Title: Effect of Ultrasonic Welding Process Parameters on the Crystallinity of GF/MWCNT/PP Composites

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ABSTRACT

Ultrasonic welding (USW) is a rapid and efficient joining method for thermoplastic polymer composites. This joining technique offers a high bonding strength between composite materials to form lightweight, durable structures in a cost-effective way, compared to other traditional joining methods. Crystallinity at the bond line can influence mechanical properties and chemical resistance. However, despite technological development of the USW process, the underlying crystallization mechanisms at the welded interface are still insufficiently understood. This paper explores the use of polypropylene (PP) and multifunctional multi-walled carbon nanotube (MWCNT/PP) films to perform USW between glass fiber (GF)/PP adherends and the resulting effect of welding parameters on the crystallinity degree at the bonded interface. After the USW process, PP and MWCNT films were isolated from glass fiber laminates using Kapton tapes. The effect of MWCNT content (0, 5, 10, 15, 20, 25 wt.%) and welding parameters on the crystallinity of the films and the welded interface was analyzed using differential scanning calorimetry (DSC) and Scanning electron microscopy (SEM), respectively. It was found that increasing MWCNT percentage resulted in a lower degree of crystallinity (44-34%) for the films and for the welded composite interface (23-9%). Four sets of different parameters (welding force and vibration amplitude) were used in this experiment, (500N, 38.1μm), (500N, 54.0μm), (1500N, 38.1μm), and (1500N, 54.0μm), representing different welding times. It was found that increasing force and amplitude resulted in a higher and lower degree of crystallinity, respectively, at the welded interface. Thus, it is expected that the change of crystallinity during USW may be partially attributed to strain-induced crystallization mechanisms.

INTRODUCTION

Several industries require lightweight structures, such as aerospace and automotive, leading to a demand for cost-effective manufacturing methods. Thermoplastic materials have been used in a variety of sectors over the last few decades due to their low weight, quick processing, recyclability, and corrosion resistance, when compared to traditional metals and alloys [1, 2]. One promising manufacturing technique for thermoplastics and their composites is ultrasonic welding. Ultrasonic welding is a process that uses high-frequency vibrations to join materials together. The materials to be joined are prepared and clamped securely. The vibrations are generated by an ultrasonic transducer, which creates friction and heat at the contact point between the materials. This heat melts the thermoplastic material, allowing it to flow and bond with the other material. In the case of metals, the vibrations help break down surface oxides and create a solid-state bond. Once the vibrations stop, the molten material cools and solidifies, forming a strong bond. Ultrasonic welding is a fast and efficient method that offers benefits like high bond strength, minimal heat-affected zone, and the ability to join different materials.

Polypropylene (PP), a polyolefin, is one of the most widely used semicrystalline thermoplastic polymers in applications ranging from automotive, aircraft, military, construction, and medical care to appliances, furniture, and clothing [2-4]. Its processability, good physical and chemical characteristics, and low cost make it attractive for these applications. However, PP possesses low electrical, mechanical, and thermal properties, when compared to other thermoplastics [2, 5]. To strengthen the different properties of the polymer, reinforcing fibers (short and continuous for thermoplastic composites (TPCs)) or fillers (carbon nanotubes (CNTs), carbon nanofibers (CNFs), etc.) are added to the polymer matrix [6-8]. For instance, multi-walled carbon nanotubes (MWCNTs) have demonstrated promise as excellent nanofillers for increasing polymer characteristics, such as mechanical, electrical, and thermal properties [9-11]. Moreover, in our previous work on ultrasonically welded thermoplastic composites (TPCs), it was demonstrated that MWCNT-based nanocomposite films offered dual functionality of heat generation during welding and ability to monitor structural damage through electrical resistance changes [12-14]. One important aspect of welded TPC joints is crystallinity at the interface, as it may influence chemical resistance and mechanical performance [15]. Since USW is a fast process where melting and solidification occur in a matter of seconds (< 4 s), it is expected to have a significant effect on the interface's crystallinity. The parameters of ultrasonic welding, such as welding force and vibration amplitude, have a significant impact on the crystallinity degree and crystal perfection of PPS (polyphenylene sulfide). High welding force and vibration amplitude result in predominantly amorphous PPS and imperfect crystals. Lower welding force and vibration amplitude increase crystallinity and crystal perfection to moderate levels. Despite high cooling rates during ultrasonic welding, PPS can still achieve a semi-crystalline structure due to the high strain rates and molecular chain orientation in the melt. By adjusting welding force and vibration amplitude, a semi-crystalline welding interface can be obtained.

