Effect of Welding Parameters on the Heat Affected Zone and the Mechanical Properties of Friction Stir Welded Poly(ethylene-terephthalate-glycol)

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ABSTRACT: The bond strength and the heat affected zone (HAZ) of friction stir welded seams made of poly (ethylene-terephthalate-glycol) are analyzed in the article. The seams were prepared with various settings (rotation speed and feeding rate) and with various welding tools. Using the welding parameters (rotation speed, the feed rate, and the tool diameter) a *K*-factor characterizing the welding process was defined. The novel *K* factor is related to the welding heat input, which is in direct correlation with both the range of the HAZ and the mechanical properties of the seam. The HAZ formed was analyzed by stress optical method, the mechanical properties by flexural test. It has been demonstrated that the efficiency factor of the welded joint is closely related to the width of the HAZ and it depends on the welding parameters and the tools. © 2012 Wiley Periodicals, Inc. J Appl Polym Sci 125: 2231–2238, 2012

Key words: thermoplastics; stress; imaging; mechanical properties

INTRODUCTION

Friction stir welding (FSW) started at the beginning of the 1990's from a patent[1] assigned to Thomas, which soon proved to be a well applicable mechanical welding technology, mostly for aluminum, light metals, and their alloys. In the time-period since then several publications appeared related to the understanding of phenomena of FSW of metals. Most researched area includes the mechanical properties, the investigation of morphological structure in the seam and in its immediate neighborhood, in the so-called heat affected zone (HAZ).[2] A significant amount of literature has been generated in relation to the geometry of the welding tool, the energy consumption during welding and to the flow phenomena during welding for metals.[3] In the FSW of polymers, the aspects observed with metals are applicable but due to the structural and morphological differences between polymers and metals, the HAZ and the residual internal stresses after welding should be studied carefully.[4]

FSW of metals is performed according to the principle shown in Figure 1. First a rotating tool is pushed in-between the sheets to be welded during welding, the friction heat melts the plates, then the tool is moved along a predetermined direction, the seam is formed behind the moving tool. The technology has been mainly developed for butt-welded seams.[2] The welding tool is usually equipped with a pin of special geometry and with a shoulder. The pin resembles mostly a milling tool; its role is—in addition to ensure the proper friction heat—to mix the material of the two sheets, thus forming the welded seam. The tool also contains a shoulder part, which is supposed to contain the material being close to its melting point and to ensure a smooth seam surface. Therefore, the seam needs no post-treatment.

Relatively few articles have been so far published on the application of FSW to polymeric materials and on the feasibility of the method itself under such conditions. This is so in spite of the fact that welding of polymers is a very important industrial task, beginning from the ultrasonic welding of mostly smaller, injection molded products up to huge, mirror-welded containers of several 10,000 L size with large wall thickness.[5,6]

The Welding Institute patenting FSW modified the original process for welding polymers, using a vertically vibrating sheet instead of rotating tool for producing polymeric seams.[7] Nelson, however, in his patent suggests geometrical features for the surfaces

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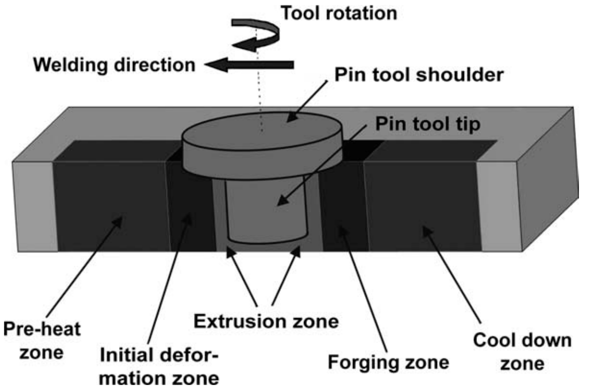

Figure 1 Scheme of the principle of FSW in metals.

of rotating tools, to be used with polymers according to the original FSW method.[8]

Similarly, to metal welding classification, plastic welding technologies can also be classified according to the heat generation. Such are irradiation (e.g., laser), heat conduction (e.g., hot gas welding), and mechanical-friction (e.g., ultrasonic and vibration welding). Hot gas welding is still very important, where we have studied in our earlier work[9] the microstructure formed in the HAZ of welded PP seams. The structure of the seam was divided into three main parts, starting from the raw material to the seam. We have shown by measurements that the width of the HAZ exhibits strong correlation with the strength properties of the seam, so by measuring the width of the HAZ the welding itself can be well qualified.

