fillers lead to improvements of 2.6%, 10.8% and 13% on Yield stress, Young's modulus and toughness, respectively, at quasi-static (10^{-2} s^{-1}) loading rate. In the case of 'very high strain rate' ($2.2*10^4$ s^{-1}), however, dramatic improvements in impact modulus (506%) and impact toughness (53%) are observed. While, slight reduction (3.5%) in plateau (Yield) stress happened. These results show that rate dependent matrix materials need to be characterized prior to designing FRPC body armour system for a particular threat perception. The same is true for other impact applications.

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