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Nanostructure of isotactic polypropylene/ poly(methyl methacrylate) composite prepared in supercritical carbon dioxide fluid

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Introduction

The present study is devoted to establish a new method to prepare a nanocomposite of isotactic polypropylene (PP) with amorphous poly(methyl methacrylate) (PMMA) dispersed on nanometer scale in the amorphous phase of PP. In general, amorphous PP and PMMA cannot be well blended by the conventional method. In this method, supercritical carbon dioxide ($scCO_2$) was used as a processing medium to facilitate effective impregnation of methyl methacrylate monomer (MMA) into the amorphous phase of PP and, subsequently, the impregnated MMA is allowed to polymerize radically, *in situ*. The nanostructure of the composite thus prepared and the temperature dependence were analyzed mainly by means of small-angle X-ray scattering (SAXS). The details of the study were reported in the paper [1, 2].

Experimental

Synthesis Procedure for Nanocomposite PP

PP was commercial grade, having the number-average molecular weight M_n=11.1×10^4, M_w/M_n=8.4, triad micro-tacticity *mm* 0.96; *mr* 0.02, *rr* 0.02 and the melting point of 169ºC. The procedure for preparation of PP sheets and its blend with PMMA is described in the papers [1, 2]. PP sheet was soaked in the MMA/ AIBN / CO_2 solution for 5 min (Sample A: PP/ PMMA =100 / 123) and 1 h (Sample B: PP/PMMA=100/142) at 40 ºC and 6.3 MPa, followed by the *in situ* polymerizations of MMA.

Analysis of the Structure

SAXS experiments were carried out using synchrotron orbital radiation as an X-ray source set up in the Photon Factory of the High Energy Accelerator Organization at Tsukuba, Ibaragi, Japan. The wave length of the X-ray was SAXS was 0.1488 nm and the scattered intensity was detected with a one-dimensional position sensitive proportional counter (PSPC) with 512 channels. The details of the instrumentation and the procedure are described elsewhere [3].

Results and Discussion

Information regarding microstructural periodicity of the samples might be obtained by SAXS, assuming that the morphology would be globally isotropic but locally lamellar. Fig. 1 shows the plots of the Lorenz-corrected SAXS intensity Iq^2 vs. scattering vector q for the samples thus prepared. The original PP sample shows both the first- (q= 0.439 nm^{-1}, corresponding d=14.3 nm) and second- reflections, assigned to the long period (d) corresponding to the sum of thickness of a crystalline

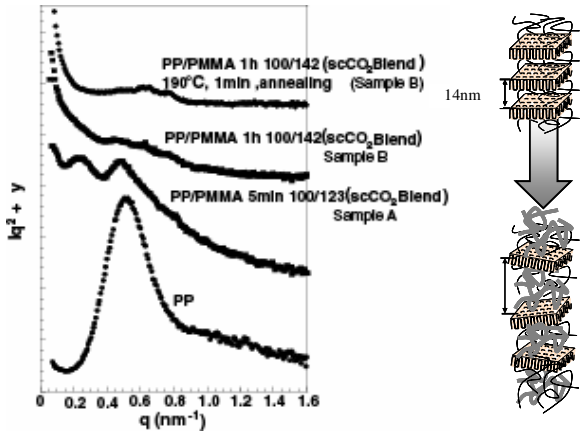

Fig. 1 Plots of Lorenz-corrected SAXS intensity Iq^2 vs. q, together with a schematic illustration for formation of $scCO_2$ Blend.

layer (PPc) and an amorphous layer (PPa) in a semicrystalline lamella. In the sample A, both intensities of the first- and second- reflection tend to become weaker, and a new maximum appears at q of *ca.*0.20 nm^{-1}, corresponding to a dimension of *ca.*30 nm and the scattering intensity in a q range smaller than 0.1 nm^{-1} clearly increased. This suggests that the dimension of a part of the lamella structures markedly increased by PMMA impregnated in PPa. The SAXS profile of Sample B shows that the structural development proceeds by long soaking time. However, annealing treatment of Sample B at 190 ºC for 1 min has brought about no further structural change (Fig. 1).

These results suggest that polymerization of impregnated MMA occurs in PPa and complicated entanglement of PMMA chains with amorphous PP chains occurs over various morphological regions, as shown in the schematic illustration of Fig. 1.

References

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