

The International Journal of Advanced Manufacturing Technology

The effect of weld line on tensile strength of polyphenylsulfone (PPSU) in ultrasonic micro-moulding technology

--Manuscript Draft--

Manuscript Number:	JAMT-D-18-02464R2
Full Title:	The effect of weld line on tensile strength of polyphenylsulfone (PPSU) in ultrasonic micro-moulding technology
Article Type:	Original Research
Keywords:	ultrasonic micro-moulding; ultrasound technology; polyphenylsulfone; PPSU; Amplitude; weld line
Corresponding Author:	Joaquim Ciurana, Professor University of Girona Girona, SPAIN
Corresponding Author Secondary Information:	
Corresponding Author's Institution:	University of Girona
Corresponding Author's Secondary Institution:	
First Author:	Tomasz Dorf, MSc
First Author Secondary Information:	
Order of Authors:	Tomasz Dorf, MSc Ines Ferrer, PhD Joaquim Ciurana, Professor
Order of Authors Secondary Information:	
Funding Information:	
Abstract:	Melting and processing polymers via ultrasonic vibration is now possible, but the weld line that results and the influence it may have on tensile strength has yet to be investigated. When producing products characterised by high mechanical strength, the parameters of the process should be considered in relation to the weld line on the parts. Here, four sets of process parameters (three using ultrasonic technology and one using the conventional process) were used to produce parts whose weld line strengths were then determined and compared. Amplitude values were found to significantly affect the V-notch size and consequently the strength of the samples. Samples from parameters where the amplitude was high (52.2 μm , 58 μm) were characterised by a strength similar to that reached with conventional injection-moulding technology. Moreover, the experiments showed that when using specific process parameters the weld line strength is characterised by values akin to those of pure samples. Finally, the investigation revealed that this novel technology is a potential alternative to micro-injection moulding of high requirement polymers such as PPSU.

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Tomasz Dorf
ChM sp. z o.o.,
Lewickie 3B, 16-061 Juchnowiec Kościelny
POLAND
tomasz.dorf@gmail.com

Inés Ferrer
Department of Mechanical engineering and civil construction
Universitat de Girona
Maria Aurèlia Capmany, 61
17003 Girona, SPAIN
ines.iferrer@udg.edu

Joaquim Ciurana¹
Department of Mechanical engineering and civil construction
Universitat de Girona
Maria Aurèlia Capmany, 61
17003 Girona, SPAIN
quim.ciurana@udg.edu
Joaquim.ciurana@gmail.com

¹ Corresponding author, Tel: +34 972 418265, Fax: +34 972 418098

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Tomasz Dorf¹, Inés Ferrer², Joaquim Ciurana²

¹ ChM sp. z o.o., Lewickie 3B, 16-061 Juchnowiec Kościelny, POLAND. tomasz.dorf@gmail.com

² Department of Mechanical engineering and civil construction, Universitat de Girona, Maria Aurèlia Capmany, 61 17003 Girona, SPAIN. ines.iferrer@udg.edu, quim.ciurana@udg.edu

Abstract

Melting and processing polymers via ultrasonic vibration is now possible, but the weld line that results and the influence it may have on tensile strength has yet to be investigated. When producing products characterised by high mechanical strength, the parameters of the process should be considered in relation to the weld line on the parts. Here, four sets of process parameters (three using ultrasonic technology and one using the conventional process) were used to produce parts whose weld line strengths were then determined and compared. Amplitude values were found to significantly affect the V-notch size and consequently the strength of the samples. Samples from parameters where the amplitude was high (90%, 100%) were characterised by a strength similar to that reached with conventional injection-moulding technology. Moreover, the experiments showed that when using specific process parameters the weld line strength is characterised by values akin to those of pure samples. Finally, the investigation revealed that this novel technology is a potential alternative to micro-injection moulding of high requirement polymers such as PPSU.

Key words: ultrasonic micro-moulding; ultrasound technology; polyphenylsulfone; PPSU; amplitude; weld line.

1. INTRODUCTION

Ultrasonic micro-moulding technology that uses vibrations above 20 kHz to plasticize polymers is now available. The main advantage of the process contrary to the standard micro injection moulding is its potential to melt and inject the material needed for one cycle. Micro injection moulding technology as one of the most efficient process is dedicate especially for the large scale production. The weight of the micro parts often represents only a few percentage of the whole shot weight what can lead to material waste in a small scale production. Moreover, the polymer is plasticized from a thermal and mechanical heating supplied by a screw in a barrel and small dozes increase a risk of long residence time, which can cause degradation of the polymer in a barrel. In ultrasonic micro-moulding technology, where the material is processed by ultrasounds, there are no heaters so there are no residence time. Additionally, the molten polymer shows reduced viscosity, which allows the use of lower moulding pressures compared to the standard injection moulding processes. All these advantages put ultrasonic micro-moulding process as a real candidate for medical sector where single parts made from expensive polymers are sometimes needed and the purity and mechanical properties requirements prohibit the use of re-granulate. It is very important that applications made of plastics should characterised by the same properties regardless of the technology in which they were made. Thus, it is crucial to constantly compare ultrasonic micro-moulding technology to the standard micro injection-moulding that has been employed for decades.