Varga et al.[10] welded together α and β-crystallized PP sheets made of various types of PP (homo, random, and block copolymers, different MFI values) by vibration method. The HAZ of the welded seams was studied by polarization microscopy and the effect of morphological structure developed during welding was investigated in terms of the mechanical properties of the seams. In the case of polymer joints, the appearance of new structural materials[11–13] makes necessary the development of new welding technologies in addition to the traditional ones.

Strand used a stationary shoe equipped with an ancillary heating[14] instead of the shoulder rotating together with the tool for FSW of polymers. They welded PP sheets in their work with various tool diameters and welding parameters and studied the effect of feed rate of the welding tool as well as the effect of the temperature of the smoothing shoe. It has been established that with increasing welding feed rate the maximum stress measured at the three point bending and the maximum deflection tolerated by the welded seam decreased. In the case of three point bending distinction was made whether the root of the seam was on the same side or on the opposite side with respect to the loading head. Based on the experiments, it was concluded that in the case when the root of the seam was exposed to tension during bending the flexural strength decreased.

Arici and Sinmaz[15] welded PE sheets of 5 mm thickness by FSW. As they realized the inferior mechanical properties described by Strand caused by weak welding roots, they prepared two welding seams on both sides of the PE sheets. With this solution, they could obtain very good seam strength identical to the yield strength of the raw material. Two-sided welding, however, takes twice as much time and more energy than single-sided welding, therefore the main advantage of the process, namely its simplicity is significantly reduced.

Rezgui et al.[16] also dealt with the FSW of PE, they studied the effect of welding parameters using the Taguchi method. They have shown that the area of the welding tool and its rotation speed influences strongly the temperature and the seam strength developing during welding. These two parameters affect each other mutually, as the peripheral speed changes with both the tool diameter and with the rotation rate, but they considered the effect of rotation speed most important. In our earlier work,[17] we studied the effect of welding parameters on friction stir welded polypropylene welded seams.

Having reviewed the literature it can be seen that the HAZ and the morphological structure developing in the polymer-welded seam have a profound influence on the strength of the welded seam.

The investigation of the HAZ developing during welding and the direct study of the residual stresses in the joint are not easy with semicrystalline materials. Therefore, to study internal stresses we changed PP to poly(ethylene-terephthalate-glycol) (PETG) as a model material, and investigated the seams made of this material by stress optics. The use of PETG can be rationalized by its proper level of transparency and low crystallization tendency.

The aim of this article is to demonstrate the relation between the internal stresses arising in the seam and in the HAZ and the welding technology, and to demonstrate the effects of all this on the mechanical properties.

MATERIALS AND METHODS

The study of FSW was performed on automatic welding equipment, which was developed directly for this method. The device made the examination of all important parameters effecting strength of the welded seam possible. Using this equipment it is possible to perform welding in a broad range of rotation speed and feeding rate. The equipment allowed constructing and studying various tool geometries and smoothing shoes. In preliminary experiments, it has been demonstrated that it is not necessary to use ancillary heating with the smoothing

TABLE I
Welding Parameters and Tools

Investigated parameters	Values			
Rotation speed (1/min)	750	900	1200	2550
Feed (mm/min)	50	75	90	100
Tool diameter (mm)	8		12	
Number of tool edges (–)	4		8	

shoe, so it was prepared from PTFE instead of metal. Welding was made with a tool of 8 and 12 mm diameter, with 4 and 8 edges. Welding experiments were made with the parameters listed in Table I.

