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FRICTION STIR WELDING OF POLYMERS

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1. Introduction

Engineering polymers enjoy a fast development even in industrial sectors where they used to be unimaginable so far. Simultaneously with this the need for more advanced forms of processing technologies also increases that render the application of new materials more reliable. These two processes complement each other: There is no new structural material if it cannot be processed by a proper technology and cannot be included into existing engineering structures. It is an important tendency nowadays that the borderlines of so far strictly separated metal, polymer and ceramic structures become somewhat blurred and by the combination of these new materials are being developed. The same occurs in the field of production technologies, where also more and more overlapping can be observed. A good example of this is the new joining technology presented in this dissertation, friction stir welding.

Welding technologies based on friction have been well known for both polymers and metals. Fairly well known are the various rotation and vibration welding methods. Friction stir welding appeared relatively recently in the literature which has been successfully applied to aluminum and its alloys and to other non-ferrous metals. This patented technology is related to the name of Thomas and his coworkers. The essence of the technology is that a rotating tool resembling the geometry of a milling tool is pushed in-between the plates to be welded, where the base material becomes molten under effect of the friction heat. If the flow of the melt is enhanced by using special grooves of the tool a truncated seam can be created along the borderline of the plates. Using this technology even thick welded seam can be created with high productivity, but the main advantage is the simplicity of this method. In addition to proper tools only a milling machine is needed and it is possible to create simple welded seams. With a multi-axial processing center or by using welding robots it is of course possible to produce complicated 3D seams as well.

The main goal of the dissertation is to analyze the applicability to polymeric materials and to explore the factors influencing the strength of the welded seam. An addition goal is to study the applicability of the method to polymer composites with thermoplastic matrices, as for this dynamically evolving class of structural materials there is no reliable welding method.

2. Critical evaluation of the literature, goal of the thesis

Welding of polymers is performed in the industry by well-proven technologies, based on irradiation (e.g. laser), on heat conduction (e.g. heat-mirror, hot gas), or on friction (e.g. ultrasonic) principles. Each technology has of course its advantages that make them suitable to solve certain engineering problems. In this sense it is hard e.g. to compare the heat-mirror welding technology of pipes with ultrasonic welding, which is generally used to weld smaller, injection molded parts. The basis of the comparison is mostly the energy used to achieve the welded joint and the quality factor of the welded joint. The quality factor is the ratio of a certain physical property of the welded seam and the same property observed in the base material.

When considering the applicability of the various methods the disadvantages attached to these technologies should also be considered. Hot gas welding widely used by the industry requires complicated surface preparation and the welding process itself requires the use of a filler material. Heated element welding mostly used in the production of pipelines also requires surface preparation (planing), but its largest disadvantage is the fact that the geometrical form of the heated element can be used only for the given welding process. Those welding methods which directly utilize the heat creating ability of electrical energy (e.g. high frequency, induction, resistance heating) usually need metal additives or wires and by themselves can be applied to a few polymers. Laser welding can be used for overlapping joints only if the plate to be welded is transparent to the laser beam from the beam direction. It is not negligible that the methods mentioned later are relatively expensive ones.

As a new welding method FSW belongs to friction welding, within that to methods based on rotation. The materials to be welded do not need special preparation, even thick (>10 mm) plates can be welded in a single step without any filler material. The tool geometry and its effects on the seam structure and on the joint strength have been extensively studied for the welding of metals. Several simulation and modeling techniques have been developed for metals that allow accurate prediction of temperature and flow conditions during welding.

Rheological properties of polymers (visco-elasticity) are different from those of metals, so especially in friction stir welding, where the resulting flow fields play an especially important role, the joint strength strongly depends on the tool used. Because of these factors the FSW method to be used in polymers requires special tools and the effect of welding parameters depends on the geometry of these tools.

In engineering constructions various continuous fiber reinforced composite structures with thermoset matrices are fairly widespread but composites with thermoplastic matrices are also being developed dynamically starting from short fiber reinforced, injectable materials to systems containing glass or carbon fiber textiles. Widespread use of composite structure raises the necessity of welding these materials. As for the welding of composites reinforced with continuous fibers it can be said that the reinforcing structures do not intermesh (there is no overlapping), so in any welded structure only the matrix material bears the loads. If welding short fiber reinforced composites (mostly by ultrasonic and vibration technologies) the main problem is the unfavorable distribution and orientation of the fibers within the seam.

