

Introduction

The XRD portion focuses on blends of polystyrene (PS) and polyvinylidene fluoride (PVDF). PS is largely amorphous, while PVDF is semicrystalline and can exist in several polymorphs such as α , β , γ , and δ . Because the two polymers are not fully compatible, the study examines how adding PS affects the crystalline structure of PVDF.

Objective

The main objective was to better understand PS-PVDF mixtures, specifically how the crystalline PVDF phase changes as the PS content changes. The XRD work aimed to determine whether the PVDF crystalline lattice survives blending and whether the crystal structure becomes distorted as more PS is added.

Experimental Procedures

The main PVDF diffraction peaks were located at:

$2\theta_1 = 17^\circ 40'$

$2\theta_2 = 18^\circ 32'$

$2\theta_3 = 19^\circ 57'$

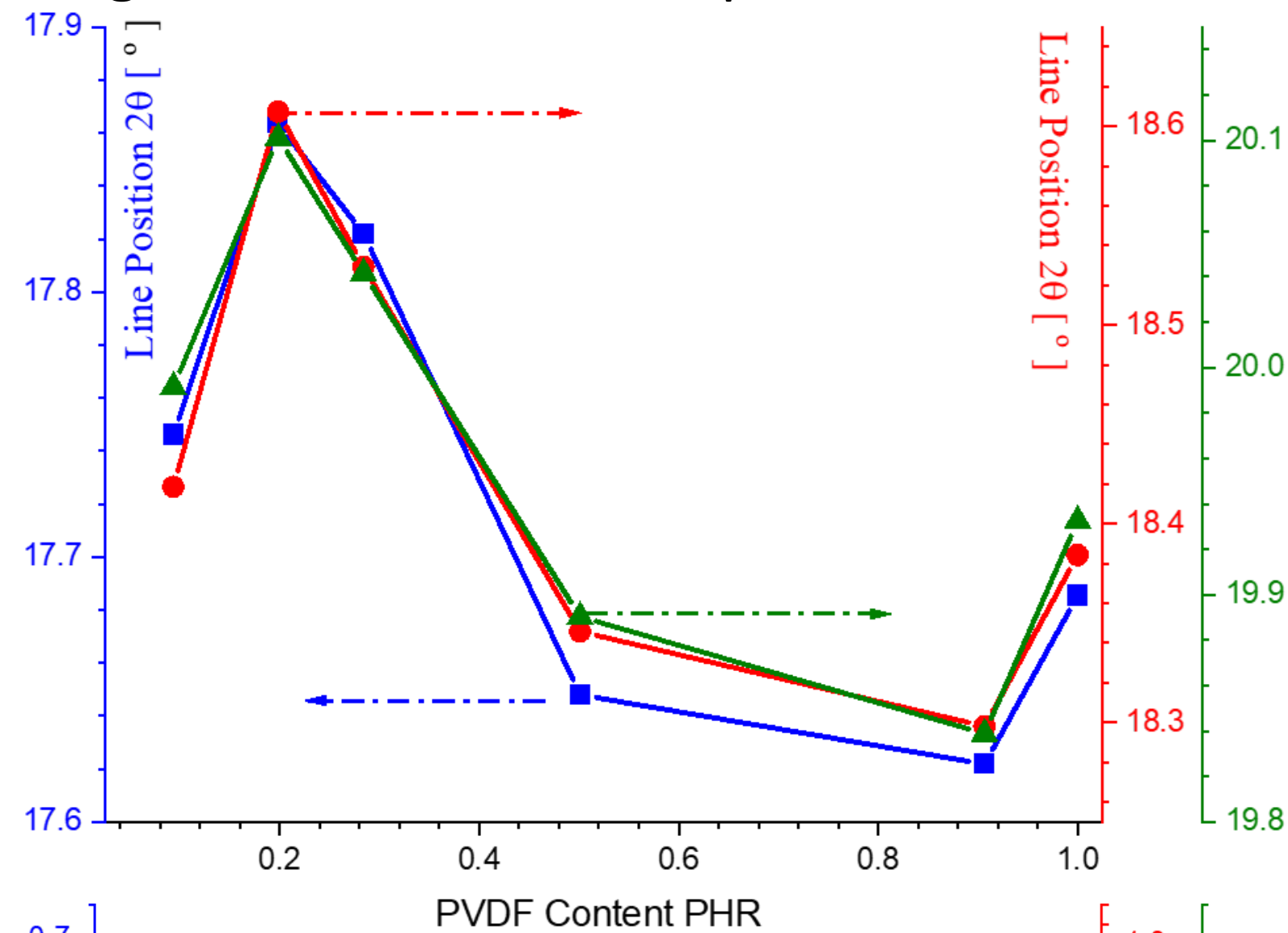
A weaker feature near $2\theta \approx 26^\circ 55'$