To characterize the mechanical properties of the seam three point bending was used. Flexural tests were made according to the DVS-2203-5 standard, using computer controlled Zwick Z005 universal tester. The side of the seam pointing toward the tool is called crown side, while the side at the support is called root side. When performing the flexural tests distinction was made between the cases when the specimens were loaded from the crown or from the root side (in the case of crown side loading the upper side of the seam contacted the loading head, while in the case of the root side testing the lower side).

To study the fracture surface of the seams a JEOL JSM-6380 electron microscope was used, the surface of the specimens was rendered conductive by evaporating a thin gold layer.

To study the behavior of the HAZ seam during loading photoelasticity method was used. The principle of the photoelasticity is because most amorphous (homogeneous, isotropic, and transparent) material becomes birefringent is exposed to external forces, due to the stressed state. If putting the investigated specimen between two polarization filters and illuminating it by white light colored and dark fringes appear. From these fringes one can determine the stress state. For this study, we needed a material that is transparent, exhibits proper stress optical activity, not prone to crystallization, and processed by a technology resulting in quasi-homogeneous structure. Based on these criteria, we have chosen a 10 mm thick PETG sheet of SIMONA (flexural strength: 38 MPa, $E = 2200$ MPa). When studying the three-point bending of welded seams, the tests were complemented by two circular polarizer filter (to filter out the orientation fringes) and a FUJI Finepix S7000 digital camera. The load was increased in 10 steps by 5*N*, the stress state was photographed in each step.

Although the stress levels in the material can be determined by stress optical method, due to the near melting point temperature in the seam and in the HAZ and due to the strong shearing effect caused by the tool the orientation of polymer molecules has also to be taken into account. As the separation of orientational birefringence from the internal stress state is not easy, with did not want to express the stress levels numerically.

RESULTS AND DISCUSSION

In our earlier work, we have already investigated the tensile strength of seams for polypropylene sheets,[17] then, however, we used the shoulder rotating together with the tool (which proved to be a good solution for metals), and the smoothing shoe was used only in our later experiments. In our tests, we have shown that from the parameters studied rotation speed exerted the greatest effect on the HAZ of the seam (Fig. 2). The heat amount necessary for the development of a proper seam can be mostly influenced by this parameter. If the rotation speed is too low, the material does not melt to the required degree, which can even lead to the damage of the welding machine itself. If, however, too much heat is evolving, the material may degrade, which may lead to a lower quality seam. If using smoothing shoe for PP, in a rotation speed range of 2500–4000 rpm joints with an efficiency factor of 90% were created. In case of welding of PETG, the achieved maximum efficiency factor was near 90%, however, the rotation speed was much lower.

In the case of the amorphous PETG sheets used for photoelasticity the rotation speed values observed with PP proved to be too high. This is a consequence of the differences in material structure, in softening points and flow properties. It is obvious that with decreasing rotation speed the mechanical energy transferred to the seam also decreases, that is, heat evolution also decreases. In the case of PETG seams of acceptable quality were made with lower rotation speeds of about 1200–1800 rpm. The experiment with 2500 rpm was retained to demonstrate the picture also of the over-melted seam, and because analogies can be observed between the high rotation speed and the high tool diameter.

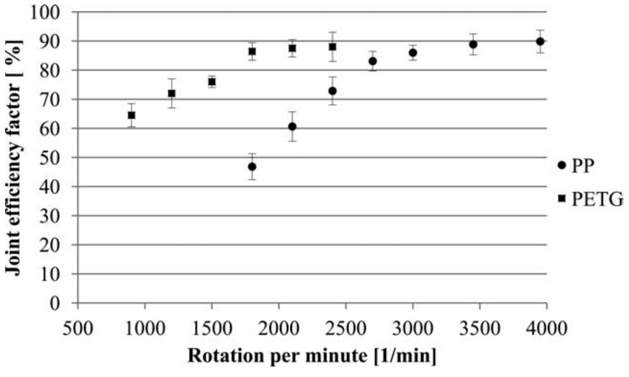

Figure 2 Rotation speed and the achieved efficiency factor (based on the tensile strength of the raw material) for friction stir welded polypropylene and poly(ethylene-terephthalate-glycol).

