A new sample preparation technique for SEM observation of polyolefin microstructure

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In general, transmission electron microscopy(TEM) is usually used in the investigation of polymer microstructure. Microtoming, solution casting and carbon replica method are frequently introduced to the study of the polymer morphology with TEM, however the sample preparation procedure of those techniques is very difficult, and it takes a long time.

The purpose of this study is to develope a new sample preparation technique which is suitable for the observation and the investigation of the various shapes and species of polyolefin microstructure by scanning electron microscopy(SEM). By modifying the conventional chemical etching method, we developed a new chemical etching technique and sample prepartion procedure that are suitable for SEM study of polymer microstructure.

In this study the permanganate etching method is introduced and the optimum etching condition are determined by simply adjusting the etchant formulation, the concentration and the etching time.. This technique has shown good reproducibility and its morphological results agree well with other works on various types of microstructures such as spherulite characterization of isotatic polypropylene (α/β), polyethylene and polypropylene copolymer characterization, and the study of lamellar growth pattern of unsheared or oriented materials. This technique has also been applied to the industrial fields for characterization of polyolfin film, automobile products and the others.

Reference

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- J.Varga, *J.Mater.Sci.*, 27, 2557(1992).
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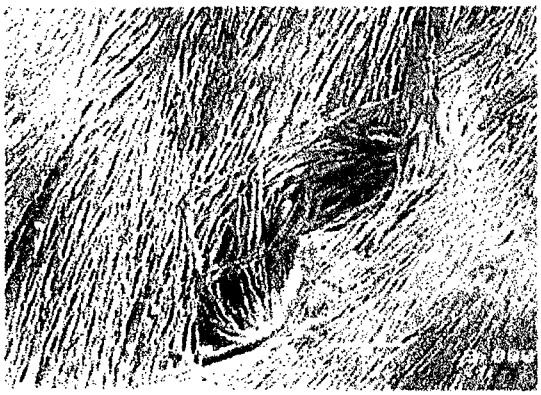
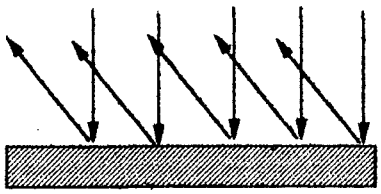
■ β-spherulite morphology of Isotatic Polypropylene

Banded Structure

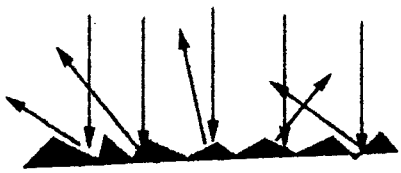


Rigid Growth

SEM 상의 iPP β-spherulite Morphology로 구정성장 Pattern과 Brightness로 Characterization



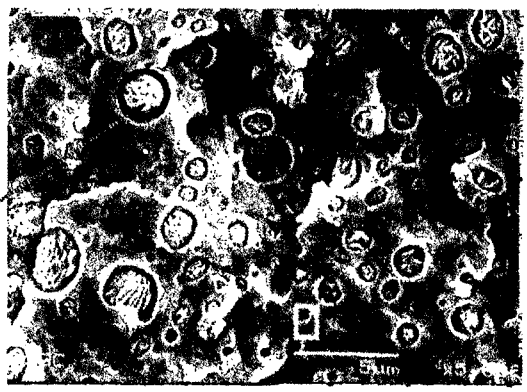
α-phase → dark image



β-phase → bright, Twisted image

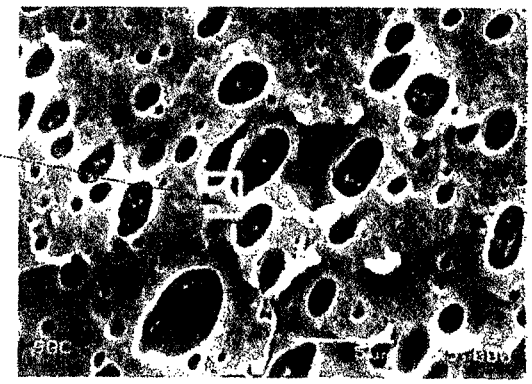
■ *Block PP Morphology Characterization*

Post reactor Blend →



Block PP / PE Blend

Block PP Morhology
(x30,000)

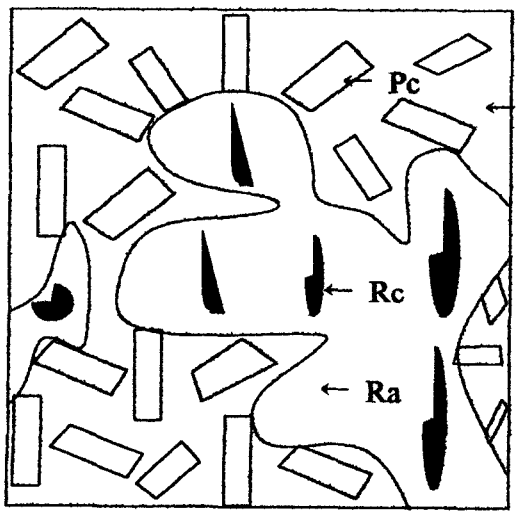


Block PP / Rubber Blend

PER Size distribution & Characterization

▶ Crystalline EPR (실타래 모양의 PE 구조확인)