1 The ability to plasticize polymeric materials using ultrasonic energy was first reported by W. Michaeli et al.
2 in 2002. [1] when they used the new plastification system in a prototype of a micro injection-moulding
3 machine [2]. The initial version, where a small amount of material was plasticized in an electrically heated
4 cylinder, was later replaced by plastification using ultrasonic energy. Their experiments showed the concept
5 not only to be very efficient in terms of plastification time, but also the processed polyoxymethylene (POM)
6 was characterised by a homogeneous structure. This achievement prompted many researchers to explore
7 the topic further and, as such, a number of investigations where this innovative processing method is
8 applied to different polymers have been carried out to date. Some researchers were interested in
9 investigating the commodity and engineering of the polymers, while others focused on the medical grades.
10 The process's parameters and the influence they have on selected properties of the polymeric parts made
11 from polypropylene (PP) were investigated by W. Michaeli et al. [3], K. Zeng et al. [4] and P. Negre et al. [5].
12 Moreover, high density polyethylene (HDPE) were also processed using ultrasonic moulding by B.Y. Jiang et
13 al. [6], poly(methyl methacrylate) (PMMA) by W. Wu et al. [7] and ultra-high-molecular-weight
14 polyethylene (UHMWPE) by X. Sánchez-Sánchez et al [8] . Medical grades of polymers such as polylactide
15 (PLA), polybutylene succinate (PBS) and polyamide (PA12) were studied by M. Sacristán et al. [9], M.
16 Planellas et al. [10] and J. Grabalosa et al. [11], respectively. Only one study, that by T. Dorf et al., concerns
17 high performance polymers, namely PPSU [12].
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23 As the ultrasonic process has already been proved capable of manufacturing micro parts from different
24 polymers, the next step should be directed towards investigating how the process influences the typical
25 defects of the polymeric parts that conventional injection moulding produces. One such defect is the well-
26 known weld line or knit line that reduces the mechanical properties of the final product as well as affecting
27 its appearance. The weld line forms where two or more streams of material meet and combine together
28 [13]. The strength of the weld line area depends on whether the processing conditions are adequate and/or
29 if the macromolecular chains diffuse through the original interface and provides the weld with a strength
30 close to that of the pure material; otherwise the strength will drop dramatically.
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32 Cheng-Hsien Wu et al., using the Taguchi method, found that four process variables i.e., melt temperature,
33 mold temperature, injection speed, and packing pressure, have the greatest influence on the weld line
34 strength of PP and HDPE polymers [14]. Later on, Lei Xie et al. investigated the relationship between weld
35 line strength and processing parameters in micro injection moulding of polypropylene parts. The results
36 showed that the mould temperature, followed by the melt temperature, most affected weld line strength.
37 Additionally, V notch measurements showed that the size in the middle region of the specimen was larger
38 and deeper than at the edge. Finally, a smaller V notch area leads to a stronger micro weld line [15].
39 Furthermore, these authors also indicated that the gate dimension coupled with processing conditions have
40 a significant effect on the weld line strength of PP and HDPE parts [16]. Researchers not only focused on
41 factors that affect the weld line strength, but also on those that reinforce it. Chang Lu et al. [17] investigated
42 whether the presence of ultrasonic oscillation could enhance the weld line strength of polystyrene (PS) in
43 a way that would improve molecular diffusion. For PS/HDPE blends, they indicated that the melt
44 temperature is crucial to enhancing the strength of the weld line through ultrasonic oscillation. When the
45 melt temperature is 230°C, ultrasonic oscillation can increase the weld line strength of the blends, which is
46 contrary to when the melt temperature is 195°C. The advantage of using ultrasonic oscillation to reinforce
47 weld line strength in micro injection moulding processes was also reported by Lei Xie et al. [18]. They
48 studied two ultrasonic inducing modes and their results showed that Mode 1 when the oscillation is induced
49 from injecting to ejection moment produced better effects than Mode 2 did (the oscillation is induced from
50 injecting moment to packaging finishing). For reasons of reinforcement, the weld line strength using
51 ultrasonic oscillation they introduce is the curving weld line and the almost disappeared V-notch.
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1 In this study, the authors subjected PPSU polymers to ultrasonic micro-moulding technology to investigate
2 the resulting weld line strength. For the first time the weld line formed from this novel technology has been
3 studied. Three different sets of ultrasonic process parameters were used to fabricate the complete filled
4 samples and the best set from which it is possible to obtain the parts characterised by the highest tensile
5 strength were introduced. Furthermore, the results from the best set were compared to those obtained
6 from standard micro-injection moulding technology.

7 8 **2. EXPERIMENTS**

9 10 *2.1. Material*

11 The Radel® R-5100 GY1137 polyphenylsulfone used for this study is a commercially available material
12 supplied by Solvay Ltd. PPSU is an amorphous material characterised by some outstanding properties;
13 particularly its thermal (207°C) and chemical resistance. The material's general properties are shown in
14 Table 1. Thanks to these features, the material is often used in the medical industry for a variety of
15 applications such as nebulizers, humidifiers, flow controls, dental and surgical instruments, fluid containers,
16 heart valve cases, microfiltration apparatus and other kinds of equipment [19], [20].

17 18 *2.2. Micro-moulding equipment*

19 The ultrasonic micro moulding machine Sonorus® 1G, developed by Ultrason S.L., was used to manufacture
20 the small specimens (complying with the EN ISO 527-2/1BB standard) (Fig.1) [21]. The machine mainly
21 consists of a bench, a mould system moved by skates and linear guides, an ultrasonic head and a feeding
22 system that provides the pellets. The ultrasonic head converts the electrical waves, produced by an
23 electronic ultrasonic generator (maximum power 1500 kW), into mechanical vibrating at an ultrasonic
24 frequency of 30 kHz. This mechanical vibration is then transmitted to a sonotrode (Fig. 1). The material
25 being processed begins to melt as it encounters the vibrating sonotrode (this is due to the thermal energy
26 or (latent) heat of fusion, which occurs because of ultrasonic wave absorption and the frictional movement
27 between the surfaces). Next, the melt is pushed by a plunger (maximum available force equal 6000 N)
28 through the channel to the cavity where it is solidified and finally ejected (Fig.2). The adjustable process
29 parameters are: amplitude (μm), force (N) and ultrasonic exposure time (s). It is important to mention that
30 the machine has 9 available levels of amplitude values. The maximum value of 58 μm equals 100%. The
31 amplitude can gradually be decreased in increments of 10 percent, e.g. 90%, 80% and so on. It can be
32 explain by the fact that, the mechanical amplitude is provided by the ultrasonic transducer at full power.
33 Then, the amplitude value can amplify or reduce by means of mechanical elements, such as booster and
34 the sonotrode, what is related to its geometry. For example the standard stepped sonotrode of the Sonorus
35 1G has an approximate gain of 6.25. On the other hand, the machine allows to limit the voltage given to
36 the converter in steps of 10%, which will limit its output amplitude.