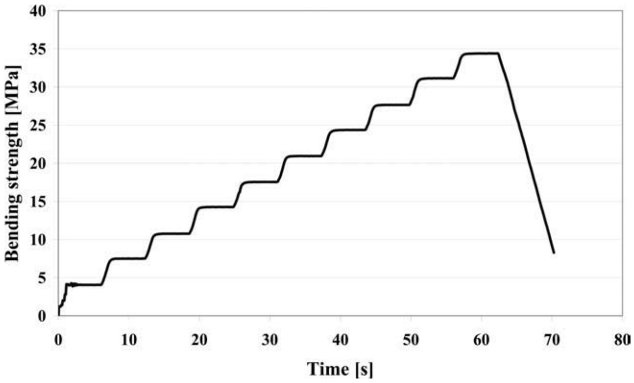

Figure 3 Stepwise loading and bending stresses belonging to the load levels selected and presented in Figure 4.

Mechanical test

The stress optical photographs indicate the presence of residual stresses in the sheets after welding. Three-point bending was performed with the stepwise loading presented in Figure 3 such that a photograph was made at each step. The diagram indicates those stress levels, which are emphasized in Figure 4. It shows the cross section photographs of the specimens machined perpendicularly to the longer axis of the seam, (a) shows the bending for the crown side, (b) from the root side. It can be observed that already before the beginning of the load significant internal stress was present in the HAZ. Color fringes and high order numbers observed on the stress optical photographs can be explained by the double effect of FSW. Color fringes exhibit simultaneously the presence of molecular orientation coming from the rotating movement of the tool and the residual internal stresses caused by the heat effect. Separation of these two effects is extremely complicated. At the same time from the changes of the color fringes in Figure 4, it was possible to draw conclusions on the stress state of various areas of the welded seam (e.g., based on the densification or direction changes of the colored fringes) without numerically determining the stress levels afterwards.