Crystallinity is one of the key determinants of many polymer properties like thermal and electrical conductivity and mechanical performance [16-20]. Crystallinity refers to the degree to which a polymer material is composed of crystalline regions. The addition of nanoparticles can further affect the crystallinity of semicrystalline polymers by influencing the growth and arrangement of crystalline structures. The nanoparticles can serve as heterogeneous nucleating agents, causing the formation of smaller and more uniformly distributed crystallites. This results in an increase in the overall crystallinity of the material. Understanding the impact of nanoparticles on nucleation and crystallinity development in semicrystalline polymers provides crucial information for quantifying the structure-property relationships of these materials. Various researchers have extensively studied the impacts of carbon nanotubes (CNTs) on the crystallization behavior of composites [21-23]. The impact of CNT loading on the crystallinity (Xc) of polypropylene (PP) varies, with some studies reporting no change, slight decline, or a small increase in Xc. For instance, Qiu et al. did an experiment on the addition of MWCNT to the polymer and found that crystallinity increased by up to 6% with 1 wt.% MWCNT [24]. Mertens and Senthilvelan found that PP crystallinity increased by up to 5% with the addition of 1 wt.% CNT, but it decreased with the addition of 5 wt.% of CNT [25]. They assumed this result is the consequence of blocking the PP chains, which occurred from the high percentage of CNTs amount. Ersoy and Onder conducted DSC analysis on PP/CNT composites with varying CNT weight fractions (0, 1.8, 4.6, and 8 wt.%) [26]. They observed an increase in Xc from 39.7% to 44.7% at 4.6 wt.% CNT. However, a further increase in CNT content to 8 wt.% resulted in a decrease in crystallinity to 42%. In summary, weight fraction of CNT has a significant effect on the crystallinity of MWCNT/PP composites.

The purpose of this work is to characterize crystallinity of an ultrasonically welded glass fiber (GF)/PP interface with pure PP films and MWCNT/PP films. In this work, the effect of ultrasonic welding process parameters (force and amplitude) on the degree of crystallinity at the welded interface will be investigated, as well as the effect of MWCNT weight fraction (0, 5, 10, 15, 20, 25 wt.%) on the degree of crystallinity. Scanning electron microscopy (SEM) was first used to observe the microstructure at the welded interface. Then, differential scanning calorimetry (DSC) experiments were performed on the films before and after welding under different parameters to assess their degree of crystallinity.

MATERIALS AND METHODS

Materials

This section provides a summary of the materials utilized for manufacturing nanocomposite films and TPC adherends. In this study, MWCNTs, sourced from Cheap Tubes Inc. (Grafton, VT, USA), were employed as nanofillers for the preparation of nanocomposite films with varying weight percentages ranging from 5% to 25%. The MWCNTs possessed a high purity level of over 95% and exhibited an outer diameter within the range of 10 to 20 nm, with a length of 10 to 30 μm. Solvent dissolution was used for 5 and 10 wt.% MWCNT/PP, while masterbatches (provided by Cheap Tubes Inc.) were used for 15, 20, and 25 wt.% MWCNT/PP. For solvent dissolution, anhydrous p-Xylene (1,4-dimethylbenzene) with a minimum purity of 99% was acquired from Sigma-Aldrich, (St. Louis, MO, USA). Polypropylene (PP) pellets, provided by Goodfellow (Coraopolis, PA, USA), were used as the polymer matrix.

GF/PP laminates were produced using a heated press and a stacking sequence of eight UD GF/PP prepreg layers. The laminates had dimensions of 254mm × 254mm and were consolidated at 180°C and 1 MPa pressure for 15 minutes, followed by cooling to room temperature. The resulting thickness was approximately 1.8 mm, with a void content below 1.5%.