These peaks are consistent with a dominant α -phase PVDF structure. As the PS content increased, the PVDF peaks became weaker and broader, indicating that the crystalline PVDF structure was being disrupted by the added PS.

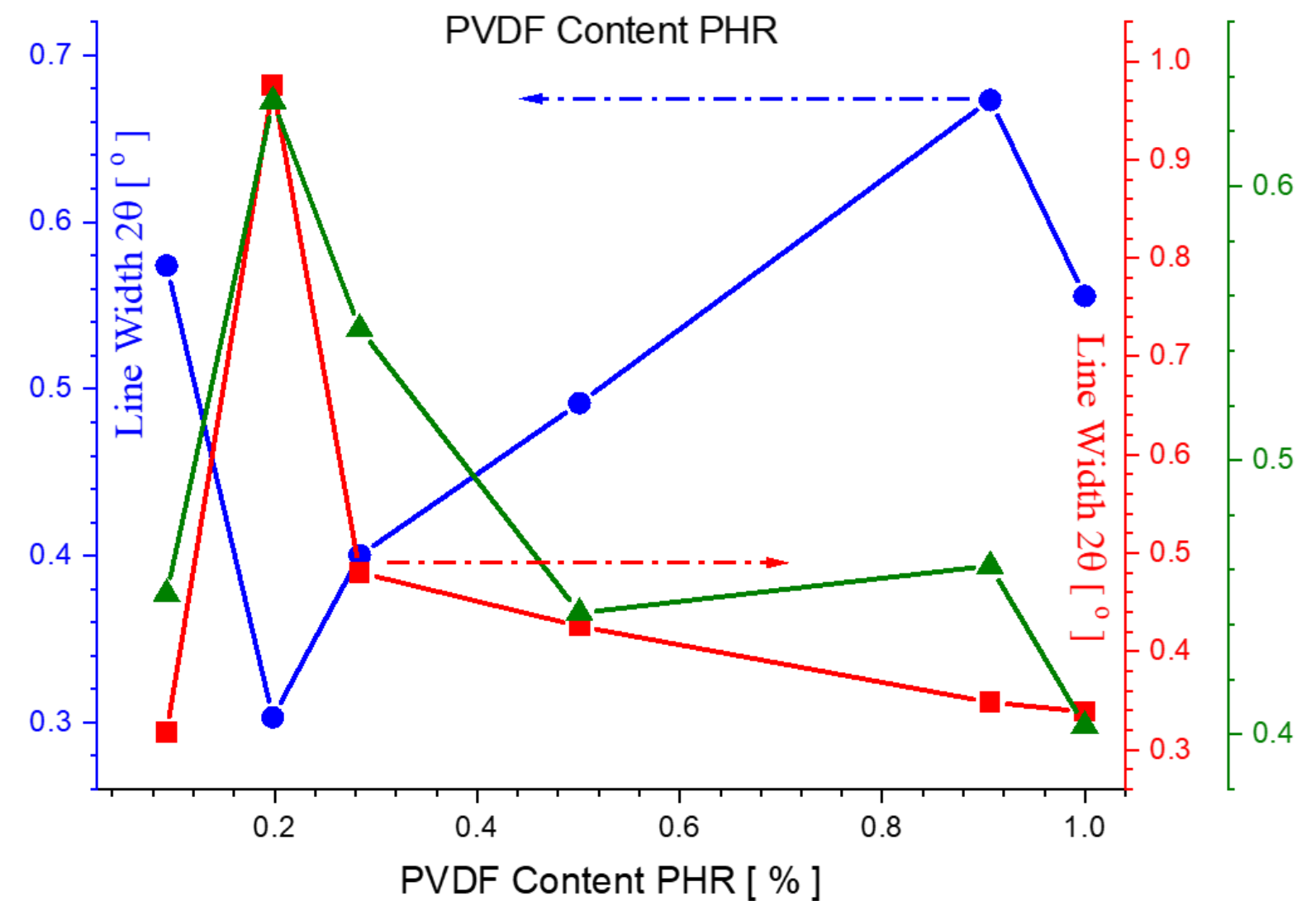
The line-position plots show that the diffraction peak locations shifted slightly with changing PVDF concentration. These small peak shifts suggest that the PS affects the PVDF lattice spacing and slightly distorts the crystal structure. The line-width plots show that the peak widths also changed with composition. Broader peaks generally indicate smaller crystallite size, more disorder, or lattice strain in the PVDF crystals.