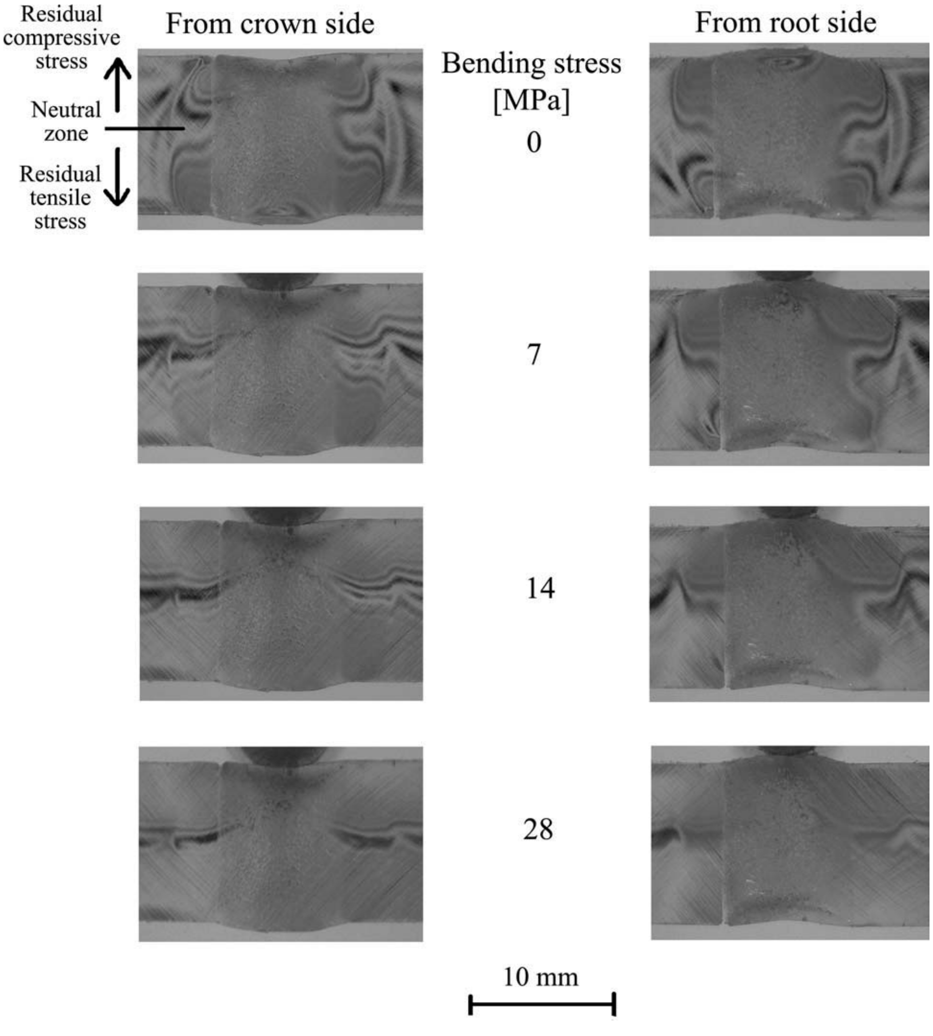

Figure 4 Internal stresses changing with bending stress in the HAZ under (a) crown side loading and (b) root side loading in three point bending tests.

TABLE II
K-Factor with Welding Parameters and Tools

Rotation speed (1/min)	Feed (mm/min)	Tool diameter (mm)	K (−)
750	75	8	80
900	100	8	72
1200	50	8	192
1200	90	12	160
1200	50	12	288
1200	100	8	96
2550	100	8	204
2550	50	8	408

At the half of the sheet thickness there is a stress-free part, which behaves as a neutral section in the seam. From this part downwards residual compressive stress is found on the crown side, while on the root side tensile stress is observable. With increasing bending stress the extension of the stress-free, neutral region decreases also on the root side, but in both cases colored fringes are broken on both sides of the seam. The end of the HAZ can be found in the neighborhood of this breakage point; the welding temperature does not exert any effect at larger distances from the seam. This sharp separation of the HAZ and the raw material can be observed in all friction stir welded seams.

Using the welding parameters and tool data, based on the width of the HAZ a dimensionless K parameter characterizing the FSW technology was created [see eq. (1)]. This factor is proportional to the heat transferred during the welding and contains only parameters characteristic of welding, such as rotation speed, feed rate, and tool diameter. In preliminary experiments, when studying the effect of parameters it has been determined that the increase of feeding rate decreases the heat transfer, that is, they are inversely proportional, while the other two parameters are directly proportional to the heat transfer.

$$K = \frac{\text{rotation}}{\text{feed}} \cdot \text{tool diameter} \tag{1}$$

The results obtained during three points bending of seams obtained with technical and tool parameters shown in Table II were investigated among others in terms of the K-factor (see Fig. 5).

One can see (Fig. 5) that if the K-factor is in the range of 150–400, then the flexural strength of the seam changes smoothly and approximates the flexural strength of the raw material (38 MPa). There is a difference of 2–3 MPa between the values obtained on samples loaded from the crown and root sides respectively, usually lower strength is obtained if the crown side of the seam is located on the crown side. According to the literature,[14–17] the root side reacts more sensitively to tensile stress, which is usually attributed to the presence of root defects. This root defect was eliminated in our preliminary experiments by proper tool adjustment, by pushing the welding tool 9.5 mm deep into the 10 mm thick sheet. Above this depth setting, which can be considered as optimal, the root of the seam over-melted. If the K-factor is above 300 the differences between two loading modes diminish, which can be explained by the proportionately increasing heat transfer. If the K factor increases, the welding temperature in the seam also increases. This temperature has a relatively narrow range, wherein the flow properties of the PETG material are ideal for FSW. If the K-factor (or the temperature) is too low, the quality of the welding decreases (at $K < 150$), the material to be welded does not soften to the required degree, the welded seam contains visibly nonmelted particles. These can even lead to the breakage of the welding tool. If the K-factor is too high (i.e., if the seam is overheated), again bad seam is obtained, as the low viscosity material obtained at too high temperature is completely squeezed out by the welding tool from the seam (above $K > 500$ we could not obtain welded joints).