Based on all these the main goals of my dissertation are as follows:

1. Analysis of the application of friction stir welding (FSW) to thermoplastic polymers and to thermoplastic matrix composites, more specifically the creation of welded joints with maximum tensile and flexural strength values.
2. Exploring the inter-relations between various welding parameters of FSW (feed, rotation speed etc.) on the one hand and the analysis these parameters on the strength of the welding seam.
3. Investigation of the morphological structure of the welded joint produced by FSW and the analysis of the effect of welding parameters on the material structure within the heat affected zone.
4. Investigation of the stress state of the seam and its environment, their relation to the set welding parameters.

3. Materials and test methods used

The plate used for welding is PP-DWST polypropylene produced by SIMONA marketed in 10 mm thick extruded form. Data given by the manufacturer are as follows: Crystalline melting point: 160-165°C, density: 0,905 g/cm^3, tensile modulus: 1400 MPa, $MFI_{(230;\ 2,16)}$=0,4 g/10 min. Calculating the tensile strength from 5 parallels on test specimens of 20 mm width (similarly to the welded specimens) at 10 mm/min deformation rate the result was 25.6±2.1 MPa. The flexural strength of the base material (studied by the DVS 2203-5 standard used for the welded joints) was 56.9±3.3 MPa.

Fort he stress optical studies I needed a transparent polymer which could be purchased in 10 mm thickness and could be easily welded. The material was used as a model material in my work in order to study the stress states evolving in the welded seam. SIMONA PETG (poly(ethylene terphthalate glycol)) used by me is a material that can be easily thermoformed at relatively low temperatures without drying. It is transparent, with glossy surface and good optical properties. According to the data provided by the manufacturer its modulus of elasticity is 2200 MPa, its tensile strength is 50 MPa, the glass transition temperature is 72°C. According to my measurements its melt flow index is: MFI $_{(230;\ 2,16)}$=10,9 g/10 min.

For the production of composite plates compounds were prepared by a Labtech Scientific twin screw extruder using the following materials:

- 65 wt% Tipplen H483F polypropylene homopolymer (MFI $_{(230;\ 2,16)}$=6,5 g/10 min, elasticity modulus is 1700 MPa, tensile stenght is 34.5 MPa),
- 30 wt% glass fiber (OCV Italia, grade 995-13C) of 4.5 mm initial length
- 5 wt% Scona 8012FA coupling agent (PP grafted with 1 wt% maleic anhydride)

The material flowing from the extruder die was directly (in hot state) transferred into a molding frame and 10 mm thick plates were compression molded with a Collin P200E laboratory press. The size of these plates was 160 mm x 160 mm, which were first cut into two halves then the welded seam was produced along the cut. According to my test results the melt flow index of the composites sheet was MFI $_{(230;\ 2,16)}$=9.1 g/10 min, the flexural strength was 76.7 MPa.

The applicability of the FSW technology and the tensile strength of the seams were analyzed by tensile tests. In my experiments PP plates of 10x100x200 mm size were welded together, then from the welded joints 6 pieces of test specimens with B=20 mm width were machined so that the welded seam is situated perpendicularly to the tensile direction, at the center of the specimen. The test speed in the tensile test was 10 mm/min, the test machine used was Zwick Z020.

The flexural tests were performed on a Zwick Z2020 tensile tester, where the span length was l=90 mm, deformation rate was v=20 mm/min, using the DVS 2203-5 standard widely used by experts of plastic welding. 6 specimens were cut from the welded plates, the width of the test specimens was b=20 mm. The first test specimen was cut in 30 mm distance form the starting point of the welding to avoid the uncertainties associated with the transitory nature of the initial welding process. When bending the test specimens, bending directions from the upper (so-called crown side) and from the lower (so-called root side) side of the seam were distinguished.

In my stress optical studies the stress states of the seam and the heat affected zone were investigated. In these studies flexural tests were made according to the DVS-2203-5 standard, using a computer controlled Zwick Z005 universal tester. In the stress optical studies the width of the specimens was b=2mm. When studying the three point bending of welded seams the tests were complemented by two circular polarizer filters (in order to filter out the orientation fringes) and FUJI Finepix S7000 digital camera. The load was increased stepwise the stress state was photographed in each step.