Nanocomposites and Film Preparation

The process of preparing MWCNT/ PP films involved several steps. A solvent dispersion method was employed to manufacture MWCNT/PP nanocomposites with 5 and 10 wt.% MWCNT, as described in detail in [13]. This dispersion process ensured uniform distribution of MWCNT within the PP matrix, enhancing the mechanical and electrical properties of the resulting composite material. Subsequently, the obtained MWCNT/PP nanocomposites were ground to obtain a fine powder. This powder was then subjected to compression molding in a laboratory heated press (Dake, Grand Haven, MI, USA), at 0.8 MPa and 180ºC for 15 minutes. The same compression molding process was followed to manufacture films with higher weight fractions, 15, 20 and 25 wt.% MWCNT.

The GF/PP laminates were fabricated with a heated press as well. The layup was 8 layers in the 0º direction, consolidated under 1 MPa at 180ºC for 15 minutes. The resulting laminates were then cut into rectangular samples (101.6 mm x 25.4 mm) with a water-cooled precision saw (Pace_Technologies,_Tucson,_AZ,_USA). Further details about the manufacturing process can be found in [13, 27].

Ultrasonic Welding Procedure

Specimens were joined in a single-lap configuration (figure 1) using a displacement-controlled Dynamic 3000 ultrasonic welder from Rinco Ultrasonics (Danbury, CT, USA). The welder operated at a frequency of 20 kHz and a maximum power of 3000 W, with a booster gain of 1:1.5. To create the GF/PP joints, nanocomposite films with varying MWCNT weight percentages (0, 5, 10, 15, 20, and 25 wt. %) were placed between the GF/PP samples to act as "energy directors" at the interface. During the welding process, a 1000 N force was applied, along with a 38.1 μm vibration amplitude, followed by a solidification phase of 4000 ms. The welding process was controlled by adjusting the vertical displacement of the sonotrode, set at 60% of the initial film thickness during the vibration phase for all experiments. Those welds were first used for general microstructural and crystallinity characterization at the welded interface.

Then, to test the effect of ultrasonic welding process parameters on the degree of crystallinity at the interface, four different sets of welding force and vibration amplitude were used, as summarized in Table I. In this experiment, 20 wt.% MWCNT/PP films and 0 wt.% MWCNT/PP (pure PP) films were used. To isolate and extract the interface, the MWCNT/PP films were placed between two polyimide (Kapton) films, each with a thickness of 25 μm (Figure 2) [28]. These Kapton films served as a barrier to prevent direct contact between the GF/PP samples and the MWCNT/PP films. After the USW process, the MWCNT/PP films were carefully separated from the Kapton films for further characterization using differential scanning calorimetry (DSC).

TABLE I. ULTRASONIC WELDING PROCESS PARAMETERS

Specimen Name	**Welding Force (N)**	**Vibration Amplitude (μm)**	**Travel (mm)**
Low Force and Low Amplitude (LF-LA)	500	38.1	0.3
Low Force and High Amplitude (LF-HA)	500	54	0.3
High Force and Low Amplitude (HF-LA)	1500	38.1	0.3
High Force and Low Amplitude (HF-LA)	1500	54	0.3

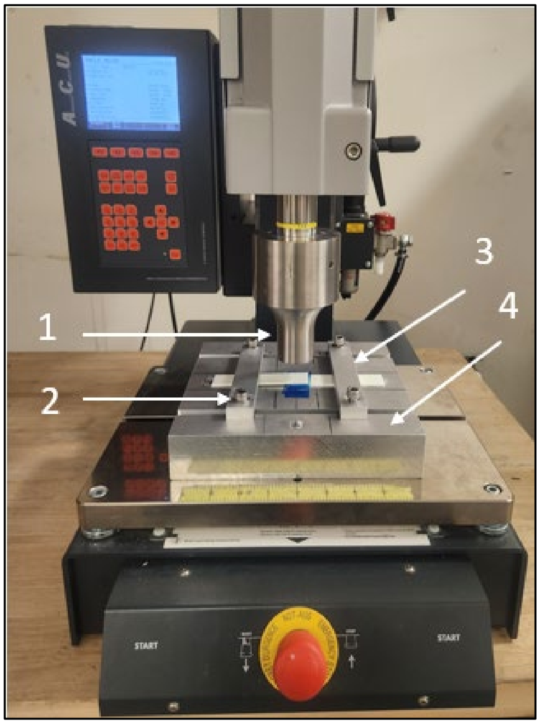

Figure 1. Welding setup utilized for conducting experiments with single lap shear samples. The components in the schematic are labeled as follows: 1. Sonotrode, 2. Lower sample clamp, 3. Upper sample clamp, and 4. Sliding platform.