The fracture surfaces of the seams were studied by SEM. Figure 6(a) shows the micrograph of the fracture surface of a seam obtained at $K = 400$, one of the optimum values. The lower part of the figure shows the elongated, the upper part the compressed side. It can be well seen that the part exposed to tensile load undergoes plastic deformation, while above a certain level the fracture happens suddenly on the compressed side too. Figure 6(b) shows the results observed on a weak seam obtained at $K = 85$. One can see that on the lower, elongated part there is practically no deformation, the seam easily separated. On the upper, compressed side one can observe that the material in the seam did not melt completely, the heat transfer characterizing the technical parameters set proved low (the K-factor was

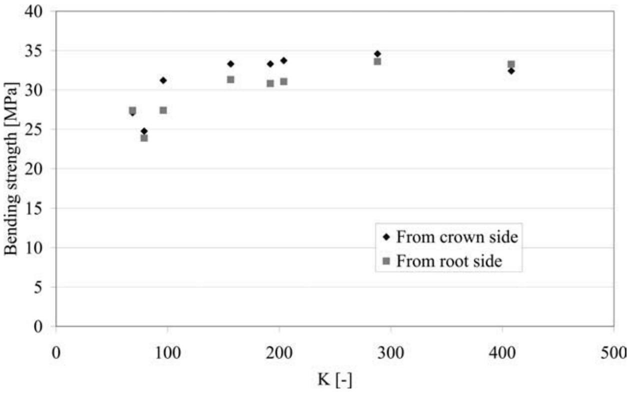

Figure 5 The results of three-point bending as a function of the K-factor.

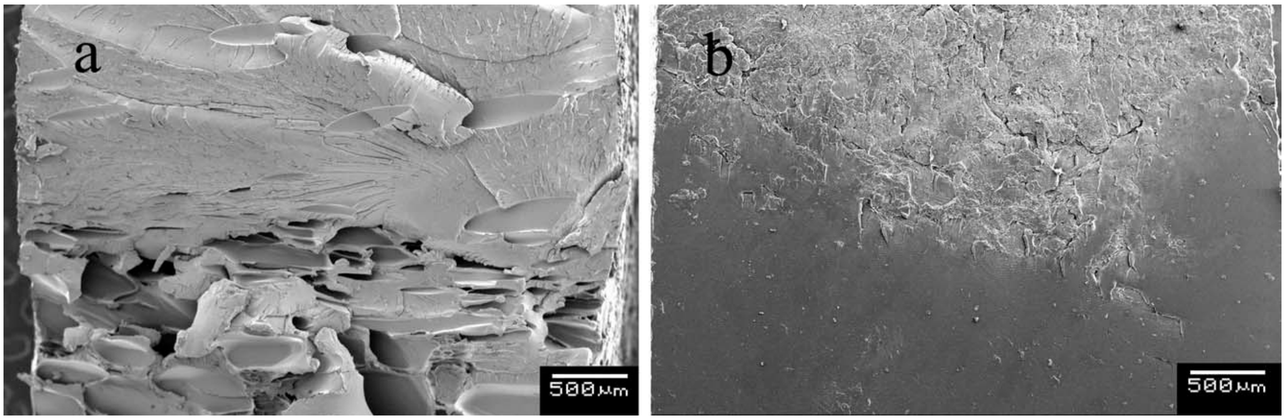

Figure 6 SEM micrograph of the fracture surface of (a) high strength and (b) low strength seam.

too small). SEM micrographs made of the fracture surfaces allow the monitoring of the seam quality from the viewpoint of heat transfer.

Photoelasticity

The interpretation the width of the HAZ is shown n Figure 7. The area being due to the width of the tool can be easily identified, this yields the effective seam, to this is added an area on both sides containing frozen in stresses being due to the high temperature. The average width of these has been taken into account.