37 The temperature of the mould is a key factor as this determines the quality of the finished PPSU part. Mould
38 temperature affects not only the shrinkage and warpage of the part, but also the level of moulded-in
39 stresses [22]. To fulfil the supplier-recommended temperature of 138-163°C, the aluminium mould was
40 built and equipped with an oil circulation flow. As the PPSU polymer must be dried to avoid cosmetic
41 defects, a standard dryer for injection moulding machines was used.

42 43 *2.3. Process parameter settings and sample manufacturing*

44 The experiments were conducted to examine how a set of ultrasonic micro-moulding parameters
45 influenced the weld line strength of the PPSU samples. Earlier, T. Dorf et al., conducted extensive
46 experiments with different process parameters and analysed their impact on the tensile strength of the
47 PPSU specimens. The mathematical model describing the process was presented. The study confirmed that

1 amplitude is the most influential parameter during the processing of the PPSU polymer [12]. That is way
2 amplitude variable was selected as the main process parameter and its levels were modified. The applied
3 process parameters resulted from technological trials, which were performed as screening experiments.
4 The experiments were carried out to avoid the situation where a decrease in strength could have been
5 caused by the degradation of the polymer. Thus, the acceptable processing parameters were characterised
6 by the ability to obtain completely filled samples without visible signs of degradation. Practical experiments
7 starting from the amplitude of 10% allowed to determine the minimum amplitude level of 80%, which is
8 capable to melt the PPSU polymer. Middle amplitude value correspond to the magnification of 10%, whilst
9 the final value is a maximum value available in the machine. Then, for three amplitude levels the ultrasonic
10 exposure time was adopted in such a way that ultrasonic exposure did not degrade the samples. This
11 provided three different sets of process parameters (see the values in Table 2). The force was constant.
12 From each set of processing parameters in Table 2, 15 specimen pieces were manufactured for further
13 analysis.
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15 In addition, the 15 specimen pieces (Fig. 3b) with weld lines for comparative purpose were manufactured
16 from an Arburg Allrounder 350 injection moulding machine equipped with a micro-injection moulding
17 module (see parameters in Table 3). According to the supplier's recommendations, the PPSU granulates
18 were dried at 149 °C for 2.5 h prior to the experiments.
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21 *2.4. Measurements*

22 The manufactured specimens (60 parts, 15 from each set of parameters) were tested for tensile strength
23 σ_M using the MTS Insight 100 kN machine. Tests were performed in compliance with the EN ISO 527-2
24 standard at a test speed of 5mm/s and a data equation rate of 25 Hz. In order to present the results,
25 statistical computations were carried out using the Minitab version 17 software.
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28 Moreover, the morphology of the three samples from each set of parameters fabricated by ultrasonic
29 micro-moulding technology, together with the one sample from conventional micro-injection moulding
30 technology, were inspected by the scanning electron microscopy Quanta 650 FEG from FEI Company. The
31 areas of measurement are present in Fig. 3. Each specimen was cut off across the weld line. Then, the
32 sample was prepared by mounting one piece in the resin and polishing it. Due to the low electrical
33 conductivity of the sample, the test was carried out in a low vacuum mode and the mounted samples were
34 coated with 15nm of carbon to remove the electric charge from the surface of the sample. The images were
35 recorded using a backscattered electron detector (BSE-vCD), which provided a material contrast. Each
36 unmounted sample was imaged at x100, x1000, x4000 and x5000.
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45 **3. RESULTS AND DISCUSSION**

46 *3.1. Tensile strength*

47 Preliminary tests carried out in order to select parameter values to obtain completely filled samples without
48 visual signs of degradation allow to observe the relation between amplitude and ultrasonic exposure time
49 shown in Figure 4. As can be notice decrease amplitude cause increase the ultrasonic duration time.
50 Amplitude directly affect on the melting phase in the way that the heat generated to the melt is based on
51 the square of amplitude. Thus small modification in amplitude value have a great effect on the plastification
52 process, mainly on the duration of it. Interaction between process parameters on tensile strength of PPSU
53 samples in a wider range is described in the work done by T.Dorf et al. [12].
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Table 4 and Figure 5 depict the results from the tensile strength tests of the three sets of ultrasonic micro-moulding technology (US) and that of the conventional injection micro-moulding technology (IM). The weld line strength from the first set of parameters has the lowest values (63.27 MPa) and the highest standard deviations. Weld line strength increased with increasing amplitude level, something which is reflected by the average values of 69.47 and 69.53 MPa, respectively. The highest tensile value and lowest standard deviations were observed in the samples from the conventional injection-moulding technology. As can be seen the micro-injection process is more stable than ultrasonic micro moulding but it must be taken into consideration that, the ultrasonic process is less known contrary to injection moulding. Lower average tensile strength may result from the signs of degradation inside the some of the samples, which influenced the overall result. Both the ultrasonic and conventional processes are able to obtain weld line samples with strengths similar to the samples without weld lines (70 MPa according to supplier specifications).

Tensile strength value, as the most important and measurable characteristic, was compared to find any differences. A histogram of results is shown in Figure 6. As seen on the histogram, the second and third sets of parameters from the US processes produced parts which have a similar average tensile strength and dispersion, whereas the specimens from the first set of parameters are characterised by the lowest strength values and have with the highest dispersions among the results. The specimens with the highest tensile strengths and lowest dispersions came from conventional injection moulding technology.

In terms of sample dimensions, the Ryan-Joiner normality test was performed to assess the normality distribution of the tensile strength [23]. As seen in Figure 6, set no. 1 is not characterised by normal distribution (p -value < 0.01), whereas the results for sets nos. 2, 3 and 4 indicate distribution close to the normal (both p -values > 0.1). The lack of normal distribution in set no. 1 indicates that the process is not stable.

To verify the hypothesis of variance equality, the Levene's test was performed [24]. Results from the test (Figure 8) proved that the variations are different. Using the first set of parameters, namely the lowest amplitude value together with the longest ultrasonic time duration, not only does the lowest mean tensile strength disqualify this parameter setting, but so too does the high level of variations. Higher amplitude levels, 90% and 100%, respectively, cause a decrease the variances. The smallest variability is characterised by the conventional injection micro-moulding process.

