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Aug 10, 2010

Dear Dr. Sajjadi,

We are pleased to inform you that your paper entitled "Effect of nano-size calcium carbonate on short-term creep of polyethylene nanocomposite" (ID: G-025) has accepted for the forthcoming The 2nd International conference on nanomechanics and nanocomposites to be held in Beijing, China between 10th and 13th Oct 2010.

This letter serves as a formal invitation for you to attend the Symposium and give a poster presentation, which you may wish to use for visa application and funding purposes. We thank you for your contributions and support to this event, which we will strive to make it a fruitful and successful forum.

If you have any further quires, please do not hesitate to contact us.  
Look forward to meeting you in Beijing in Oct.

Best regards,

A handwritten signature in blue ink, appearing to read "S. A. Sajjadi".

## Certification

To whom it may concern

It is to certify that Dr. Seyed Abdolkarim Sajjadi has presented his paper entitled with "Effect of nano-size calcium carbonate on short-term creep of polyethylene nanocomposite" to the 2<sup>nd</sup> International Conference on Nanomechanics and Nanocomposites which is held in Beijing, China during Oct. 10-13, 2010.

Sincerely yours,



Prof. Shaoyun Fu

Chairman of the 2<sup>nd</sup> ICNN

10/11/2010

## Effect of nano-size calcium carbonate on short-term creep of polyethylene nanocomposite

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**KEYWORDS:** nano-sized calcium carbonate, MDPE, nano composite, creep

To evaluate the role of nano-sized  $\text{CaCO}_3$  on creep behavior of PE, a series of PE/ $\text{CaCO}_3$  nanocomposites were fabricated. To evaluate the dispersion of nanoparticles in PE matrix, scanning electron microscopy (SEM) was employed. To elucidate the creep behavior such as creep rate of pure PE and PE/ $\text{CaCO}_3$  nanocomposites, thermo mechanical analyzer (TMA60) was used. The creep test was done at constant load of about 20 MPa at different temperature i.e. 30, 60 and 90°C. The results illustrates that the time dependency of creep strain of PE and its nanocomposites can be divided into three stages. Firstly, rapid elongation would be observed at the beginning of creep test. For all materials initial strain is time independent. The interesting result achieved in the current study is, the initial strain of PE decreases as nano-size  $\text{CaCO}_3$  added to it. This is because elastic or even plastic deformation of PE would be decreased at the presence of  $\text{CaCO}_3$ . The second stage can be named primary creep step. In fact, in the primary creep stage, the creep rate ( $d\epsilon/dt$ ) decreases rapidly with time because of the slippage and reorientation of polymer chains under persistent load. After a certain period, the creep rate reaches a steady-state value and the secondary creep stage starts. At this stage viscous flow activates and in which a dynamic equilibrium of polymer structure evolution and external load is reached.

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# **Effect of nano-size calcium carbonate on short-term creep of polyethylene nanocomposite**

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**KEYWORDS:** nano-sized calcium carbonate, MDPE, nano composite, creep.

## **INTRODUCTION**

According to literature survey done by the authors creep studies on polymer composites can be categorized to two main groups the so called short-term and long-term creeps. For example short-term creep tests on the polymer concrete using an unsaturated polyester resin based on recycled PET were carried out to predict long-time creep behavior. The creep strain of PC concrete without filler is much higher than that of with filler, because the filler plays an important role in restricting the deformation of polymer. Also, as far as the type of filler is concerned, the effect of CaCO<sub>3</sub> as filler on PC is better than that of fly ash in terms of the creep strain and specific creep. This is attributed to the larger surface area of CaCO<sub>3</sub> particles and the higher adhesion between the resin binder and the aggregates. Pegoretti et al. used layered silicate to filled recycled polyethylene terephthalate (PET) obtaining a slightly decreased creep compliance and stable creep rate at long creep period [1]. Zhong et al. showed that the incorporation of a small amount of inorganic TiO<sub>2</sub> nanoparticles into a polyamide 66 matrix restricted the motion of polymer chains and therefore significantly enhanced the creep resistance of nanocomposites [2]. Since there is no evidence of any paper focused on role of nano-sized calcium carbonate on creep behavior of MDPE nanocomposites, the authors decided to clarify the subject which is still under debate. Thus, the main goal of this research is to find out the effect of nano-sized calcium carbonate with different content on creep behavior of MDPE nanocomposites.