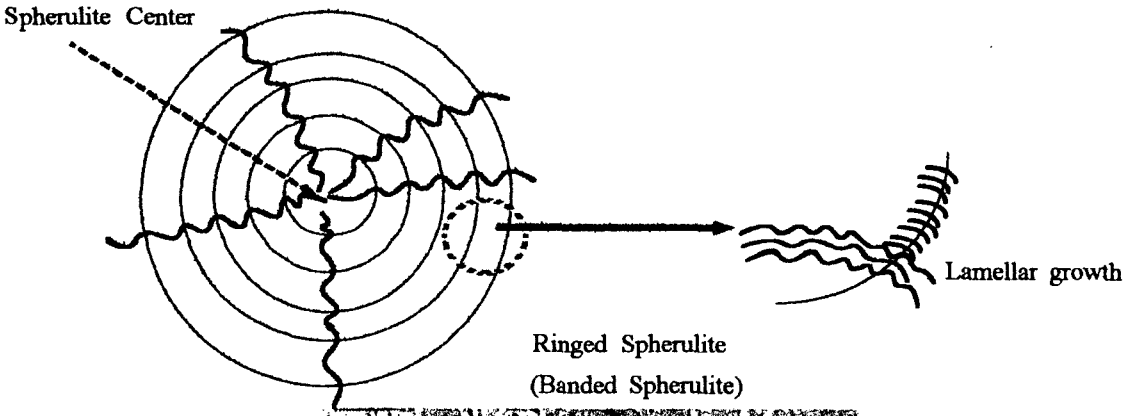
▶ PER Morphology Characterization



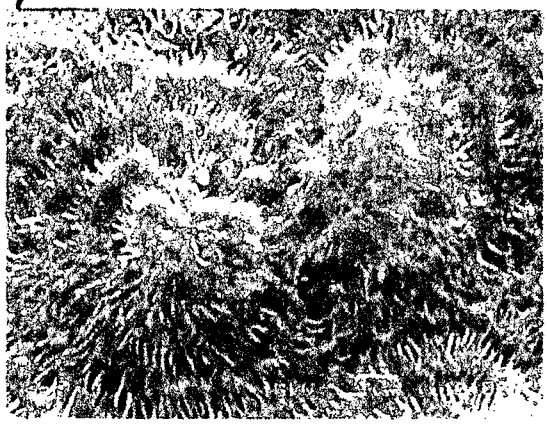
Es : EPR중 일부용해
Ra : EPR 상
Rc : EPR중 결정상
Pa : PP 비결정부
Pc : PP 결정부

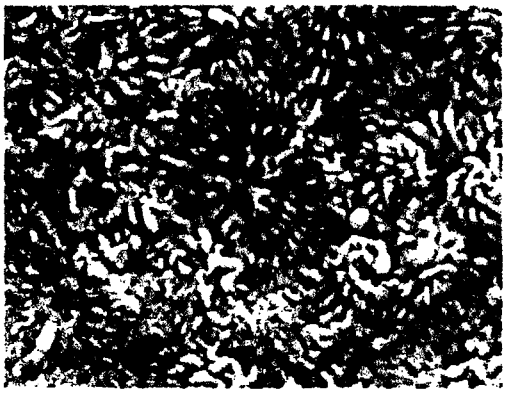
Morphology Model

■ *Banded Spherulite of Polyethylene*

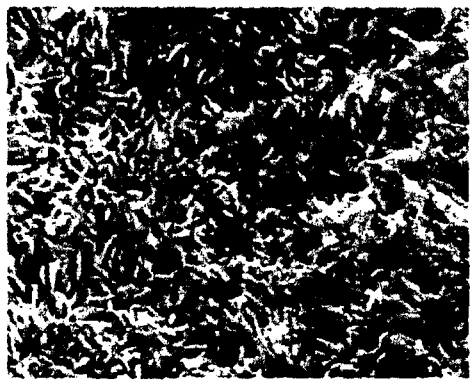


Ringed Spherulite
(Banded Spherulite)





LLDPE(x10,000)



LLDPE/HDPE blend (x10,000)

Spherulite Growth : 비스듬이 기울어진 방향 (the obliquity of growth direction)으로 성장

① lamellar가 uniform한 thick concentric shell로 성장하여

② 각 cell의 thicknesss가 각각의 ring사이의 동일한 radial spacing을 이루어 나타나는 것

③ Shell의 수와 thickness는 등온결정화 시간에 비례

④ Crystallographic c-axes 방향으로 주어진 radius에 대하여 유사하게 oriented 된다.