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Isothermal Crystallization behaviour and morphology of Poly Phenylene Sulphide PPS using Hot Stage Optical Polarizing Microscope

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Polyphenylene Sulfide (PPS) is a high-performance thermoplastic polymer widely used in engineering applications due to its excellent thermal stability, chemical resistance, and mechanical properties. The crystallization behavior and morphology of PPS significantly influence its performance characteristics. In this study, a Hot Stage Optical Polarizing Microscope (HSOPM)was utilized to investigate the isothermal crystallization and spherulitic growth of PPS at different temperatures. The morphological evolution of PPS was analyzed in real time at controlled isothermal conditions, specifically at 250°C, 252°C, 256°C, 258°C, and 260°C.

The results revealed that PPS crystallizes into well-defined spherulites, whose size and growth rate are highly dependent on the isothermal temperature. At lower crystallization temperatures, smaller and densely packed spherulites were observed, while at higher temperatures, spherulite size increased with reduced nucleation density. The radial growth of spherulites and the birefringence patterns under polarized light provided insights into the crystallization kinetics and molecular alignment within PPS. Additionally, the impact of thermal history and processing conditions on the degree of crystallinity and defect formationwas examined.

This study demonstrates that Hot Stage Optical Polarizing Microscopy is a powerful tool for understanding the crystallization behavior of PPS, providing critical information for optimizing its processing and application in high-performance industries such as automotive, aerospace, and electrical engineering. Further analysis incorporating nucleating agents and polymer blends could

enhance the control over PPS morphology, improving its functional properties.
Keywords: PolyphenyleneSulfide (PPS), Hot Stage Optical Microscope, Polarizing Microscopy, Isothermal Crystallization, Spherulite Growth, Polymer Morphology.

1. Introduction

PolyphenyleneSulfide (PPS) is a high-performance semi-crystalline polymer known for its exceptional thermal stability, chemical resistance, and mechanical strength, making it widely used in industries such as automotive, aerospace, and electronics. Thecrystallization behavior of PPS plays a crucial role in determining its final properties, and one of the key structural features formed during crystallization is spherulites. These radially symmetric crystalline structures develop during solidification and significantly affect the polymer's mechanical and optical properties [1].

To analyze the spherulitic growth of PPS, Hot Stage Polarizing Optical Microscopy (HS-POM) is an essential tool that enables real-time visualization of crystallization dynamics under controlled thermal conditions. By using polarized light, the technique enhances contrast in birefringent materials like PPS, allowing for the observation of spherulite nucleation, growth, and coalescenceas a function oftemperature and time [2].

In this study, the isothermal crystallization of PPS was investigated at different temperatures (250°C, 252°C, 256°C, 258°C, and 260°C) using HS-POM. The aim was to understand how crystallization temperature influences spherulite size, nucleation density, and radial growth rates [3]. Lower crystallization temperatures are expected to result in faster nucleation and smaller spherulites, while higher temperatures promote slower growth with larger, more well-defined spherulites.

This investigation provides valuable insights into the structure-property relationship of PPS, aiding in the optimization of its processing conditions for improved mechanical and thermal performance. Understanding the spherulitic growth mechanism is critical for tailoring PPS applications where precise control over crystallinity and microstructure is required [4].

2. Material and method:

Poly (Phenylene Sulphide) (PPS) is a prominent engineering polymer, of the high temperature and high engineering performance, with standing very high temperature (around 280–310°C). In general it exhibits borderline dual properties of thermoplastic and thermosetting. Basic structure of PPS consists of an aromatic core bonded to sulphur in the para position. This structure enables high crystallinity, dimensional stability and chemical and environmental resistance. PPS has special applications in electronics and, machinery when the unique combination of chemical inertness and excellent thermal and mechanical properties is desired. It is also used for special coating. It is produced by reacting para-dichlorobenzene with sodium sulphide and is processed mainly by injection at high temperature while the mold itself is kept at relatively high temperature. PPS, representing one of the most useful polymers among the premium engineering plastics. Poly (Phenylene Sulphide) (PPS) used in this investigation was procured from M/S Aldrich U.S.A. The melting temperature (Tm) observed from DSC of PPS is 284 °C.

Properties of Poly (Phenylene Sulphide):

Form: Off white powder,

Hardness: 93 (Rockwell M, ASTM D 785),

Viscosity: 275 poise (310 °C),

Melting point: 285 – 300 °C,

Density: 1.35 g/ml at 25 °C

The chemical structure of Poly (PhenyleneSulphide) is shown in Figure 1.