Figure 8 shows the relation between the *K*-factor values calculated from the values reported in Table II and the width of the HAZ. One can see that the relation is practically linear, that is, the increase of the *K*-factor is accompanied by the increasing width of the HAZ. Based on the measured values the correlation is very good, taking into account that the exact borderline of the HAZ cannot always be precisely determined on the stress optical photographs. Consequently, the *K*-factor is closely related to the temperature in the seam and in its neighborhood.

The changes of the internal stress in the seams produced by FSW were studied in terms of the settings and tools listed in Table I.

The only difference between the seams shown in Figure 9(a,b) is the rotation speed (1200 and 2500 rpm). It can be well observed that in the case of higher rotation speed changes occurred in a significantly broader range, which can be fully explained by the higher temperature. This is corroborated by the fact that in the case of the higher rotation speed the seam itself is also wider than in the case of the lower rotation speed, where it is just identical with the tool diameter. At the higher rotation speed, it can also be observed that the crown of the seam is strongly sunk, which can also be observed by overmelting.

Another parameter strongly related to rotation speed is feeding rate. Interestingly enough, when this parameter is nearly doubled (from 56 to 105 mm/min), one cannot observe a difference comparable to that observed in the case of changing the rotation speed [Fig. 9(c,d)]. It follows that the feeding rate can be varied within broad limits with a minimum change of the HAZ. When using a high

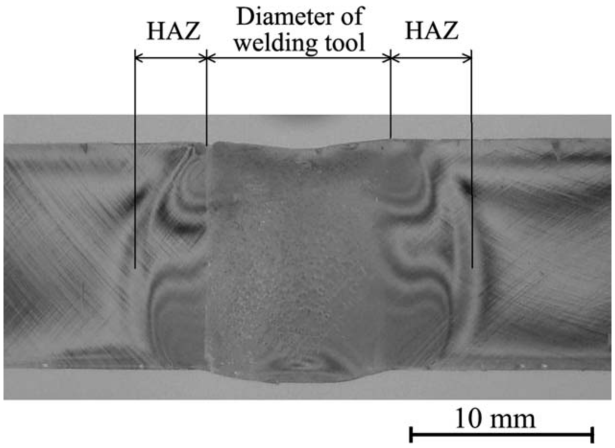

Figure 7 Width of the HAZ on both sides of the seam.

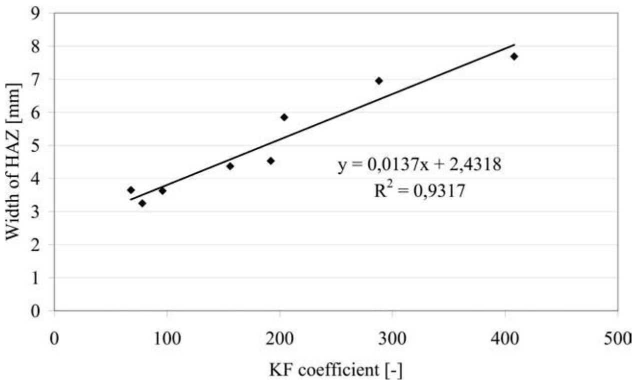

Figure 8 The dependence of the width of the HAZ as a function of the *K*-factor.

