

## Fabrication and Characterization of Micro- and Nano- $Gd_2O_3$ Dispersed HDPE/EPM Composites

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### 1. Introduction

Hydrophobic polymer mixed with  $Gd_2O_3$  can be used in nuclear industry as a neutron shield because of its neutron attenuating and absorbing property, while it was reported that the smaller particles dispersed polymer composites can enhance radiation shielding efficiency compared to larger particles dispersed ones.<sup>1-4</sup> However, preparations of such materials are difficult because of the poor dispersion of the fine particles in the polymer matrix. Surface modification of the nanoparticles is therefore required for the homogeneous dispersion of the particles in the polymer matrix.<sup>5-6</sup>

In this study, pulverization of the micro- $Gd_2O_3$  particles and simultaneous surface coating of the nanoparticles by polymeric surfactant low density polyethylene (LDPE) were performed by using one-step of high energy wet ball-mill. Dispersion and neutron shielding effect of the nano- and micro- $Gd_2O_3$  fillers in mixed polymer of ethylene propylene monomer (EPM) and high density polyethylene (HDPE) were examined.

### 2. Methods and Results

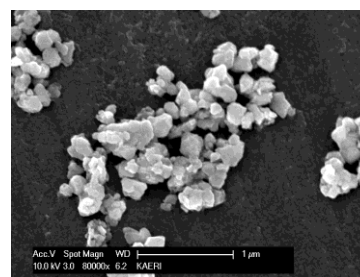
#### 2.1 Materials and Sample preparation

Nano- $Gd_2O_3$  was produced by MA (Mechanical Activation) using high energy ball milling. The surface of nano powder was coated with LDPE which was dissolved in the cyclohexane solvent to increase the degree of dispersion of the nano-particles in the melted polymer matrix. Prepared surface modified powders and HDPE/EPM thoroughly premixed by using a powder mixer prior to be blended in a polymer melting mixer with two roller blades (Eastern Engineering Inc. Rep. of Korea). The HDPE/EPM sheets (thickness ~ 3 mm) including nano- and micro-powders were fabricated by hot pressing at 190 °C and 20 MPa for 20 min after melt blended in the polymer mixer. The tensile strengths of the sheets were carried out in Hounsfield universal tester in accordance with the ASTM (American Standard Test Materials) D638. The Vickers microhardness was measured by MARK-3H instrument under a loading of 10 g and holding time of 60 s..

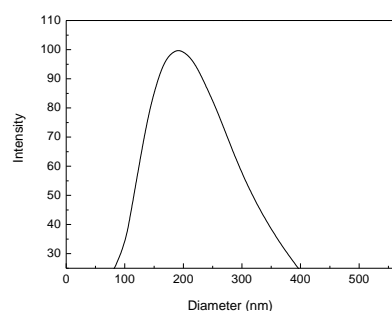
#### 2.2 Dispersion of nanopowders in polymer matrix

The wet ball mill process for synthesis of LDPE coated  $Gd_2O_3$  was performed by high energy ball mill system.

The color of as-prepared nano powders changes from white to light gray, after wet milling procedure was finished. It reflects the size variation from micron to nano size. The layer of LDPEs on the surface of  $Gd_2O_3$  may contribute to prevention of agglomeration between particles. This in-situ approach in this investigation looks convenient to produce the nanoparticles more finely during the short period time and also to treat or coat the surface of the nanoparticles using a polymeric surfactant simultaneously. The un-milled  $Gd_2O_3$  micron powder showed the particle size up to 10  $\mu m$ . As mentioned earlier, the nano powders was prepared by wet ball mill process at 700 rpm for 1 h. Morphologies of  $Gd_2O_3$ /LDPE powders obtained from the MA milling process are shown in SEM images in Figure 1(a). The size distribution was measured by particle size analyzer (PSA) and displayed in Figure 1(b).



(a)



(b)

Fig. 1. SEM images (a) for  $Gd_2O_3$ /LDPE nanopowder milled with cyclohexane at 700 rpm for 1h, and (b) PSA result after ball milling by high energy ball mill.