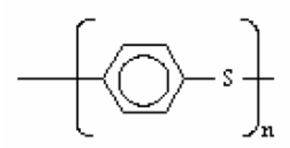

Figure 1. Chemical structure of Poly (Phenylene Sulphide)

Composite Preparation:

PPS was dried at 100 °C for 4 hours in air circulating oven, prior to further use .The PPS composites was prepared by melt compounding in a Haake (Rheomix 600) internal mixer at the co-screw temperature of 310 °C with residence time of 5 min. Neat PPS was also subjected to the same thermal treatment [5].

Optical Polarizing Microscope

The morphology development of spherulites, which appears as bright areas under polarized light in the dark background of neat PPS composites, was observed under cross polarizers using a Leica Laborlux 12 Pol S polar light microscope. The photomicrographs were taken with a Canon Powershot S 50 digital camera [6].

3. Results and discussion:

Crystallization behaviour and Morphological transition of PPS spherulite

Polyphenylenesulfide (PPS) is a high-performance thermoplastic polymer known for its excellent thermal stability, chemical resistance, and mechanical properties. Understanding its crystalline morphology is crucial, as it directly influences these properties. Hot stage polarizing optical microscopy (HSPOM), particularly when combined with polarized light, is a valuable technique for observing the crystallization behavior and morphological characteristics of PPS in real-time under controlled thermal conditions[7,8].

PPS typically crystallizes into spherulites—radially growing crystalline structures. The size and perfection of these spherulites can be influenced by thermal history, cooling rates, and the presence of nucleating agents.

Studies have shown that thermal curing of PPS affects its crystalline morphology. For instance, pure PPS exhibits spherulites approximately 40–50 µm in size. However, after curing, the spherulitic structure becomes less distinct, and a multitude of smaller crystalline entities emerge. This change is attributed to cross-linking induced by thermal curing, which acts as a nucleating agent, leading to a reduction in spherulite size [9,10].

The growth rate of spherulites is influenced by the crystallization temperature. Lower temperatures generally slow down molecular mobility, leading to slower spherulite growth, while higher temperatures can accelerate this process up to an optimal point beyond which growth may again decelerate due to reduced supercooling [11-14].

Variations in thermal history, such as different cooling rates or thermal treatments, can result in significant changes in spherulite size and distribution. Rapid cooling may lead to a higher nucleation density, resulting in smaller spherulites, whereas slow cooling typically produces fewer but larger spherulites Figure 2.

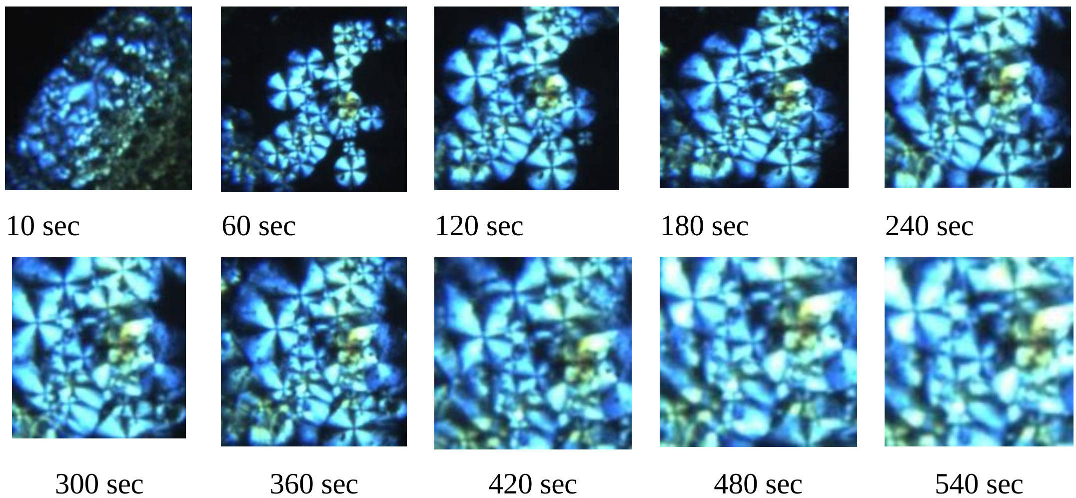

Figure 2. Hot stage optical micrographs of Poly Phenylene Sulphide PPS isothermally crystallized at 250°C with various time (sec).

4. Conclusions:

Hot stage optical microscopy serves as a powerful tool for elucidating the crystallization behavior and morphological characteristics of polyphenylenesulfide. By enabling direct observation under controlled thermal conditions, HSPOM provides valuable insights into how processing parameters influence the crystalline architecture of PPS, thereby guiding the optimization of its thermal and mechanical properties for various applications.

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