The experimental XRD spectrum of a PS-PVDF blend (50% weight), black circles. The fitted spectrum (red line) for the 3 most intense PVDF reflections includes a broad contribution due to amorphous polystyrene. The fitting parameters were A = zero correction, B = slope correction, I1, I2, I3 = line amplitudes, W1, W2, W3 = line width, P1, P2, P3 = line positions, W4 = width of the broad line, P4 = position of the broad line, and I4 = amplitude of the broad line.

The agreement between experimental data and fitting was very good.



Left figure on top represents the dependence of the main line positions on PVDF content. It is observed that these lines have similar displacements, indicating that the lattice symmetry does not change.



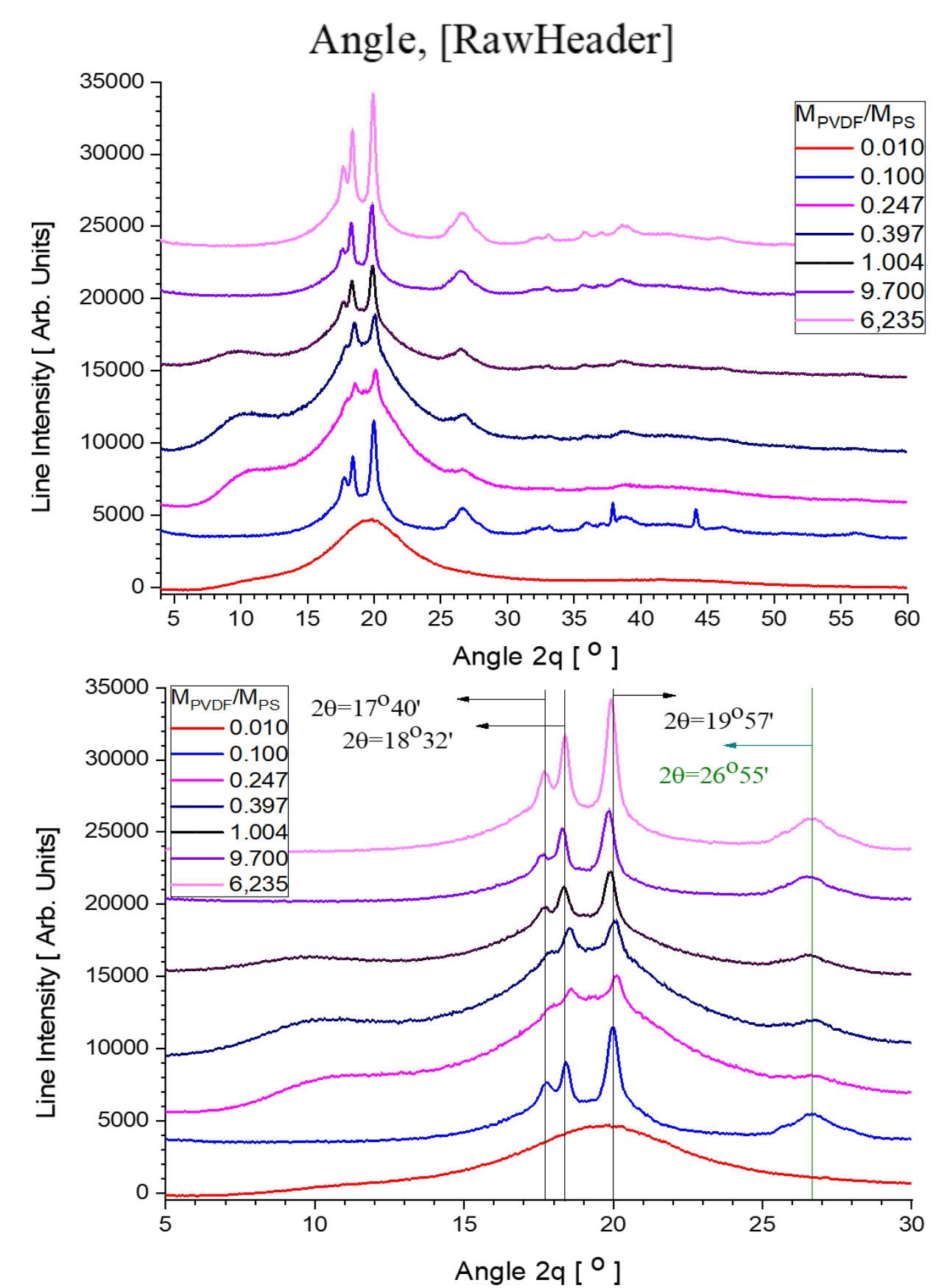
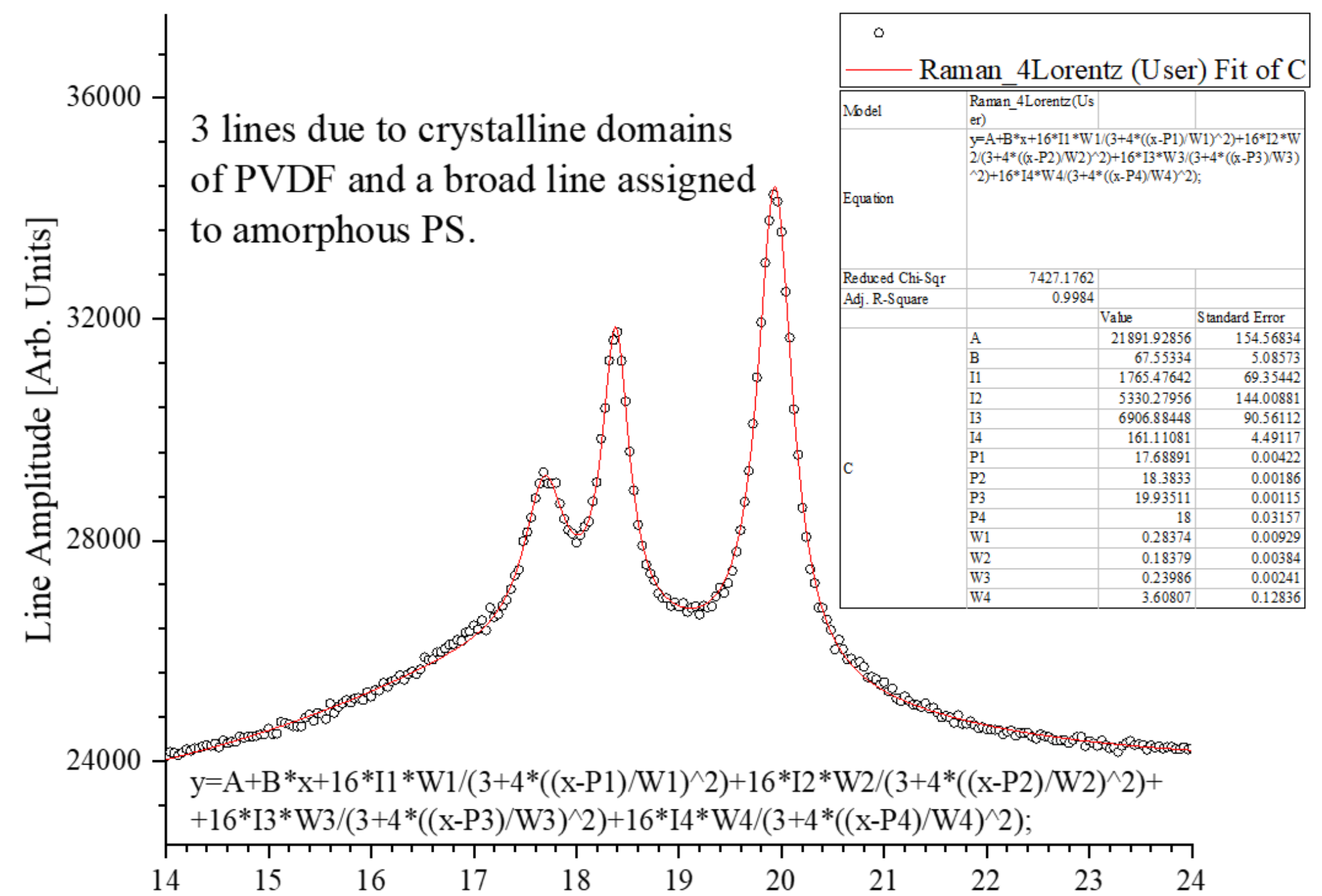
Left figure on bottom represents the dependence of the line width on PVDF content, revealing a complex change of the combined crystallites size and local strains.

