

MICRO MECHANICAL AND THERMAL ANALYSES OF POLYMER TRANSCRYSTAL USING SCANNING PROBE MICROSCOPES

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Abstract

Micro mechanical and thermal properties of transcrystal of isotactic polypropylene were analysed using atomic force microscope and scanning thermal microscope, respectively. The relationships between morphology and these surface properties were investigated. At the interface of the transcrystalline layer, Young's modulus was evaluated as 0.1 GPa, and melting temperature were found to be lower in comparison with those of the transcrystalline layer itself. These results indicate that the crystallite size at the transcrystalline interface was smaller than that of the transcrystalline layer. In addition, the thickness of the transcrystalline interface was evaluated to be less than 2 μm from micro thermal analysis.

1 Introduction

In fiber reinforced polymer composites, transcrystallization of the matrix on the fiber surfaces is a well-known phenomenon. When nuclei are densely generated on the fiber surface, lateral crystal growth is restricted, then the crystallites are grown toward one dimension. As a result, lamellae are arranged in one direction from the fiber surface to the matrix, which is called as transcrystal (TC) [1]. There is a controversy about whether the presence of TC region is effective in the reinforcement of the polymer composites or not [2, 3]. To resolve this controversy, the relationship between morphology and properties of TC region should be clarified. Because of the restriction of the dimension of TC

layer (typically $\sim 100 \mu\text{m}$ thick), evaluation should be performed in the order of micrometer.

Scanning probe microscopes are well used for the purpose of micro analyses. Recently, we proposed the novel analytical technique on the surface mechanical properties of polymer materials evaluated by atomic force microscope (AFM) [4, 5]. In addition, scanning thermal microscope (SThM) was introduced in 1998. This can be useful to analyze the surface thermal properties of materials.

In this study, we analysed micro mechanical and thermal properties of TC layer and its interface (Fig. 1.) using AFM and SThM, respectively. We investigated the relationship between morphology and these surface properties from the microscopic point of view.

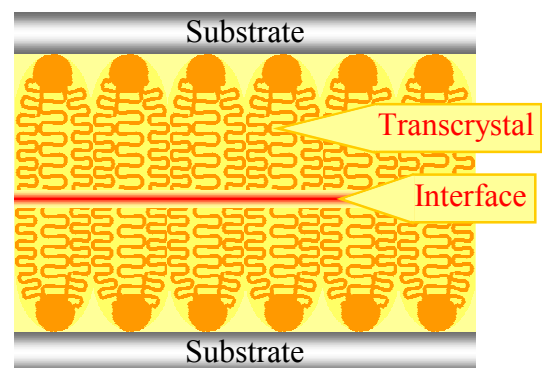


Fig.1. Morphological model of the transcrystalline *it*.PP.

2 Experimental

2.1 Sample preparation

Isotactic polypropylene (it. PP) (NOBLEN HD 100G2) used in this study was supplied by Sumitomo Chemical Co., Ltd.. *it. PP* was melted at

200 °C for 20 minutes, being sandwiched between two polytetrafluoroethylene sheets. Then it was crystallized at 135 °C for 90 minutes, followed by quenching in ice water (film thickness of 100 μm).

2.2 Measurements

Mechanical and thermal properties of the TC layer and its interface were analysed using AFM and SThM. Cross section of the sample film was subjected to a tensile load with the stretching device at room temperature attached to AFM (TopoMetrix, TMX-2100, Explorer) (Fig. 2. (a)). The melting temperature of transcrystalline layer and its interface was measured using SThM (TA Instruments, Micro-Thermal Analyser, μTA^{TM} 2990) (Fig. 2. (b)).

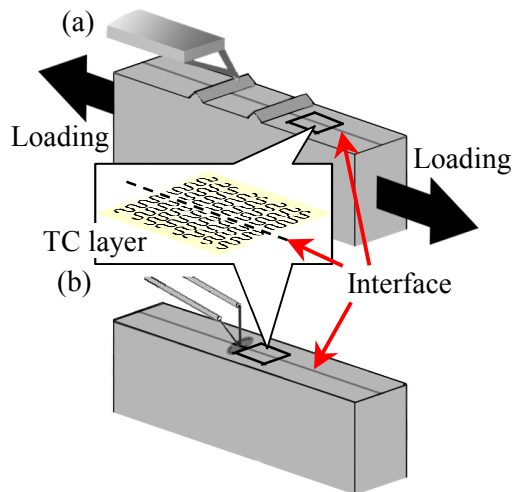


Fig. 2. Schematic illustrations of micro analyses using (a) AFM and (b) SThM.