3.2. Morphology

The weld lines produced in PPSU specimens were determined. According to the literature, the V-notch has a significant influence on the weld line properties because by decreasing the V-notch, the strength of weld line is increased [14], [18], [25]–[27]. In this paper, the V-notch appeared in each of the processing parameter settings and its impact on the tensile values obtained was examined. In the form of width and depth, Figure 9 shows the weld lines present on the specimen before being cut (left side) after being cut off (as explained in Figure 3), and after polishing (right side). In analysing the specimens from the ultrasonic micro-moulding process (sets 1,2 and 3), it can be seen that the biggest V-notch depth (422.5 μm), which is characterised by the lowest average tensile strength of 63 MPa, is observed in the first set of ultrasonic parameters. Increasing the amplitude level firstly to 90% and then to 100%, caused a decrease in the V-notch depth to 4,6 μm and 2.8 μm , respectively, which led to the improved mechanical properties of the samples. It can be also noticed that such as small changes of V-notch depth between the 2nd and 3rd parameters settings does not influence on tensile strength values and there are resulting in comparable values. This may be explained by referring to the experiments performed by W. Wu et al. [7]. They reported that amplitude level has a significant impact on the interfacial friction phenomenon and their results

1 showed that the average heating rate increased from 460.4 to 1687.5°C/s as the amplitude increased from
2 10 to 30 µm. Moreover, PMMA granulates were plasticized from 30 to 160°C. The higher amplitude level
3 is, the greater the ultrasonic plasticized rate is. Hence, the amplitude level in the ultrasonic micro-moulding
4 process directly affects the melting temperature and it is well-known from the conventional injection
5 moulding process, that higher temperature values decrease the size of the v-notch. Experiments also
6 showed that V-notch width does not influence the weld line strength. This is similar to the conclusion drawn
7 by E. Debondue et al. [28]. The smallest V-notch was observed in the samples fabricated using the
8 conventional micro-moulding technology.
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10 **4. CONCLUSIONS**

11 The effect weld lines have on the tensile strength of polyphenylsulfone in ultrasonic micro-moulding
12 technology was investigated. Three different amplitude levels and ultrasonic exposure times were used to
13 fabricate samples from which tensile strength values were then determined and compared to the tensile
14 values of the samples made using a conventional injection-moulding process. The experiments conducted
15 allows the following conclusions to be drawn:
16

- 17 1. Both in ultrasonic and conventional processes using appropriate parameters the presence of weld
18 line do not reduce tensile strength of the samples. Similar values for PPSU samples without weld
19 lines were reported by T. Dorf et al. [12].
- 20 2. Weld line strength from the second and third sets of parameters for the ultrasonic micro-moulding
21 technology is comparable to that from conventional injection micro-moulding technology, but the
22 process is more unstable.
- 23 3. The depth of V-notch directly affects the strength of the weld line in such a way that its
24 magnification reduces the properties of the weld line.
- 25 4. Amplitude levels directly affect the formation of the weld line. The higher its value is, the greater
26 the weld line strength.
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28 Performed experiments gives the knowledge of the weld line formation in ultrasonic micro-moulding
29 process. Results proved the ability to utilize the ultrasound to produce micro parts from PPSU as an
30 alternative method to micro injection moulding technology overcoming all limitations resulting from
31 standard technology in case of low volume production. Features, such as low energy consumption, material
32 savings, low moulding pressures will definitely lower the production costs and allow the products of high
33 performance applications to be more affordable when single parts needed.

34 In future works it is highly recommended to do more experiments for better understanding the ultrasonic
35 micro-moulding process. The experimental plan should be extended for 20 kHz frequency, which allows to
36 utilize higher amplitude at the same ultrasonic power level. Additionally, mathematical modelling in the
37 terms of energy balance and thermodynamic laws would be beneficial to estimate nonlinear polymer
38 behaviour in the process.
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40 **5. ACKNOWLEDGEMENTS**

41 The authors would like to acknowledge the financial support received from the Spanish Ministry of
42 Economy and Competitiveness (MINECO) through the PhD scholarship grant DPI2016-77156-R and from
43 the University of Girona (Spain) through the grant MPCUdG2016/036. The authors would also like to
44 express their gratitude to ChM sp. z o.o. and Ultrason S.L. for their support and assistance.
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The effect of weld line on tensile strength of polyphenylsulfone (PPSU) in