Morphological and Crystallinity Characterization

Scanning electron imaging was conducted using an FEI Quanta 3D FEG field emission scanning electron microscope. Prior to observation, the specimens were coated with a thin layer of gold using an EMS550X sputter coater at 25 mA and a vacuum of 1×10^{-1} mbar for 2 minutes. The scanning electron microscope (SEM) utilized an accelerating voltage of 5 kV for imaging. Sample preparation include the following procedures: samples were initially embedded in epoxy resin molds. They were then subjected to a grinding process using SiC pressure-sensitive adhesive (PSA)-backed abrasive papers with grit sizes of 180, 360, 600, 800, and 1200. Following the grinding step, the samples underwent polishing using diamond solutions of 6 and 1 μm on polishing pads respectively. Representative SEM images for 0, 5, 15, and 25 wt. % MWCNT/PP films are shown in figure 3. A PerkinElmer Differential Scanning Calorimetry (DSC) 4000 was used to determine the crystallinity of the different samples. Crystallinity was measured for three types of samples: pure PP and MWCNT/PP films before welding, films extracted from the interface after welding, and welded interfaces with film. For the latter, the welded joint was cut, then the interface was collected with a sharp blade for DSC experiments. All samples weighed between 5 mg and 10 mg, sealed in a 5 mm diameter aluminum pan. The pan was placed in the DSC with a second empty aluminum pan for reference. The samples were heated up to 200 °C at 10 °C/min, then remained at 200 °C for 2 minutes. They were cooled down to room temperature at 10 °C/min. After 2 minutes at room temperature, the cycle was repeated a second time. The crystallinity was calculated using equation 1. Here, Xc is the percent crystallinity, ΔHm is the measured specific melting enthalpy in J/g and ΔH°m is the specific melting enthalpy of an ideal crystal (209 J/g) [17].

$$Xc = \frac{\Delta Hm}{\Delta H°m * (1\text{-}\alpha)} * 100$$

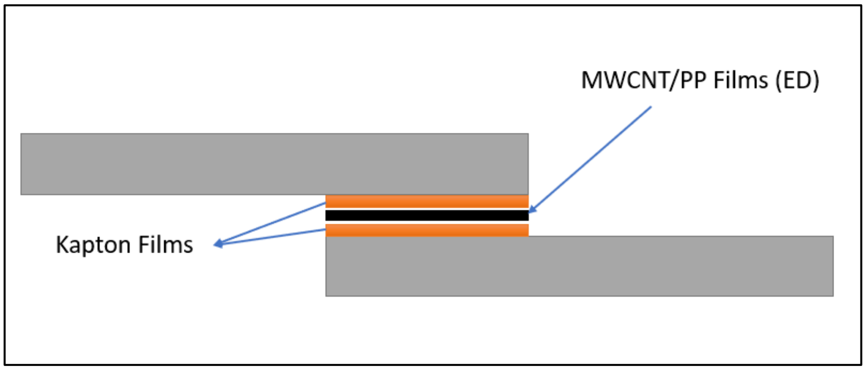

Figure 2. Schematic of the specimen setup designed for removing the energy directors from the welded interface, allowing direct crystallinity measurements. Note that the dimensions in the schematic are not depicted to scale.

RESULTS & DISCUSSION

Morphological Analysis by SEM

In this work, the purpose of SEM analysis on welded joints was to detect the interface area morphologically for different wt.% of MWCNT/PP samples. Figure 3 shows representative SEM images for four MWCNT weight fractions: 0, 5, 15, and 25 wt.%. The interface location is indicated by two arrows. The welded interface is not distinguishable for 0 wt.% MWCNT (Figure 3a), but as the percentage of MWCNT increased, the interface became more distinguishable at 15 and 25 wt.% (Figure 3c-d). This indicates a sharp transition between the MWCNT films and the GF/PP adherends, suggesting the nanotubes do not significantly diffuse into the adherends during the USW process.