Materials & Setup

- Atactic PS was purchased from Sigma Aldrich.
- PVDF was purchased by Alfa Aesar.
- They have been used as received.
- PS-PVDF blends, with various PVDF content, were prepared by melt mixing using a HAAKE PolyLab equipment with 2 counter rotating screws using a 4-step program (175 °C, 40 rotations per minute (RPM) for 5 minutes; 190 °C, 60 RPM for 30 minutes; 210 °C, 80 RPM for 15 minutes; and 180 °C, 60 RPM for 30 minutes).
- Atactic PS is completely amorphous.
- Wide Angle X Ray Scattering (WAXS) measurements were performed using a Bruker Discovery 8 Spectrometer.
- WAXS had been fitted in Origin Pro with one or more superimposing Lorentzian line, and corrections for the baseline (position, slope, and curvature).
- An excellent agreement between experimental data and computer-generated spectra was achieved.

Results & Conclusions

The XRD results show that PVDF remains crystalline even after blending with PS, but the crystalline order is progressively weakened as more PS is added. The dominant crystal form remains α -PVDF. The decrease in peak intensity, the broadening of the peaks, and the small shifts in peak position all suggest that PS distorts the PVDF crystal lattice and reduces the degree of crystalline order. Overall, the XRD results support the conclusion that PS and PVDF are not fully compatible and that PS interferes with the normal crystallization of PVDF.



References & Acknowledgments

- "DSC and the Degree of Crystallization: A Critical Review"
- "Preparation and Nonisothermal Crystallization Behavior of Polypropylene/Layered Double Hydroxide Nanocomposites," published in Polymer