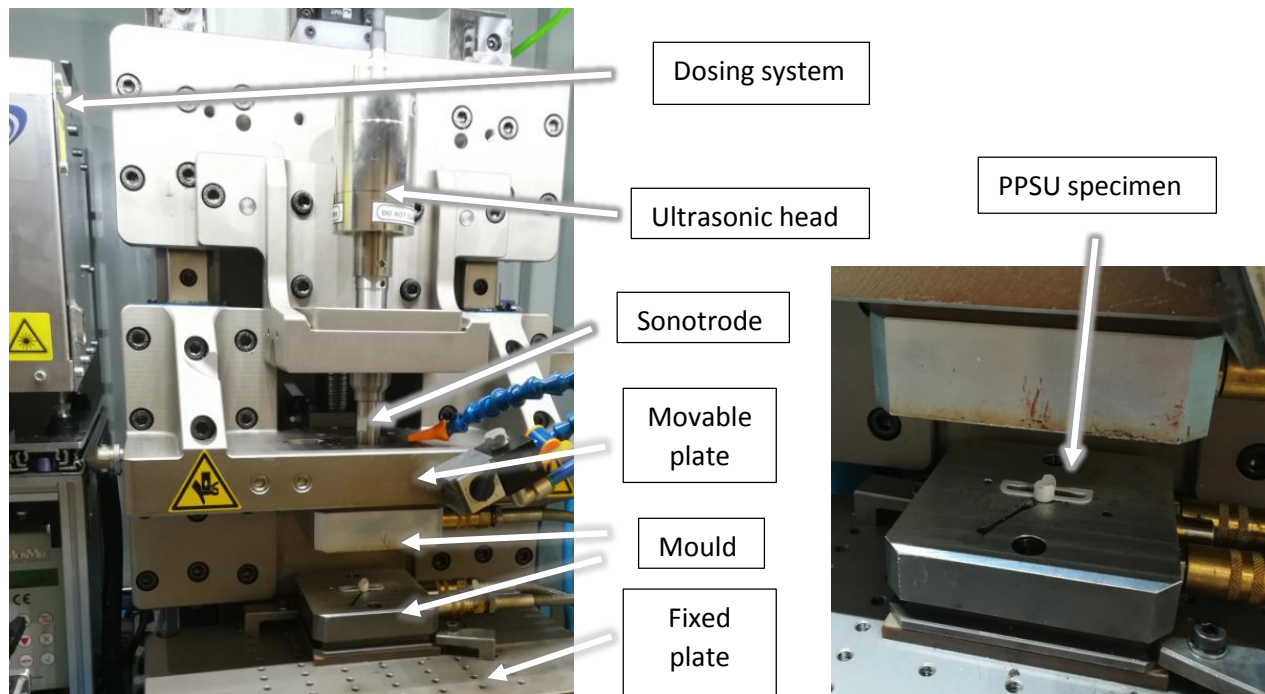


Fig.1. Experimental setup: a) Sonorus® 1G moulding machine, b) mould

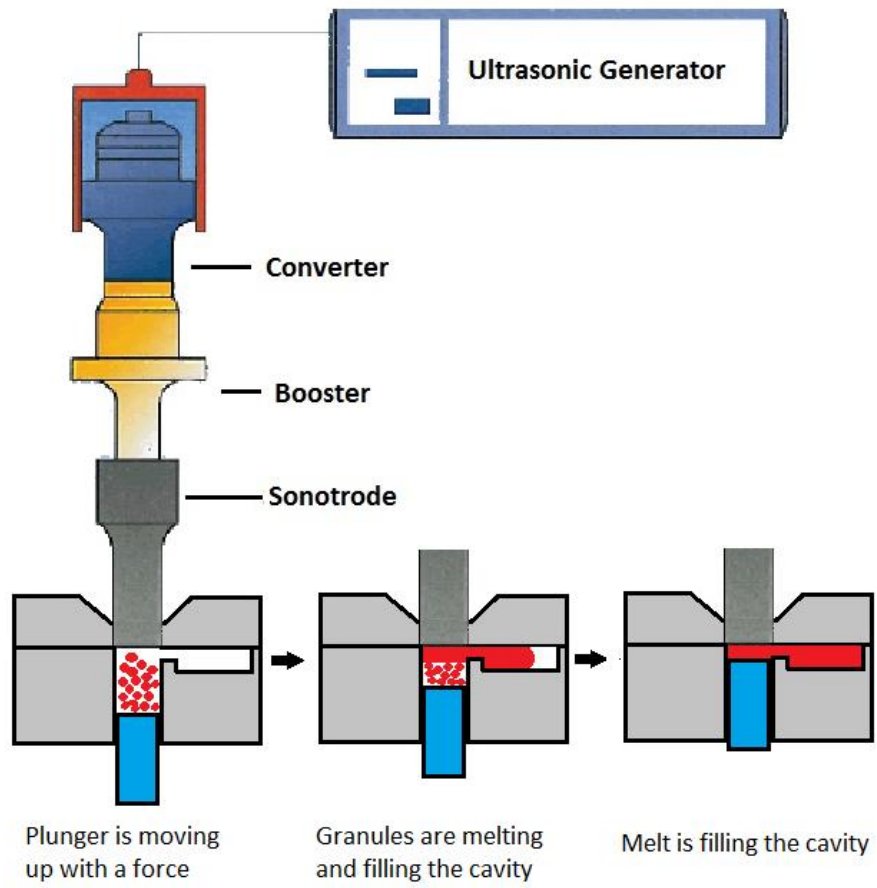
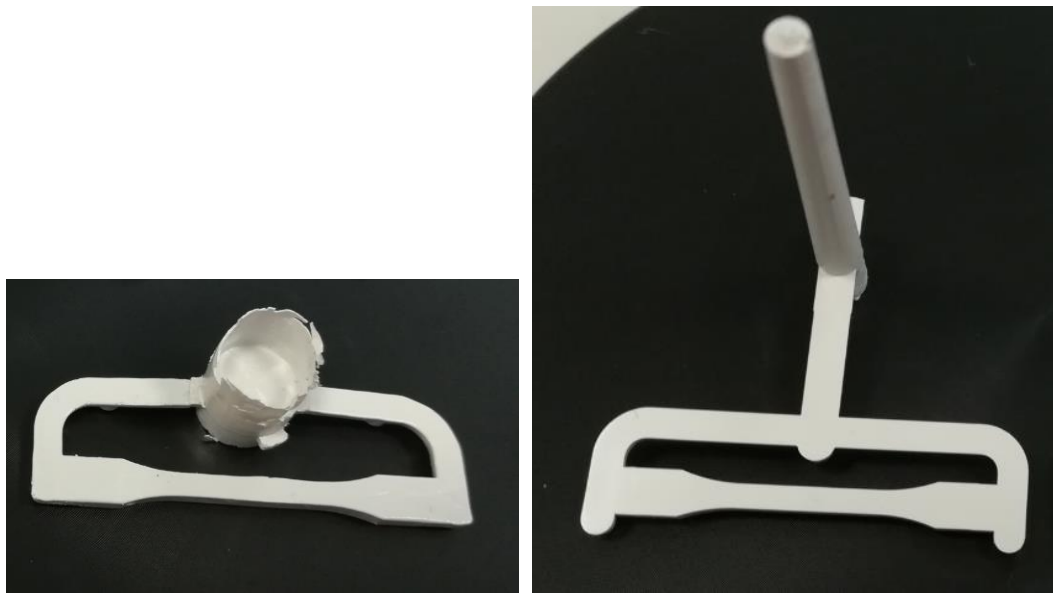


