Thermal Analysis of B_4C dispersed Polyethylene

Jinwoo Jung, Young Rang Uhm, Jung Gu Lee, Donghoon Park, Chang Kyu Rhee and Jaewoo Kim*
Lab for Nano-Materials Applications, Nuclear Materials Research Division, Korea Atomic Energy Research Insititute
Daedukdaero 1045, Daejeon, Republic of Korea 305-353
**Corrresponding author: kimj@kaeri.re.kr*

1. Introduction

Neutron were generated from the spend fuel in a container or storage, nuclear reactor, etc. It inflicts a bodily injury on a person. Therefore neutron shielding is extremely important to protect human body.

Boron and boron-compound generally used for thermal neutron shielding due to large absorption cross-sections [1]. Neutron shielding was enhanced by nanosized particles due to decreasing mean free path of neutrons colliding in the medium [2]. UHMWPE (Ultrahigh molecular weight polyethylene) has a superior effect on the neutron slowing down [3]. Moreover, easy to molding and, it has excellent characteristic of thermal and physical, etc.

In the study on property of a polymer, thermal characteristic is very important. The DSC (Differential Scanning Calorimetry) and TGA (Thermal gravimetric analysis) is a convenient and highly accurate method for performing thermal analysis on a wide variety of materials. The DSC is a technique for the energy necessary to establish a nearly zero temperature difference between a sample and a reference material. And, the weight loss with heating is a common phenomenon for polymers due to degradation and the loss of the residual materials. The weight loss on heating performed using the TGA [4-5].

In this study, we investigated the analysis of thermal characteristic on borated - UHMWPE by the DSC and TGA test. We will take up the thermal stability and other thermal characteristic of borated polymer neutron shielding material. Also, we would like to explain the fabrication process of nano - particle from micro - B_4C by mechanical alloy and fabrication of sheet form by the compression molding.

2. Methods and Results

2.1 Materials and Sample preparation

Nano - B_4C (Boron Carbide) powder was produced by MA (Mechanical Alloy). Micro - B_4C (~ 10 μm) powder was ground by a high ball mill under rpm, mechanical alloy operating hour. Balls into MA jar are 6 mm size steel ball and, the weight ratio of B_4C and PVA is equal. It was found from the result that optimizing conditions were 700 rpm, 50 min. In this condition, average particle size was 50 nm. Figure 1 shows the TEM image of Nano - B_4C and PVA (Poly vinyl Alcohol) by MA (Operating condition: 700 rpm, 50 min)

Nano - B_4C (MA treated) powders, Micro - B_4C (untreated) were mixed with UHMWPE. To make a sheet form of samples, it was pressed by hot press. In operating condition of hot press, pressure is 500 kgf/cm, temperature and operating hours are 200 ℃ , 20 min. Table 1 shows content ratios of the Nano - B_4C , micro - B_4C and Pure UHMWPE for sheet form fabrication of neutron shielding materials.

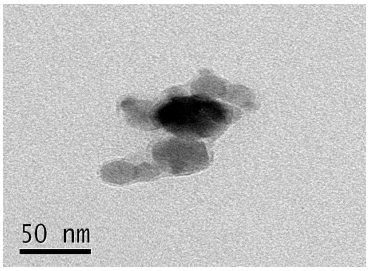

Fig. 1. TEM images showing Nano - B_4C and coated PVA

Table 1. The content ratio of sheet form samples

	B_4C	UHMWPE
Nano - B_4C	3 wt.%	97 wt.%
Micro - B_4C	1.5 wt.%	98.5 wt.%
UHMWPE	0 wt.%	100 wt.%

2.2. DSC/TGA Test

The DSC (DSC Q100, TA instrument Co. USA) samples were cut into small piece and machined using a mechanical grinder to maintain sample weight from 5 mg to 6 mg. The operating condition was a 10 ℃ /min heating rate from 30 ℃ to 250 ℃ in nitrogen (first run). The reason for choosing the nitrogen atmosphere is that the polymers never give any reactions by increasing the temperature in the nitrogen atmosphere. The sample was then cooled until 30 ℃ and a second run was performed under the same heating rate, due to characterize the post-processing product.

The TGA (SDT Q600, TA instrument Co. USA) sample weight from 11 mg to 15 mg. The operating condition was a 10 ℃ /min heating rate from 30 ℃ to 700 ℃ in nitrogen.

2.3 Thermal analysis (DSC/TGA)

The TGA test results are shown in Fig 2, Pure UHMWPE showed some higher thermal stability than nano - $\mathbf{B_4C}$, micro - $\mathbf{B_4C}$. However, as see the Fig 2, thermal stability of nano - $\mathbf{B_4C}$ and micro - $\mathbf{B_4C}$ almost corresponds to Pure UHMWPE. It means maintainance of superior thermal stability UHMWPE. In residue of samples, nano - $\mathbf{B_4C}$ residue (2.49 wt.%) was smaller than initial weight (3.00 wt.%), due to being melted the PVA.