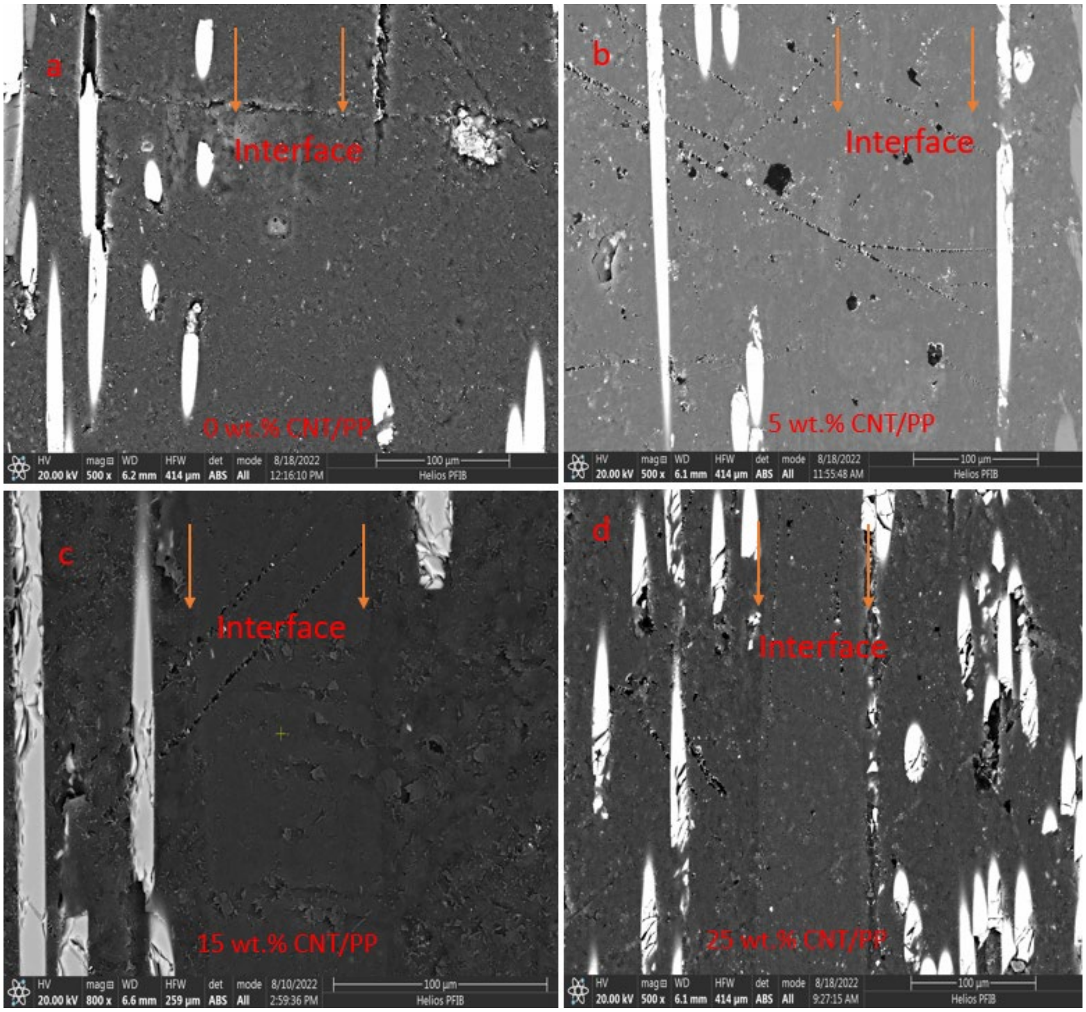

Figure 3. Representative SEM images for GF/PP joints welded with a) 0 wt.% MWCNT/PP films, b) 5 wt.% MWCNT/PP films, c) 15 wt.% MWCNT/PP films, and d) 25 wt.% MWCNT/PP films. All scale bars: 100 µm.

Crystallinity Analysis by Differential Scanning Calorimetry (DSC)

Table II shows the melting enthalpy of MWCNT/PP films (before welding) and welded GF/PP interfaces with all MWCNT/PP films. It is observed that melting enthalpy of the films and the welded interfaces decrease with the addition of MWCNT from (5-25) wt.%. The lowering of the melting enthalpy ultimately results in the lowering degree of crystallinity of PP composites. It was found that increasing MWCNT weight fraction resulted in a lower degree of crystallinity (from 43.5% to –34.5%) for the MWCNT/PP films and for the welded interfaces (from 22,7% to –8.6%) (Table III and Figure 5).