The crystallinity of the welded specimens was determined by differential scanning calorimetry using a Perkin Elmer DSC2 equipment (temperature range was 23–725ºC sensitivity was 0,42 mW). Test specimens were cut from four characteristic sites of the welded plates, then they were placed into the DSC equipment and studied at a heating rate of 10°C/min in the 80-230 °C temperature range.

Scanning electron microscopy (SEM) was used for the study of fracture surfaces of samples made of different base materials (PP, PETG, glass fiber reinforced PP) and for studying the crystalline structure of the welded seam. SEM studies were performed on a Jeol JSM-6380-LA equipment.

The crystalline structure was investigated using crossed polarizer (linear polarizer and analyzer) in transmission mode, using an Olympus BX 51 optical microscope. For this about 10 µm thick slices were cut from the seam using a Bright 5040 microtome and 3-5 µm slices using a Leica EM UC6 ultramicrotome with an FC7 cryochamber. Several smaller slices were prepared to study the welded seam: some cut from the center of the seam, others from the base material and from the borderline between the two zones.

In order to determine the residual glass fiber length samples cut form the specimen were heated in a ceramic crucible until the polymer parts of the sample were burnt away. The residual organic material was removed by 500 °C incineration in an oven for 30 minutes. When measuring the length distribution the fibers were spread onto a microscopic slide and dispersed by acetone. Measurements were performed by an Olympus BX 51 optical microscope in transmission mode using the AnalySIS Steel Factory image analysis software. 1000 length tests were made for constructing the relative frequency diagrams and for determining the average fiber length.

Melt flow indices (MFI) of the polymeric materials used were determined under conditions prescribed by the MSZ EN ISO 1133:2005 standard using a CEAST 7027.000 computer controlled capillary plastometer at 230 °C using 2.16 kg load. When determining the MFI values smaller test specimens were cut form the plates which were later milled by a Fritsch Pulverisette 15 cutting mill so that it could be fed into the MFI testing machine. 5 parallels were measured at each composition in order to determine the averages.

Internal stress states can be developed by the so-called thermo-retardation analysis (TRA) method. TRA is base on the fact that in partially crystalline polymers, if heated above the T_m temperature, polymer chains can freely reorient and can assume the thermodynamically most favorable configuration. For this a silicone oil bath allowing free shape change was necessary, wherein PP can float. Stresses released during retardation can be determined both qualitatively and quantitatively from the geometrical differences before and after the test. In our studies where the material extends, we can infer residual compressive stress and, vice versa, in the case of residual tensile stress shrinking of the material is expected. PP was studied at 190 °C in silicone oil for 20 minutes.

4. Summary of the results

In my dissertation I proved the feasibility of the FSW method to polymers by welding PP, PETG and glass fiber reinforced PP plates. In preliminary experiments I have established that the tool and the shoulder rotating together with it (which proved to be good for metal welding) resulted in bad surface quality for polymers, therefore the FSW technology was modified so that a non-heated PTFE shoulder was used as smoothing shoe. I proved that a properly selected endmill is needed for welding that should be rotated in a counter-rotating direction with respect to the milling direction. In order to weld the polymer plates by automated FSW technology a welding bench was constructed and the necessary prerequisites of the stable technology (root-support and cutting depth) were determined.

In my work done on polypropylene sheets joints with a quality factor of 80-90% could be prepared using a rotation speed of 2500-4000 rpm. Using a factorial experimental design the effects of welding rotation speed, feeding rate, tool diameter and cutting depth as parameters were investigated and it has been established that the most important effect was caused by the rotation speed. In three point bending tests two different loading conditions were distinguished: in one case the loading head contacted from the crown side of the tool, in the other from the root side of the seam. Bending strength can be determined from both directions, and to achieve a nearly similar value a proper setting of cutting depth proved to be essential.

Changing from PP to PETG as a model substance the properties of the heat affected zone (HAZ) were investigated as a function of the setting of the welding parameters and the tools used. It has been established that the root and HAZ is exposed to residual internal bending stress, which was compressive on the crown side and extensional on the root side of the welded seam. The presence of residual internal bending stresses has been proved by thermo-retardation analysis for PP.

In order to characterize the set welding parameters and the tools a K factor characterizing the heat transfer during welding was created. It was shown that this K factor is closely related to the extension of the heat affected zone of the seam and to the mechanical properties of the seam.

