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# Novel HDPE nanocomposites containing aluminum nitride (nano) particles: Micro-structural and nano-mechanical properties correlation

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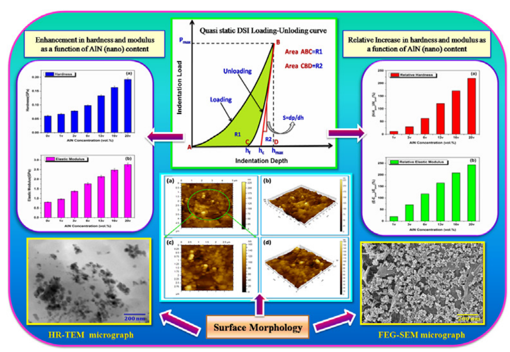
## HIGHLIGHTS

- 0-20 vol% of AlN (nano)/HDPE composites are melt mixed and characterized.
- Hardness & Elastic modulus improved ~219.30% and 242.42% at 5000 μN for 20 vol% AlN.
- Plasticity index reduced with increasing AlN (nano) content in pristine HDPE.
- Improved interfacial adhesion between HDPE polymer-matrix and AlN (nano) particles.
- Nano-mechanical properties and micro-structure analysis are correlated.

## GRAPHICAL ABSTRACT

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## ABSTRACT

The inclusion of inorganic nano-fillers with high mechanical strength in polymers could enhance efficiently the nano-mechanical properties of polymer-matrix (nano) composites. In this article, low-cost, high mechanical strength and stable crystal structured Aluminum nitride (nano) particles were incorporated into a High-density polyethylene (HDPE) thermoplastic polymer-matrix fabricated by melt-blending, compression moulding followed by hot pressing for investigation of nano-mechanical properties of HDPE/AlN nanocomposites. Nano-mechanical properties of HDPE/AlN (nano) composites (0-20 vol%) were measured by *Quasi-static nanoindentation* technique. A standard Berkovich indenter was used to make indentations under four different normal loads viz., 700, 1000, 3000 and 5000 μN, respectively. The surface topographies of the indented regions were monitored by AFM. The micro-structure of AlN (nano)/HDPE composites were characterized by intermittent tapping mode AFM, XRD, FEG-SEM and HR-TEM. The results showed that the homogenous distribution of AlN (nano) particles in the HDPE polymer-matrix affected the nano-mechanical properties of AlN (nano)/HDPE composites significantly. Nano-mechanical properties of the composites display substantial improvement with increasing AlN (nano) content in HDPE. The results are discussed with reference to AlN (nano) particles dispersion, interfacial adhesion and effective load transfer in AlN (nano) particles filled HDPE polymer composites.



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## 1. Introduction

In the last two decades, nanoindentation has developed into an increasingly popular mechanical characterization technique. As a novel and advanced technique, nanoindentation also known as depth sensing indentation (DSI), can give a wealth of important quantitative information concerning the mechanical properties, including plastic, elastic and visco-elastic properties of various polymeric composite systems [1–4]. This technique relies on the local deformation induced on a material surface with a sharp indenter with precise geometry under the application of a specific load [5]. On comparison with other methods of mechanical testing in the sub-micron range, nanoindentation has a relatively simple instrumental setup and easy sample preparation process. One of the main advantages of nanoindentation is that at specified locations on the material surface the nanoindenter could probe the mechanical properties of the materials and measured properties could be correlated quantitatively with the micro-structural properties of the material. In addition, nanoindentation leaves a small imprint on the material surface and is commonly perceived as relatively nano-destructive [6].

The incorporation of inorganic nano-fillers into thermoplastic polymers is scientifically well explored, the reduction in dimension of the inorganic constituent into the nano-size, and the simultaneous increase of the interfacial area, results in new extraordinary materials properties which needs further research. Over years, considerable attention has been paid to the use of thermoplastic polymer/inorganic (nano) particles in a variety of technological fields particularly in electronic packaging [7–11]. Among the diverse categories of thermoplastic polymers, high-density polyethylene (HDPE) is important due to its unique combination of mechanical and thermal properties. HDPE is presently one of the most widely used commercial polymer. Its resistance to abrasion and corrosion, reasonable cost for processing and low energy demand make it ideal for numerous applications [8–11]. Extensive nanoindentation studies on various other polymers, including HDPE have been reported earlier [12–14]. On the other hand, among the various types of inorganic nanoparticles, aluminum nitride (AlN) nano-particles is attractive due to its high mechanical strength, high melting point, excellent thermal conductivity, low coefficient of thermal expansion, high electrical resistivity, low cost, lack of toxicity, and stable crystal structure [8,15]. Consequently, thermoplastic polymer (HDPE) reinforced with AlN nano-particles is considered to be a potential candidate for applications in electronic packaging [8].