In the case of PP, the crystallization process is typically facilitated by the presence of nucleating agents such as fillers or additives. These agents provide nucleating sites for the PP chains, allowing them to align and form well-defined crystalline regions. When MWCNTs are added to the PP matrix, they introduce a high percentage of carbon

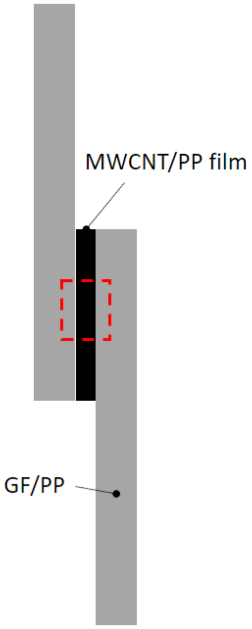

Figure 4. Schematic of the welded interface for the SEM imaging location

nanotubes into the system. These nanotubes have significantly different structure and properties compared to the PP chains. The presence of a high weight fraction of MWCNTs can physically hinder the access of PP chains to the nucleating sites. The rigid and entangled structure of the carbon nanotubes can obstruct the movement and alignment of the polymer chains required for crystallization. As a result of the blocking effect, the PP chains are unable to form well-defined crystalline regions. This leads to a decrease in the overall crystallinity of the composite material as the concentration of MWCNTs increases.

TABLE II. MELTING ENTHALPY OF DIFFERENT SPECIMEN SAMPLES

Specimen Name	Melting Enthalpy of Film (J/g) (Before welding)	Melting Enthalpy of Welded GF/PP Interfaces (J/g) (After welding)
0 wt.% CNT/PP (Pure Film)	70.8	40.5
5 wt.% CNT/PP	86.4	45.1
10 wt.% CNT/PP	74.3	36.2
15 wt.% CNT/PP	66.6	34.0
20 wt.% CNT/PP	60.6	23.7
25 wt.% CNT/PP	54.1	13.4

TABLE III. CRYSTALLINITY OF DIFFERENT SPECIMEN SAMPLES

Specimen Name	Crystallinity of Film (Xc %) (Before welding)	Crystallinity of Welded GF/PP Interface (Xc %) (After welding)
0 wt.% CNT/PP (Pure Film)	33.9	19.4
5 wt.% CNT/PP	43.5	22.7
10 wt.% CNT/PP	39.5	19.2
15 wt.% CNT/PP	37.5	19.1
20 wt.% CNT/PP	36.3	14.2
25 wt.% CNT/PP	34.5	8.6