In the case of PP plates reinforced with 30 wt% glass fiber the applicability of FSW to polymer composites with thermoplastic matrix was proved. The highest flexural strength for glass fiber reinforced composite sheets with H483F PP matrix was achieved with a four-edge tool of 8 mm diameter at a rotation speed of 2100 rpm. This maximum value was 48.9±4.1 MPa, which exhibits a quality factor of 65% with respect to the composite base material. In the case of non-reinforced plates made of H483F matrix, using the same tool the optimum bending strength could be achieved at only 1500 rpm rotation speed. Its value was 38.3±3.8 MPa, and the higher value observed for the composite plate could be explained by the reinforcing activity of the fibers in the welded seam.

In my thesis the supermolecular structure of polypropylene seams prepared by friction stir welding was studied by optical and electron microscopy. A welded seam of lower strength (welding at a rotation speed of 2000 rpm) and the maximum tensile strength welded joints (welded at 3000 rpm) were compared. At the central part of the seam a spherulitic structure similar to the base material evolved, the average diameter of the spherulites observed here (10-20 μm) is about half that of the base material (25-30 μm). The spherulitic structure observed in the central part of the seam developed due to the relatively slow cooling rate in the central part of the seam. In the samples welded with a rotation speed of 2000 rpm a transition zone of 400-500 μm width was observed at the border of the base material and the seam. The width of this transition zone reduced to about half (200-300μm) a rotation speed of 3000 rpm. More supermolecular structures were identified in the transition zone, and at the outer perimeter of the tool (in the side line of the mantle) a straight borderline was observed with cylindritic structure.

5. Theses

Based on the results presented in my dissertation the following thesis points are stated:

1. I proved that friction stir welding is applicable to thermoplastic polymers (polypropylene, poly(ethylene terephthalate glycol) and polypropylene reinforced with

30 wt% glass fiber) but, instead of the shoulder moving together with the tool, used with metals, a polytetrafluoroethylene based smoothing shoe containing no additional heating, non-rotating, but moving together with the tool should be used. I proved that for friction stir welding of 10 mm polypropylene plates (most widely used in practice) an 8 edge endmill of 8 mm diameter can be used (counter-rotating with respect to the milling direction), resulting in 80-90% quality factor in the 2700-4000 rpm rotating speed range at a feed rate of 30 mm/min [1, 2].

2. Using a full factorial experimental design, covering the most important technical parameters of friction stir welding, I have demonstrated that from feeding rate, rotation speed, tool diameter and cutting depth rotation speed and cutting depth have the largest effect on the flexural strength of the welded seam in polypropylene. I have proved by measurements that, if welding 10 mm thick polypropylene plates at a cutting depth of 9.6 mm and using 1 mm grooved root support the difference between the flexural strength values measured from the crown side and from the root side disappears, and a quality factor of 90% can be achieved at 3000 rpm rotation speed and 50 mm/min feed rate [6, 7].
3. Using stress optical measurements on seams produced from 10 mm thick poly(ethylene terephthalate glycol) model substances I have introduced a dimensionless K parameter characterizing the heat energy transferred into the friction stir welded seam, which incorporates feed rate, rotation speed and tool diameter:

$$K = \frac{rotationspeed}{feed} \cdot tooldiameter$$

I have shown that the K factor is a value that can be determined from the set welding parameters and that can be used to assess the strength of the welded seam. Based on flexural strength data of welded seams of poly(ethylene terephthalate glycol) plates I have shown that a value of 150-400 of the K factor produces on the average a quality factor of 70-80% [9].
4. Based on stress optical tests performed on poly(ethylene terephthalate glycol) plates I have demonstrated that there is a residual bending stress in the seam: a compressive stress on the crown side and a tensile stress on the root side. When studying the width of the heat affected zone I have demonstrated that the average width depends linearly on the K energy factor incorporating the welding parameters (rotation speed, feed rate an tool diameter) [9].