Literature review reveals that, mechanical properties of polymer composites using nanoindentation have been performed using a variety of fillers such as carbon nano-tubes [16–19], graphene [20,21], nano-clay [5,22], nano-diamond [23], carbon nano-fiber [24], spherical nano-fillers [25–28] etc. Molazemhosseini et al. [29] investigated polyether-ether-ketone (PEEK) based hybrid composites reinforced with $SiO_2$ nano-particles and short carbon fibers using nano-indentation and nano-scratching methods. The composites were fabricated by melt-mixing process at 400 °C. Surface topography of the nano-indented regions was evaluated using atomic force microscopy. Tian et al. [30] investigated mechanical properties of polycarbonate (PC) reinforced with graphite nanoplatelets (GNPs) using *Quasi-static nanoindentation* technique. The authors reported an improvement of nearly 7% in modulus and hardness for 1 wt% GNP/PC compared to that for neat PC matrix. Shen et al. [31] studied the morphology and nanoindentation behaviour of the injection-molded specimens of PA6/clay polymer nanocomposites prepared by melt compounding process. Li et al. [32] performed nanoindentation and nanoscratch experiments on epoxy composites reinforced SWCNTs with varying concentration (0, 1, 3, and 5 wt %). The authors reported that elastic modulus and hardness of 5 wt% SWCNTs is enhanced respectively by 75% and 30%, compared to that for pure epoxy. Zhu et al. [33] reported that addition of silica nanoparticles into PVC polymer matrix improves the mechanical properties of the nanocomposites significantly. Rahman [34] and Das et al. [35] investigated nano-mechanical properties of graphitic nanoplatelet (GNP)/polymer composites via nanoindentation technique. From these studies one of the most common observations was that, the elastic modulus and hardness increases with increasing nano-filler concentrations. However, agglomeration of nano-scale filler reinforcements may become an issue leading to poor nano-mechanical properties for higher concentrations of nano-filler. Ahn et al. [36] found that introduction of organically modified $SiO_2$ nanoparticles in PVC matrix displayed excellent mechanical properties of the prepared nanocomposites. More recently, Flores et al. [37] studied local mechanical properties of modified graphene added HDPE nanocomposites using depth sensing indentation.

However, to the best of our knowledge, no systematic studies have so far been reported on the assessment of mechanical properties at the (nano) scale combined with the micro-structural characterization for AlN (nano)/HDPE composites. In view of the above, a systematic investigation has been carried out to investigate the reinforcing effect of AlN (nano) particles on HDPE polymer using *Quasi-static nanoindentation* technique. The micro-structural analysis of AlN (nano)/HDPE composites have been studied by X-ray diffraction (XRD), Field emission gun-scanning electron microscopy (FEG-SEM) and High resolution transmission electron microscopy (HR-TEM), respectively. The 2D/3D topography and phase analysis of various vol% loading of AlN (nano)/HDPE composites (0-20 vol%) has been investigated by intermittent tapping mode atomic force microscopy (AFM). The estimated nano-mechanical properties for various vol% loading of AlN (nano) are discussed with reference to the dispersion of AlN (nano) particles, interfacial adhesion and effective load transfer in the AlN (nano) particles reinforced HDPE polymer nanocomposites.

## 2. Experimental

### 2.1. Materials and nanocomposites preparation

Aluminium nitride (AlN) nanoparticles used in this study is of spherical shape (diameter 15–40 nm, density 3.26 g/cm$^3$) and has been procured from Sky Spring Nano, USA. High-density polyethylene (HDPE) is of commercial grade (density of 0.94 g/cm$^3$) supplied by Loba Chemicals (India). The weight-average molecular weight of HDPE is 127,000 g/mol. Polymer nanocomposites containing 0, 1, 3, 6, 13, 16 and 20 vol% of AlN (nano) particles with appropriate amount of HDPE polymer powder are prepared by melt-mixing in a 'Sigma-mixer' (M/S S. C. Dey & Co., Kolkata, India) fitted with temperature controlled roller blades having a volumetric capacity of 50 cm$^3$. In order to achieve a homogenous dispersion of AlN (nano) particles in HDPE, the two components are physically premixed before being fed in the sigma mixer. Melt-blending is performed at 150 °C with 50 rpm for 30 min. The resultant homogeneous mixture of HDPE and AlN (nano) is then slowly cooled to room temperature. Pieces cut from the solidified AlN (nano)/HDPE mixture are transferred to a stainless steel die and is subjected to hot compression moulding at 120 °C. After complete solidification under pressure, AlN (nano)/HDPE composite pellet is carefully taken out of the die.

### 2.2. Micro-structural characterizations

The phase identification of the samples are investigated by X-ray