Fig.2. Scheme of the ultrasonic micro-moulding process



a)

b)

Fig.3. Mouldings from: a) ultrasonic micro-moulding process vs b) micro-injection moulding process

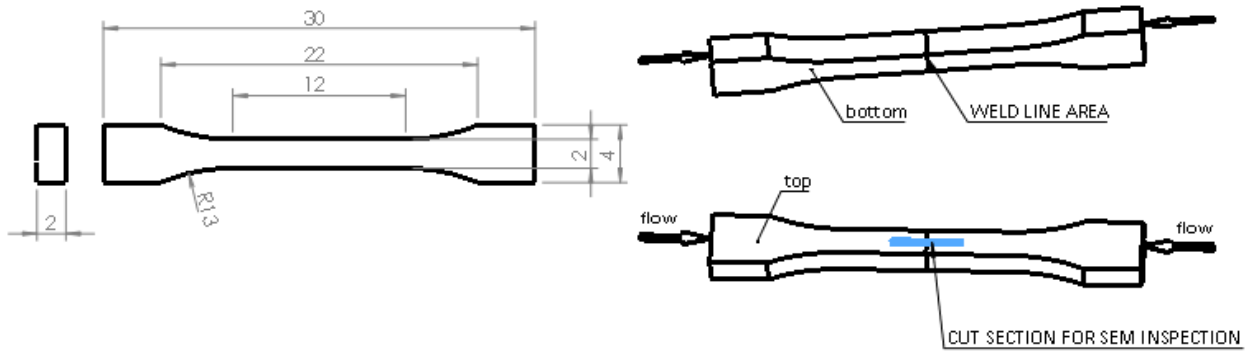


Fig.4. Dimensions of manufactured specimen (unit: mm) and scheme of SEM areas of weld line