5. I have established that in the case of polypropylene seams prepared at 2000 rpm, exhibiting a quality factor of 50-55%, because of incomplete melting a strongly sheared transition zone of 400-500 μm width appears at the borderline between the seam and the base material. The width of the transition zone decreases to 200-300 μm in the case of polypropylene seams produced at 3000 rpm, having a quality factor of 85%. I have shown that the average diameter of the spherulites in the seam is about half of those situated in the base material [10].
6. I have proved that friction stir welding is an effective welding method even in the case of 30 wt% glass fiber reinforced polypropylene matrix composite plates. The flexural strength of seams produced on 10 mm thick composite plates with a four edge endmill of 8 mm diameter, at 2100 rpm rotation speed is by 27% higher than those of seams produced on non-reinforced plates with parameters providing optimum flexural strength. This improvement of the flexural strength in composite plates proves the presence of overlapping reinforcing fibers at the borderline between the seam and the base material. The quality factor of the seam is 64% if compared to the flexural strength of the basic material of the polypropylene composite sheet. Below 2100 rpm the flexural strength of the seam decreases because of the incomplete melting of the polymer base material, while above this rotation speed the fragmentation of the reinforcing glass fibers results in a decrease of the flexural strength [11].

6. List of publications related to the topic of the dissertation

Conference and journal articles

[1] **Kiss Z**., Czigány T.: Automatizált hegesztőpad építése polimerek kavaró dörzshegesztéséhez. Gép, **58**, 59-62 (2007).

[2] **Kiss Z.**, Czigány T.: Applicability of friction stir welding in polymeric materials. Periodica Polytechnica, **51**, 15-18 (2007).

[3] **Kiss Z**., Czigány T.: PA6 anyagú elektromos csatlakozó ultrahangos hegesztésének optimalizálása. Műanyagipari Szemle, **4**, 71-79 (2007).

[4] **Kiss Z**., Czigány T.: Polimerek hegesztése és hegeszthetősége. Anyagvizsgálók Lapja, **17**, 98-102 (2007).

[5] **Kiss Z.**, Czigány T.: Invested energy needed friction stir welding. 6. Országos Anyag-tudományi Konferencia – Gépészet 2008. Május 29-30, 2008. Proceedings of Sixth Conference on Mechanical Engineering. Budapest, Paper ID: G-2008-H-15, p. 5

(2008).

[6] **Kiss Z**., Czigány T.: A kavaró dörzshegesztés átalakítása polimer anyagokhoz. Műanyag és Gumi, **47**, 129-133 (2010).

[7] **Kiss Z**.: Kavaró dörzshegesztésssel készült polimer varratok szilárdsági elemzése. 25. Jubileumi Hegesztési Konferencia. Budapest, Május 19-21, 2010. Cikk: MTESZ Gépipari Tudományos Egyesület, Budapest, 431-437 (2010).

[8] **Kiss Z.,** Kmetty Á., Bárány T.: Investigation of the weldability of the self-reinforced polypropylene composites**.** Materials Science Forum, **659**, 25-30 (2010).

[9] **Kiss Z**., Czigány T.: Effect of welding parameters on the heat affected zone and the mechanical properties of friction stir welded poly(ethylene-terephthalate-glycol). Journal of Applied Polymer Science, IF: 1.240 *javítással elfogadva (2011).*

[10] **Kiss Z**., Czigány T.: Microscopic analysys of the morphology of seams in friction stir welded polypropylene. Express Polymer Letters, IF: 1.575 *elfogadva, megjelenés alatt (2011).*

[11] Czigány T., **Kiss Z**.: Friction stir welding of fiber reinforced polymer composites. 18th International Conference on Composite Materials (ICCM18), South-Korea, Jeju, Augusztus 22-26, 2011. Paper ID: Th11-4-AF0685, p. 6 (2011).

Conference lectures and poster presentations

[12] **Kiss Z**., Czigány T.: Kavaró dörzshegesztés alkalmazása polimer szerkezeti anyagokra. **Mechanoplast XV**. Műanyagok műszaki alkalmazása és feldolgozás-technológiája konferencia, Gyula (2007).

[13] **Kiss Z**., Czigány T.: Production and examination of butt seams made by friction stir welding on polypropylene sheets. **VI. Országos Anyagtudományi Konferencia**, Siófok (2007).

[14] **Kiss Z**.: Weldability of fiber reinforced thermoplastic polymers. **International Conference on Technical Textiles and Nonwovens.** Delhi, India, 2008.11.11-13. (2008).

[15] **Kiss Z**., Czigány T.: Friction stir welding of fiber reinforced polymer sheets. **The 4th China-Europe Symposium** – Processing and Properties of Reinforced Polymers. 2009.06.8-12 Guilin, Kína (2009).