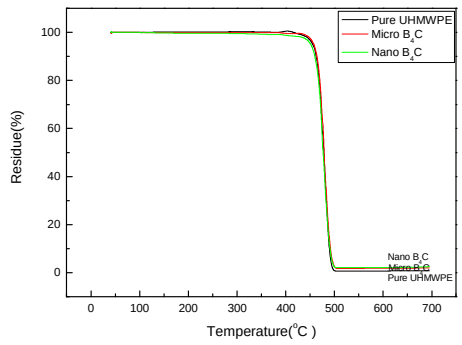

Fig. 2. TGA residue of nano, micro- $\mathbf{B_4C}$ and pure UHMWPE

Fig.3 and Table 2 show the melting point (T_m), the enthalpy of melting, degree of crystallinity (X_C), and residue of Neutron Shielding samples. The degree of crystallinity was calculated via the total enthalpy method according to the equation $X_C = \Delta H_m / \Delta H_m^+$, where X_C is the degree of crystallinity, ΔH_m is the specific enthalpy of melting of the same studied and ΔH_m^+ is the specific enthalpy of melting for 100% crystalline (288 J/g).

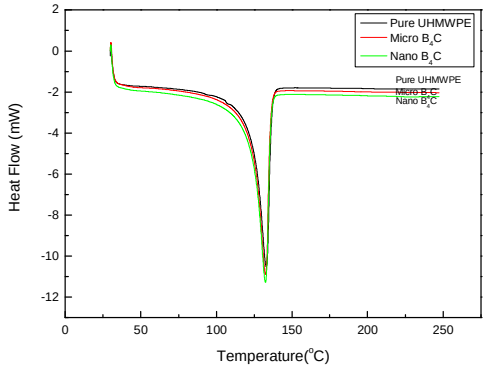

Fig. 3. DSC melting curve of nano, micro- $\mathbf{B_4C}$ and pure UHMWPE

The DSC analysis, melting temperature of nano - $\mathbf{B_4C}$ was smaller than micro - $\mathbf{B_4C}$ and pure UHMWPE. But, as the TGA results, the graph tendency of DSC nano - $\mathbf{B_4C}$ and micro - $\mathbf{B_4C}$ were almost corresponds to Pure UHMWPE. The $\mathbf{B_4C}$ content does not seem to have a few influence on the melting temperature of UHMWPE. The micro $\mathbf{B_4C}$ was the highest of the change of melting enthalpy degree of crystallinity and for the other samples.

Table 2. DSC/TGA results (1st, 2nd runs)

		Pure UHMWPE	Micro - $\mathbf{B_4C}$	Nano - $\mathbf{B_4C}$
T_m (°C)	1st	133.02	132.70	132.42
	2nd	132.79	132.62	132.53
ΔH_m (J/g)	1st	96.55	100.20	94.16
	2nd	101.90	102.18	95.94
X_C (%)	1st	33.52	34.79	32.69
	2nd	35.38	35.48	33.31
Residue (%)		0.81	1.99	2.49

3. Conclusions & Discussion

For fabrication of nano - $\mathbf{B_4C}$ and coated the PVA, nano size $\mathbf{B_4C}$ powders were fabricated by high ball milling system. We can recognize from MA test that optimized condition (700 rpm, 50 min, 6 mm steel ball) of $\mathbf{B_4C}$ powder. The particle size was measured by the TEM. TEM image reveals that coated PVA and particle size. The minimum size of nano - $\mathbf{B_4C}$ was 50 nm.

The Thermal property of $\mathbf{B_4C}$ composites with (nano and micro- $\mathbf{B_4C}$ particle) and pure UHMWPE were obtained using the DSC/TGA test. We can see from these tests that thermal stability, melting point, residue, etc. From the graph tendency of TGA/DSC, thermal property and the thermal stability of borated UHMWPE neutron shielding were preserved.

In additional, other weight ratio of nano - $\mathbf{B_4C}$ (1 wt.%, 5 wt.% and 10 wt.%) and micro - $\mathbf{B_4C}$ (0.5 wt.%, 2.5 wt.%, 5 wt.%) will be examined later. Also, it should also be added that the DMA (dynamic mechanical analyzer) test and flexural modulus test.

REFERENCES

[1] Yoshinori Sakurai "Development of neutron shielding material using metathesis-polymer matrix." Nucl Instr and Meth, A522, p455-561, 2004
[2] V. A. Artem'ev, "Estimate of neutron attenuation and moderation by nanostructural materials", Atomic Energy, 94(4), p282, 2003
[3] Soo-Haeng Cho, "Effects of Radiation Dose on Mechanical Properties of Resin-Type Neutron Shielding Materials", J. of Korean Ind & Eng. Chemistry, Vol.8, p.92-98, 1997.
[4] N.kaya, "Polymeric thermal analysis of C+H and C+H+Ar ion implanted UHMWPE samples" Nucl Instr and Meth, B261, P711-714, 2007
[5] J.A. Reyes-Labarta, "DSC and TGA study of the transitions involved in the thermal treatment of binary mixtures of PE and EVA copolymer with a crosslinking agent." Polymer, 47, p8194-820, 2006