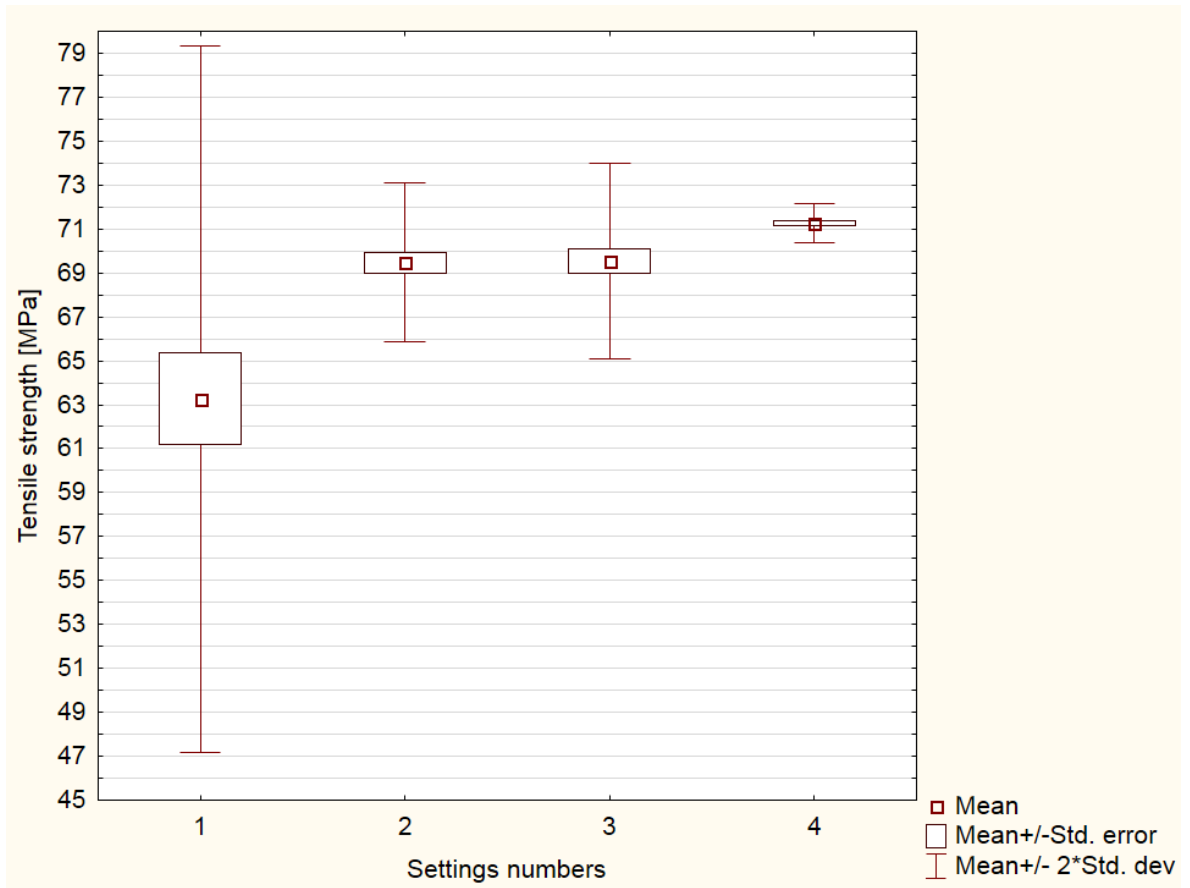


Fig.5. Comparison of samples from different sets of parameters and technologies

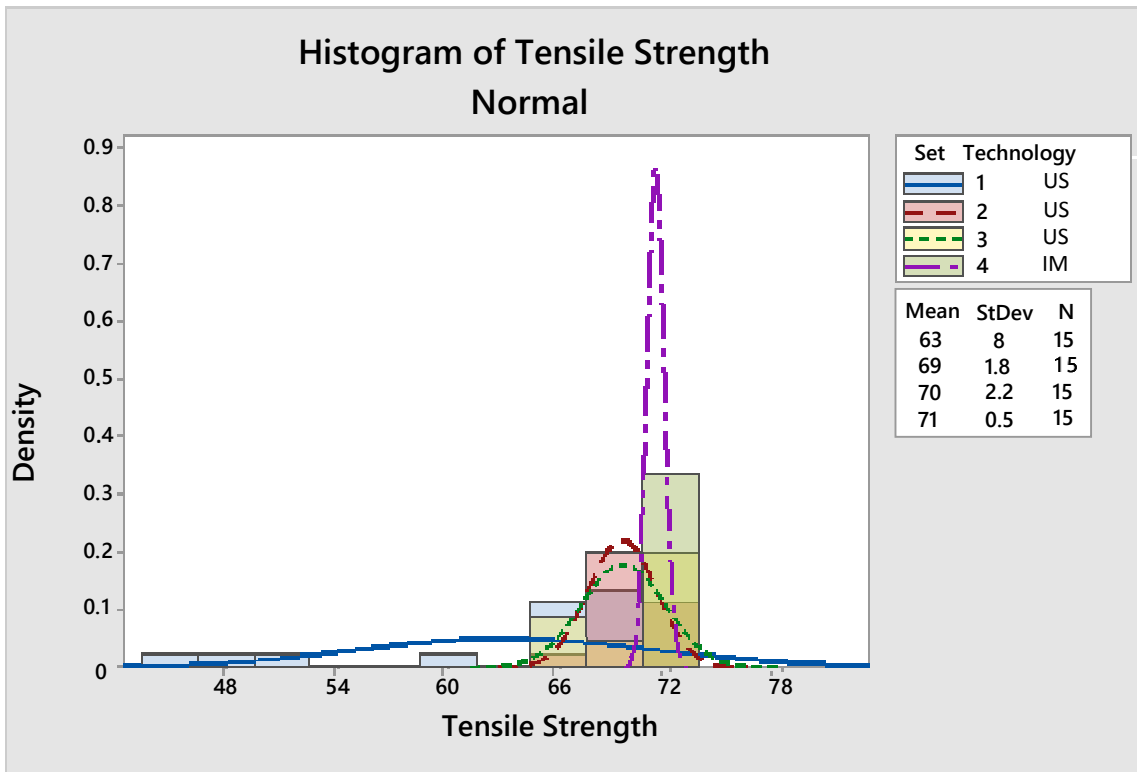


Fig.6. Histogram of tensile strength

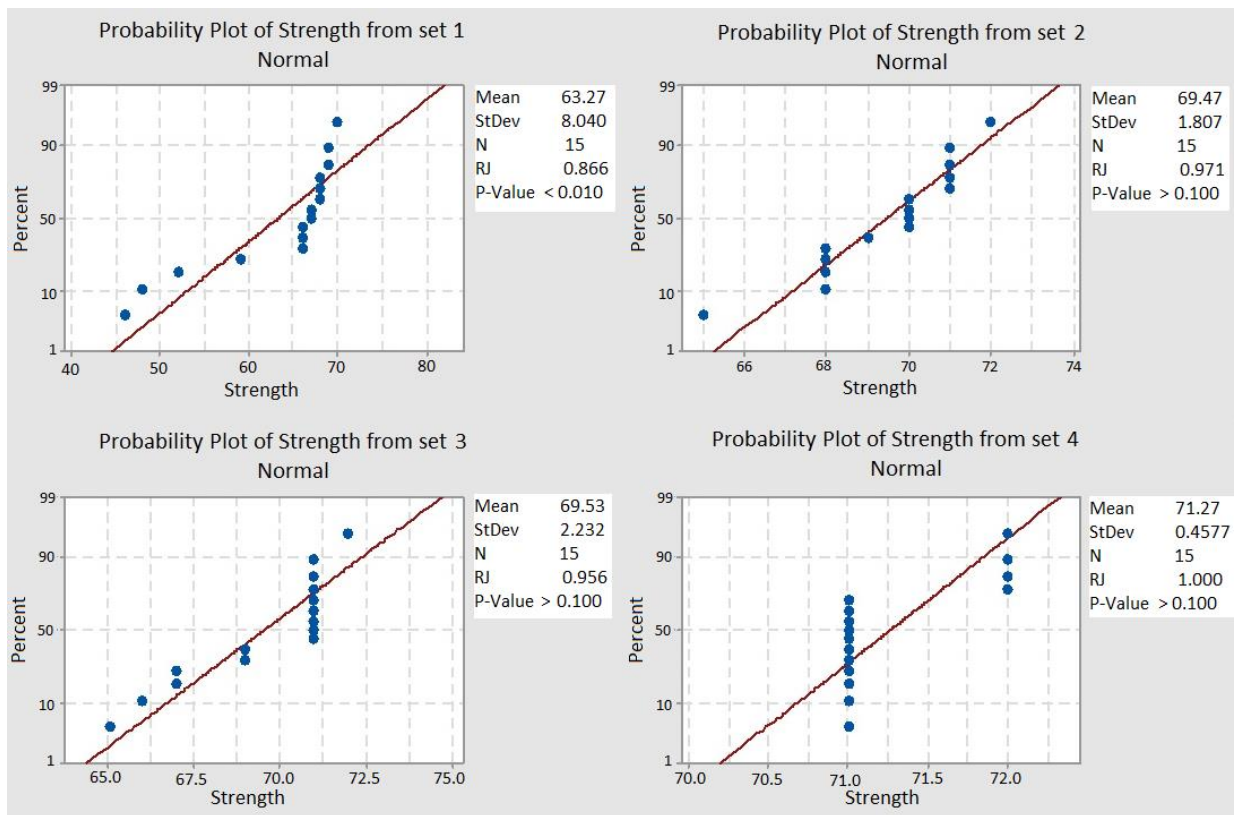


Fig.7. Probability plot of samples strength