## **EXPERIMENTS**

### **Material**

Medium density polyethylene (MDPE) was considered as matrix material, and was supplied by Tabriz Petrochemical Complex, Iran. The nano particles of CaCO<sub>3</sub> were Socal® PCC obtained from Solvay Advanced Functional Materials, Salin Degiraud, France. The detail of nano-sized CaCO<sub>3</sub> was reported in elsewhere [3].

### **Specimen preparation:**

To evaluate the role of nano-sized CaCO<sub>3</sub> on creep behavior of MDPE, a series of MDPE/CaCO<sub>3</sub> nanocomposite were fabricated. For this purpose nano-sized calcium carbonate and MDPE were first mechanically mixed in different weight ratios, to achieve desired composition. The premixed nanocomposites suspended in methanol as a media and the solution put in ultrasonic bath for 45 minutes to avoid agglomeration of nano particles and to improve the dispersion of nano-sized of calcium carbonate inside the

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matrix. Then the media was dried at 60-70°C. The nanocomposite powders were compressed under 20-30 KPa at 160-180°C for 5 minutes, followed by cooling to room temperature. Finally the samples with final dimensions 0.1\*0.1\*10 mm were fabricated. To evaluate the dispersion of nanoparticles in MDPE matrix, scanning electron microscopy (SEM) was employed.

**Thermo mechanical analyzer (TMA):**

To elucidate the creep behavior such as creep rate of pure MDPE and MDPE/CaCO<sub>3</sub> nanocomposites, thermo mechanical analyzer (TMA60) was used. The creep test was done at constant load of about 20 MPa at different temperature i.e. 30, 60 and 90°C.

### RESULTS AND DISCUSSION

Figure (1) show the fracture surface of MDPE/10wt%CaCO<sub>3</sub> (MDPE-10C) at different magnifications. Good dispersion of nano-sized CaCO<sub>3</sub> in MDPE matrix can be an evidence for adequate sample fabrication.

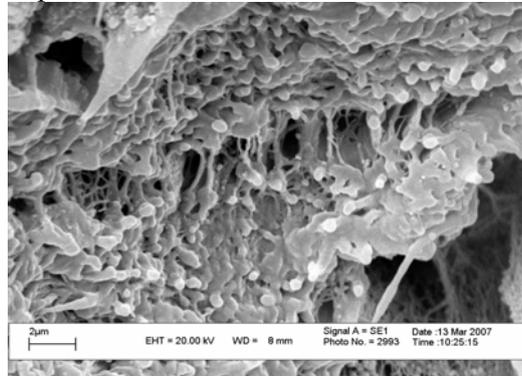


Figure 1 SEM micrograph of fracture surface of MDPE-10C nanocomposite

Figure (2) illustrates the variation of the creep strain of MDPE and its nanocomposite versus time at room temperature. As seen, the time dependency of creep strain can be divided into three stages [4]. Firstly, rapid elongation would be observed at the beginning of creep test. As it is clear for all materials initial strain is time independent. The interesting result achieved in the current study is, the initial strain of MDPE decreased as nano-size CaCO<sub>3</sub> added to it. This is because elastic or even plastic deformation of MDPE would be decreased at the presence of CaCO<sub>3</sub>. The second stage can be named primary creep step. In fact, in the primary creep stage, the creep rate ( $d\epsilon/dt$ ) decreases rapidly with time because of the slippage and reorientation of polymer chains under persistent load. After a certain period, the creep rate reaches a steady-state value and the secondary creep stage starts. At this stage viscous flow activates and in which a dynamic equilibrium of polymer structure evolution and external load is reached. It is worth noting that the duration of this stage is relatively long because of viscous flow behavior. Therefore, the tertiary stage has not observed. It means the failure of materials takes a very long period. As it can be realized from this figure, addition of nano-size calcium carbonate to MDPE matrix causes to decrease the creep strain and increase its dimensional stability. This is because the nano particles restrict the mobility of polymer chains. Indeed, inter-phase, bridging segments and junction of nano particles-matrix cause to improve the immobility of polymer chains [4].