3 Results and discussion

Figure 3 shows the stress-strain curves of the transcrystalline layer (TC layer) and its interface measured by AFM. The stress-strain curve of the macroscopic sample film (Bulk), measured by tensile tester, was also superimposed. Comparing the initial slope of the curves, Young's modulus of the bulk was smaller than that of TC layer. On the other hand, Young's modulus at the interface of TC layer was evaluated as 0.1 GPa, which was one tenth that (1.3 GPa) of the TC layer itself. This result is considered to be attributed to impurities and low-molecular weight components which condensed at the transcrystalline interface. Therefore, the reinforcement of the composites appears when TC layer dominates the macroscopic property. On the contrary, when the interface between TC layers plays major contribution, the macroscopic reinforcement is hardly observed.

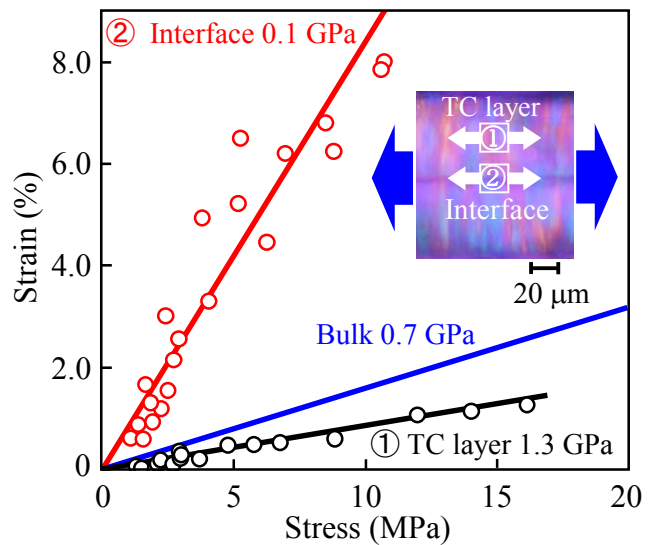


Fig. 3. Stress-strain curves of the transcrystalline *it.PP* evaluated by AFM and tensile tester.

Figure 4 shows the relationship between the melting temperature and the distance from the interface of TC layer evaluated by SThM. The melting temperature of the bulk evaluated by differential scanning calorimeter (DSC) was also superimposed with a dashed line. The melting temperature of TC layer (166 °C) was almost the same as melting temperature of the bulk. However, at the transcrystalline interface, the melting temperature was 6 °C lower than that of TC layer. This result indicates that the crystallite size at the transcrystalline interface was smaller than that of TC layer. The thickness of this interface was evaluated to be less than 2 μm .

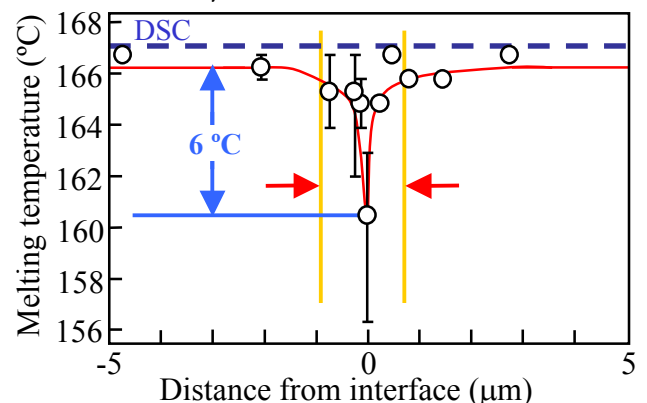


Fig. 4. Relationship between the melting temperature and the distance from the interface of transcrystalline *it.PP*.

4 Conclusions

We analysed micro mechanical and thermal properties of transcrystalline layer and its interface

using AFM and SThM, respectively. At the interface of the transcrystalline layer, Young's modulus was evaluated as 0.1 GPa, and the melting temperature were found to be lower in comparison with those of the transcrystalline layer itself. These results indicate that the crystallite size at the transcrystalline interface was smaller than that of the transcrystalline layer. The thickness of the transcrystalline interface was evaluated to be less than 2 μm . The microscopic evaluations of mechanical and thermal properties using scanning probe microscopes are found to be quite effective in discussing the reinforcement of the polymer composites.

References

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