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Damaged layer detection by nanoindentation for hydrogen ion irradiated polymers

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1. Introduction

Generally, a damaged layer in the materials can be produced by ion irradiation. The formation of damaged layer depends on a few physical quantities, for example, ion species, irradiation energy, and irradiation dose. In the range of a few hundred keV, heavy ion irradiation produces the damaged layer on the surface while light ion such as hydrogen and helium generates the damaged layer in the material as in the depth of a few micrometers. In fact, SRIM (Stopping and Range of Ion in Matter) can estimate the damaged layer when ion species, irradiation energy, and target materials are decided [1, 2]. Experimentally, SIMS (Secondary Ion Mass Spectrometry) and AES (Auger Electron Spectroscopy) can observe the composition of surface, and, thus, they can detect the damaged layer by peeling off the surface. However, they require somewhat complicate apparatus and process including ion sputtering, high vacuum, etc. In this paper, we show that a simple nanoindentation can judge if ion irradiation forms the damaged layer at the irradiation energy of ~ 100 keV.

2. Methods and Results

2.1 SRIM calculation

In this experiment, the target material is polycarbonate (PC) which is one of thermoplastic polymers. Since polycarbonate shows high impact resistance and good thermal resistance, it is applied to various engineering materials such as injection molding materials, information materials, vehicle frame materials and etc. On the other hands, weight lightening is a hot issue for automobile industry, because weight lightening yields many positive effects, for example, the better fuel efficiency, the faster acceleration, the shorter braking distance, as well as the smaller car exhaust. In fact, PC is requested for a candidate material of weight lightening due to its high impact resistance. However, PC has a low surface scratch resistance, and thus, many people have exerted an effort to overcome the drawback of PC. One of the way to enhance the surface scratch resistance is ion irradiation on PC surface [3, 4].

The selected ion for this experiment was hydrogen because hydrogen is the lightest ion in nature and thus, the small variation of the irradiation energy causes the large ion implantation depth. In addition, the width of the damaged layer by hydrogen ion is narrow compared to other heavy ions. Therefore, the hydrogen ion is one of the best ion to survey the damaged layer at the irradiation energy of ~ 100 keV.

In order to estimate the formation depth of the damaged layer, SRIM calculation has been performed. Here, we used the density of PC as 1.2 g/cm^3. Fig. 1 shows the total target displacement by the hydrogen irradiation as a function of depth. Green, blue, and red lines stands for the irradiation energy of 100 keV, 150 keV, and 200 keV, respectively. The maximum depth positions for 100 keV, 150 keV, and 200 keV are, 1.08 μm, 1.60 μm, and 2.28 μm, respectively, meaning that the higher irradiation energy forms the deeper damaged layer in the polycarbonate. In addition, the FWHMs (Full Width at Half Maximum) for 100 keV, 150 keV, and 200 keV are 0.17 nm, 0.19 nm, and 0.22 nm, respectively. This indicates that the higher energy irradiation causes the wider damaged layer in PC. In the SRIM calculation, the same number of irradiation ion has been used, so that the fact that the maximum total displacement value decreases with increasing irradiation energy is consistent with FWHM analysis.

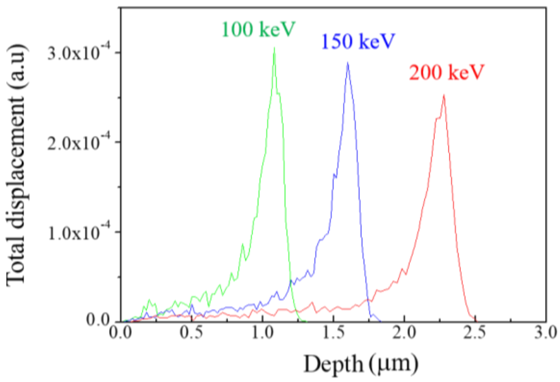

Fig. 1. Total target displacement by SRIM calculation for the hydrogen irradiation. The higher irradiation energy generates the deeper and the wider damaged layer in the materials.

2.2 Nanoindentation

The surface hardness of materials can be measured by a nanoindentation method. In order to measure hardness, the load of indenter tip should be increased and the indentation depth become deeper in accordance with the load of indenter tip. Note that the indenter tip in this measurement was a Berkovich tip. The indentation depth can be a few nanometer to a few micrometer where for the irradiation energy of ~ 100 keV, the ion range of every ion occurs in this depth. In this experiment, we used ultra nanoindentation tester (Model: CPX-UNHT) made by Anton Paar to detect a damaged layer.

In order to observe the damaged layer, a sinus mode has been adopted where the minimum load and the maximum load are 0.05 mN and 20 mN, respectively. The sinus frequency and the maximum sinus amplitude are 5 Hz and 1 mN, respectively. Fig. 2 displays the indentation load versus indentation depth for the different irradiation energy. Interestingly, when the indentation depth increases, there is a kink of the load at certain depth, indicative of the existence of the damaged layer by ion irradiation. In addition, the depth of the load kink increases with increasing the irradiation energy, which is consistent with SRIM calculation. However, the kink depth is less than the SRIM estimation. Since the nanoindentation measurement is affected by a beneath layer, the kink depth proceed the damaged layer. As SRIM calculation shows that FWHM increases with increasing the irradiation energy, the depth width of the kink also increases with increasing the irradiation energy. It is note that the depth width of the kinks for the 100 keV, 150 keV, and 200 keV are 0.08 μm, 0.14 μm, 0.19 μm, respectively. In addition, the slope of the load as a function of depth increases with increasing the irradiation energy, meaning that the surface hardness of PC increases with increasing the irradiation energy.

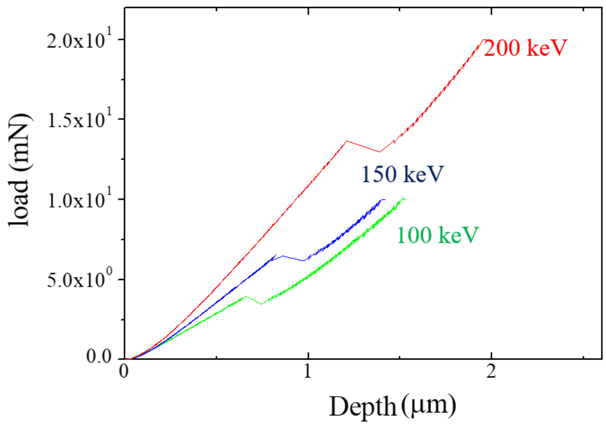

Fig. 2. Indentation load versus indentation depth. The higher irradiation energy produces the deeper indentation depth and the steeper slope of indentation load.

3. Conclusions

The damaged layer by ion irradiation can be observed by the nanoindentation measurement. In this measurements, the kink depths by the different irradiation energy are very consistent with the SRIM calculation. Moreover, the width of the kink depth also increase with increasing the irradiation energy. This is also estimated by SRIM calculation which shows that FWHM of the damaged layer by different irradiation energy increases with increasing the irradiation energy. Although the data were obtained by the hydrogen ion irradiation, we think that this results can be expanded on all the heavy ion.

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