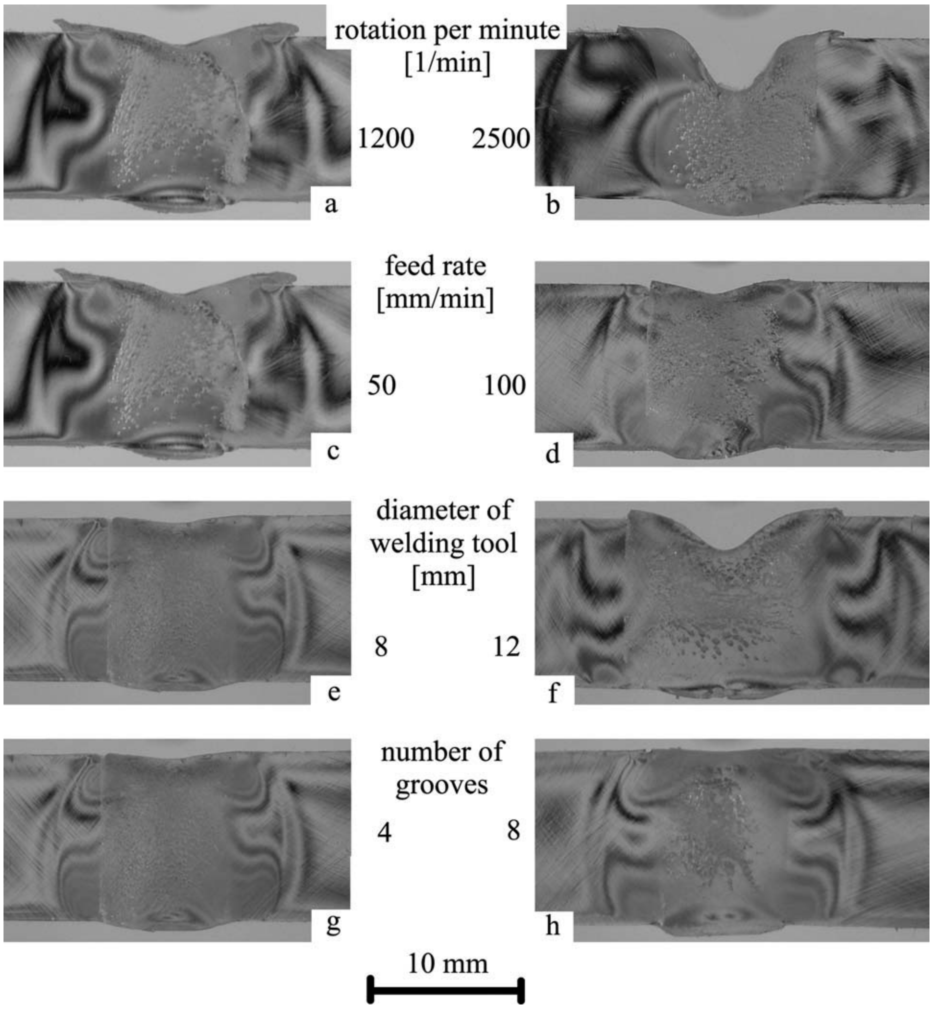

Figure 9 Comparison of seams produced with various welding parameters and with different tools.

feeding rate, however, the force acting on the material in the direction of the feed increases significantly therefore the sheets must be fixed.

The seams shown in Figure 9(e,f) were made with different tool diameters (8 and 12 mm). In the case of larger diameter, a significant sink is formed on the crown side, similarly to the effect of the larger rotation speed.

An interesting effect was observed when changing the number of edges of the tools [Fig. 9(g,h)]. The diameter and the groove slope of the tools are identical, so only the number of edges is different. If there are more edges, there are more grinding interfaces, so a more intense heat evolution is expected. The minor difference observed on the root side of the two seams seems to confirm this, even if weakly. It can be observed with the tool with eight edges that the colored fringes are less frequent, while in the case of the tool with fore edges they are more frequent. The width of the HAZ, however, increases in the case of the tool with eight edges. This phenomenon was observed at the larger tool diameter, that is, in the case of more intense heat evolution, but here the rotation speed is constant; therefore, it is proven that more edges result in more heat evolution.

SUMMARY

Internal stresses in the HAZs of PETG seams produced by FSW were studied by photoelasticity and the flexural strength of the seams was studied by three point bending. Using the welding and tool parameters, a *K*-factor characterizing the welding process was created that contains the rotation speed, the feed rate and the tool diameter as parameters. It has been shown that this *K*-factor is related to the HAZ of the seam. The mechanical properties of the seam can also be related to the *K*-factor, a proper quality welded joint can only be produced in the 150–400 range. It was shown too that a bending type stress arises in the welded seam; the crown side is exposed to compression, the root side to tension.

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