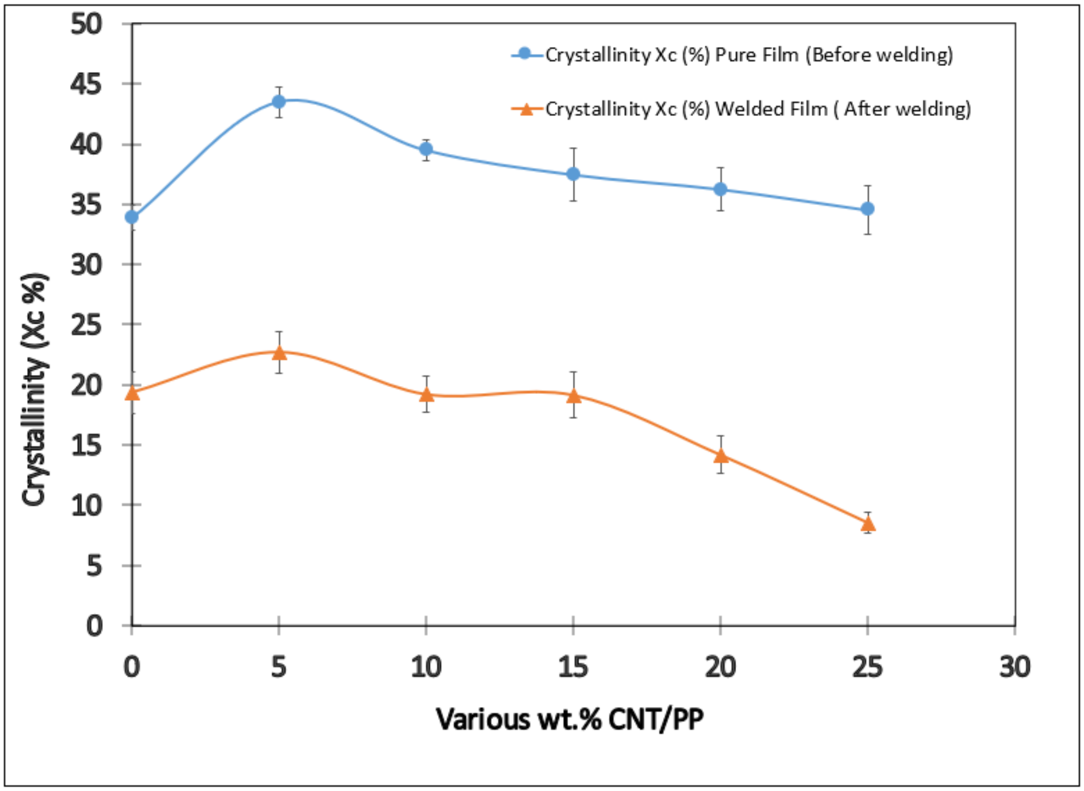

Figure 5. Crystallinity of MWCNT/PP polymer films containing 5 to 25 wt.% MWCNT (before welding) and welded GF/PP interfaces (after welding) by standard DSC measurements.

To test the ultrasonic welding process parameters on the degree of crystallinity at the welded interface for GF/PP joints, four sets of parameters (welding force and vibration amplitude) were used as shown in Table I. Table IV and Figure 6 summarize the crystallinity values for each set of parameters. Overall, it was found that an increase of force or amplitude led to an increase of crystallinity. HF-HA, representing the shortest welding time, generated the highest degree of crystallinity, while the LF-LA condition, representing a longer welding time, generated the lowest degree of crystallinity.

The analysis of the films using DSC measurements revealed that the process parameters had a significant influence on the degree of crystallinity. Specifically, when a welding force of 500 N and a vibration amplitude of 38.1 μm were utilized, the interface exhibited lower crystallinity (34.5% for pure PP films and 41.25% for MWCNT/PP films). On the other hand, employing a welding force of 1500 N and a vibration amplitude of 54 μm led to a welded interface with predominantly higher crystallinity (55.0% for pure PP films and 59.3% for MWCNT/PP films). In our previous work, Increasing the consolidation force in the ultrasonic welding process has been observed to increase the crystallinity of the polyphenylene sulfide (PPS) matrix. The higher consolidation force applied during welding promotes molecular alignment and packing, leading to a higher degree of crystallization within the PPS material. This increased crystallinity can enhance the material's mechanical properties, such as stiffness and strength [29].

Higher crystallinity typically implies a more ordered and regular arrangement of these structural units, although various factors can influence crystallinity, including temperature, pressure, and mechanical forces. When it comes to mechanical forces, such as high force and high amplitude, they can potentially affect crystallinity through different mechanisms, such as alignment of crystals and promoting nucleation. Application of mechanical force in the ultrasonic welding process can induce alignment orientation of existing crystals in a material. By subjecting a material to high force and amplitude, the crystals may align in a specific direction, leading to an increase in overall crystallinity. Moreover, mechanical forces can facilitate the nucleation and growth of crystals. The application of force can provide the energy required for the formation of new crystal nuclei, promoting crystallization. Higher force and amplitude can create favorable conditions for nucleation, leading to the growth of more crystalline regions within the material, even if cooling down occurs within seconds. This crystallization process can partially be attributed to the strain induced crystallization, but it deviates little from that kind of crystallization. Strain-induced crystallization typically involves the formation or rearrangement of crystals due to mechanical strain, while ultrasonic welding primarily relies on localized friction and heat to fuse materials together. Although crystal alignment may occur, it is primarily attributed to the heat and pressure generated during the welding process. Overall, the results presented in this study indicate that crystallinity at the ultrasonically welded interface may be controlled through applied force and amplitude.