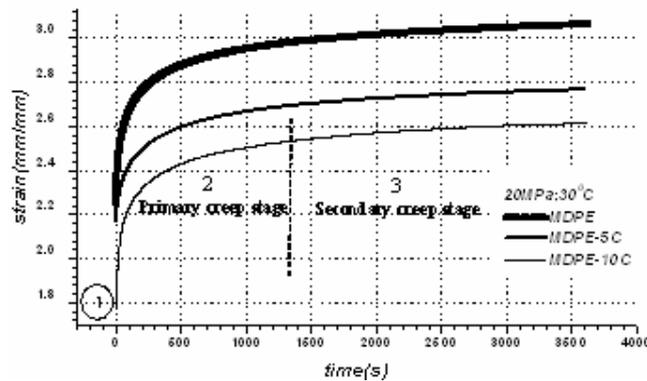


Figure 2: The variation of the creep strain of MDPE and its nanocomposites versus time at room temperature.

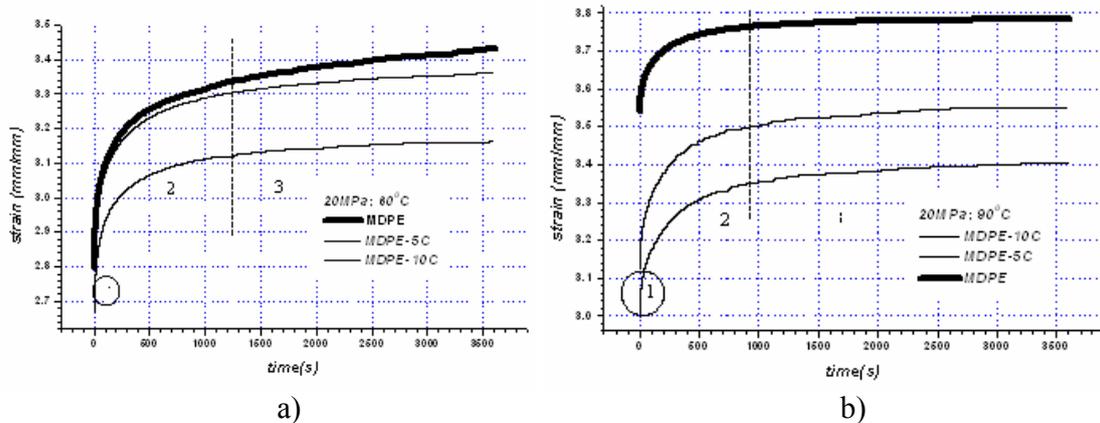


Figure 3: The variation of the creep strain of MDPE and its nanocomposites versus time at a) 60°C b) 90 °C

The variation of creep strain of MDPE and its nanocomposites versus time as a function of service temperature is shown in Figures (3-a) and (3-b). Similar to what seen in Figure (2), creep strain can be divided into 3 parts. Comparison of Figures illustrates that the creep strain strictly depends on service temperature and it can be increased as temperature increases. The reason of this variation can be referred to the mobility of polymer chains and thermally activation of polymer chains. Besides, the Figures illustrate that the creep strain of MDPE-10C is much lower than those of MDPE/5Wt%CaCO<sub>3</sub> (MDPE-5C) and neat MDPE. The true reason of this variation can be attributed to the fact that nano-size calcium carbonate hinder the mobility of polymer chains [4].

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