The SEM images containing nano- $Gd_2O_3$ /LDPE particles of 5 wt. % shows more homogeneous dispersion than that of micro-  $Gd_2O_3$  particles in HDPE/EPM. Homogeneous dispersion of nano- $Gd_2O_3$ /LDPE particles in the HDPE/EPM sheet was

confirmed. The SEM images also corroborate homogeneous dispersion of the  $Gd_2O_3$ /LDPE nanocomposite fillers in HDPE/EPM. Agglomeration of the particulate fillers wasn't observed. The average particle size for  $Gd_2O_3$ /LDPE was measured at 183 nm by BET specific surface area, which was well matched by TEM and SEM images.

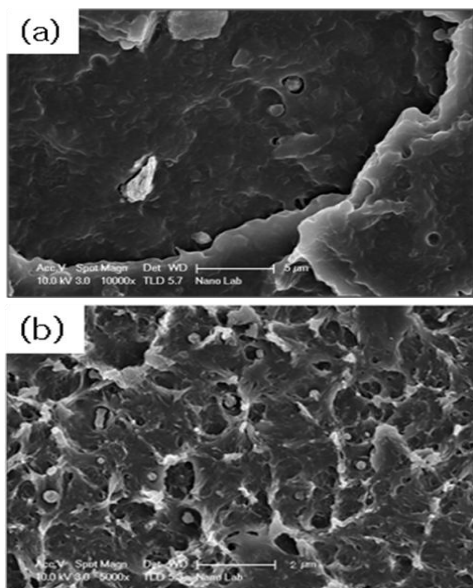


Fig. 2. SEM images for (a) micro- $Gd_2O_3$ /LDPE particles and (b) nano- $Gd_2O_3$ /LDPE particles in the HDPE/EPM composite sheet.

### 2.3 Mechanical properties

The tensile strengths of  $Gd_2O_3$  filled composites were evaluated based on the measurement of bending modulus and strength. The tensile strength slightly decrease with increasing both nano and micron filler concentration. The surface modified nanofiller by LDPE exhibits good reinforcement effect of HDPE/EPM.

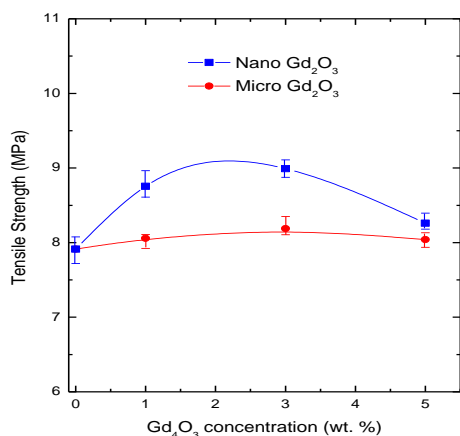


Fig. 3 Tensile strengths for (a) micro- $Gd_2O_3$ /LDPE and (b) nano- $Gd_2O_3$ /LDPE dispersed polymer sheet.

The thermal neutron shielding effect dependent of particle size were observed by means of measuring shielding ratio dependent on the thickness. The measurement was carried out at FCD beam port (neutron energy  $\sim 0.25$  MeV, wave length  $\sim 1$  Å and flux  $\sim 6 \times 10^5$  n/cm<sup>2</sup>/sec) in HANARO reactor. The  $Gd_2O_3$  nanoparticles dispersed HDPE/EPM sheets shows the higher neutron shielding effect than those for the micron composite powder dispersed ones.

### 3. Conclusions

Surface modified core/shell structured  $Gd_2O_3$ /LDPE was introduced into HDPE/EPM successfully. The sheets of HDPE composite containing nano- and micro- $Gd_2O_3$  particulate fillers were prepared respectively by hot pressing followed by melt mixing. Polymeric surfactant LDPE on the surface of nano-  $Gd_2O_3$  particles acts as a stabilizer for preventing agglomeration between the particles during ball milling as well as repressing growing of the particles in the polymer matrix. Consequently, this type of one step ball milling process can produce nano-sized ceramic powders as well as surface modification of nano-sized ceramic powder using polymeric surfactant simultaneously. The nano filler has a role of increasing both the mechanical properties and neutron shielding effect.

### ACKNOWLEDGMENTS

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