TABLE IV. CRYSTALLINITY OF DIFFERENT SPECIMEN SAMPLES FOR VARIOUS PROCESS PARAMETERS

Specimen Name	Crystallinity of Extracted Film (Xc %) (Pure PP Films)	Crystallinity of Extracted Film (Xc %) (20 wt.% MWCNT/PP Films)
LF-LA (500 N, 38.1 µm)	34.5	41.3
LF-HA (500 N, 54 µm)	44.9	46.5
HF-LA (1500 N, 38.1 µm)	39.9	43.1
HF-HA (1500 N, 54 µm)	55.0	59.3

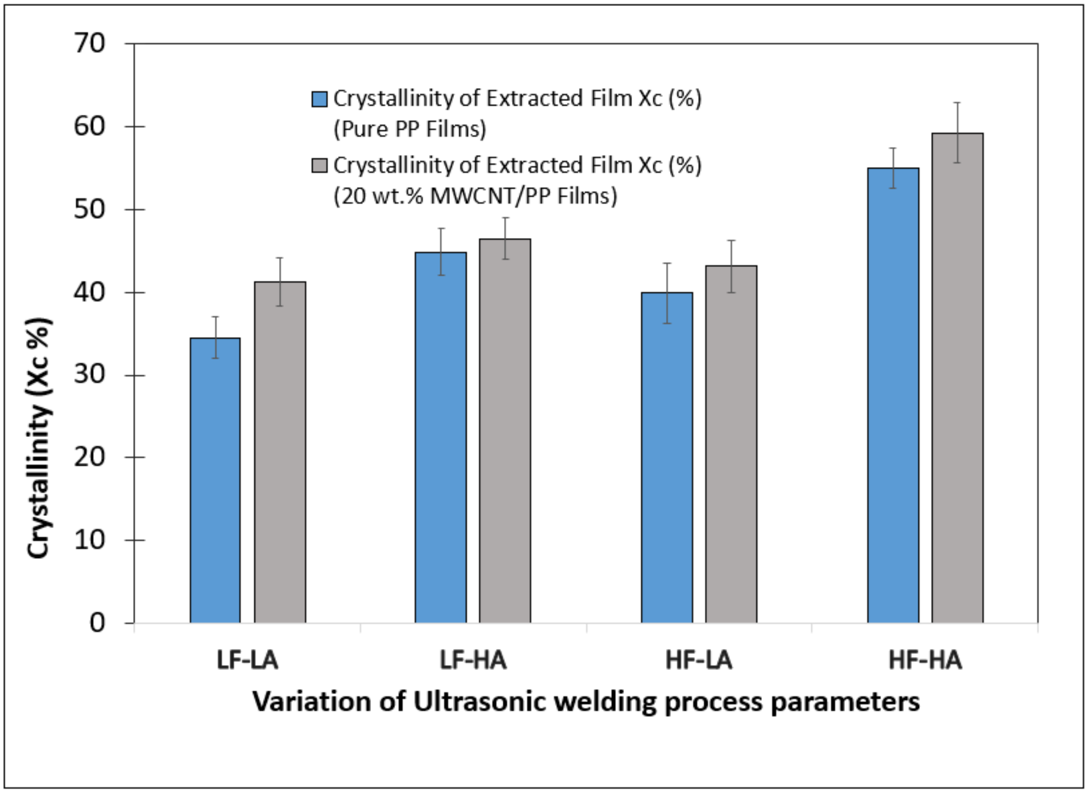

Figure 6. Crystallinity of MWCNT/PP films (before welding and extracted from the interface after welding) determined from standard DSC measurements.

CONCLUSION

In this study, GF/PP adherends were joined by ultrasonic welding using MWCNT/PP nanocomposite films. To analyze the effect of MWCNT content on the crystallinity of the films, different MWCNT weight fractions (0-25 wt.%) were analyzed by DSC and SEM morphological characterization. Moreover, the effect of USW process parameters on crystallinity of the welded interface was tested. It was found that the crystallinity decreased with MWCNT content from 5 wt.% to 25 wt.%. This is likely caused by the high concentration of MWCNTs blocking the PP chains from accessing nucleating sites, hindering their alignment and crystallization. Additionally, it was found that high force and high amplitude during ultrasonic welding results in higher crystallinity at the interface than low force and low amplitude.

Ongoing and future work include small angle X-ray scattering (SAXS) and Fourier transform infrared (FTIR) spectroscopy. SAXS will be used to examine the interface in greater detail by analyzing the scattering patterns. It will provide insights into crystallinity mapping at the interface, and the arrangement and size of nanoscale particles.

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