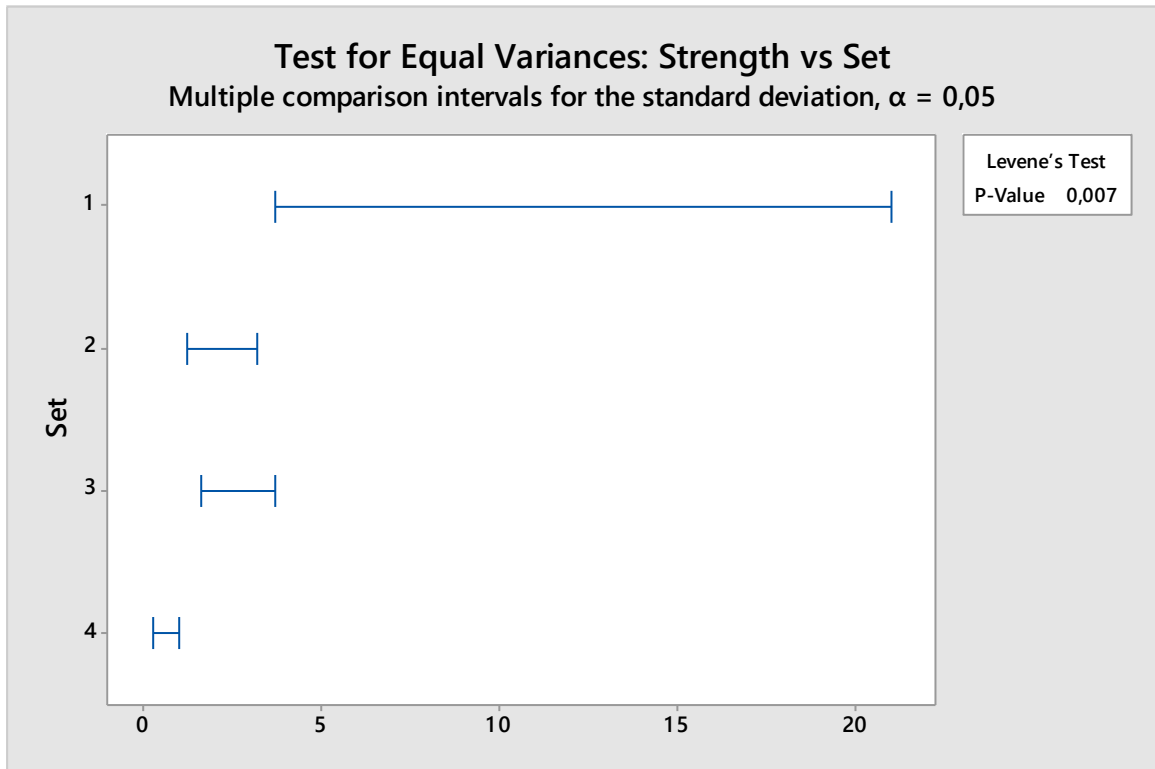


Fig.8. Test for equal Variances: Tensile strength vs Strength

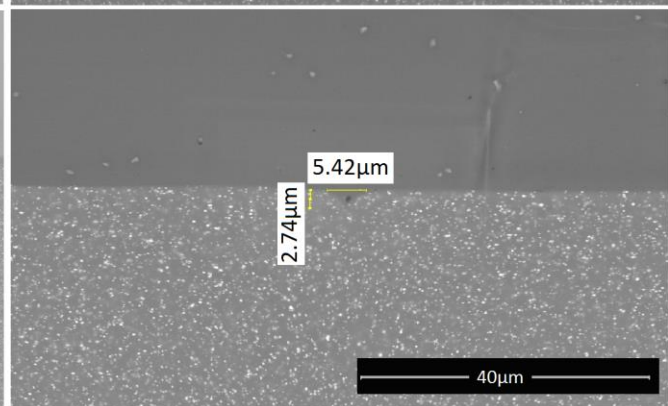
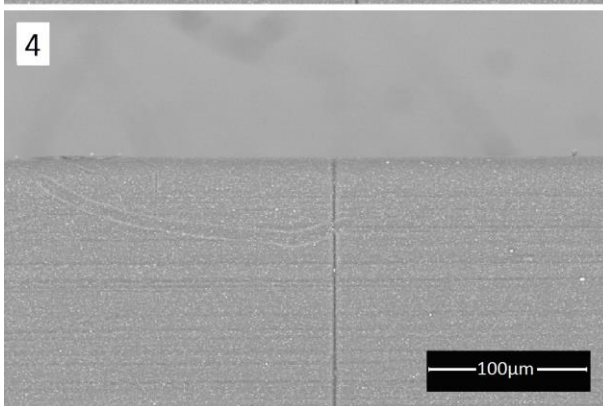
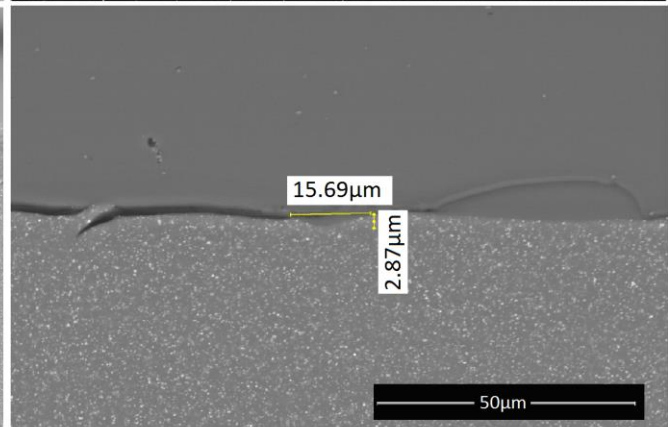
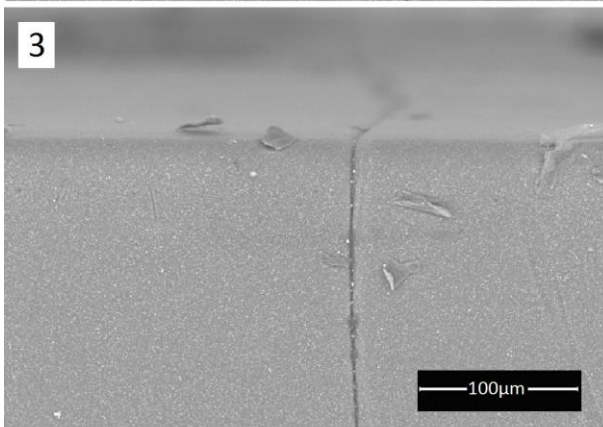
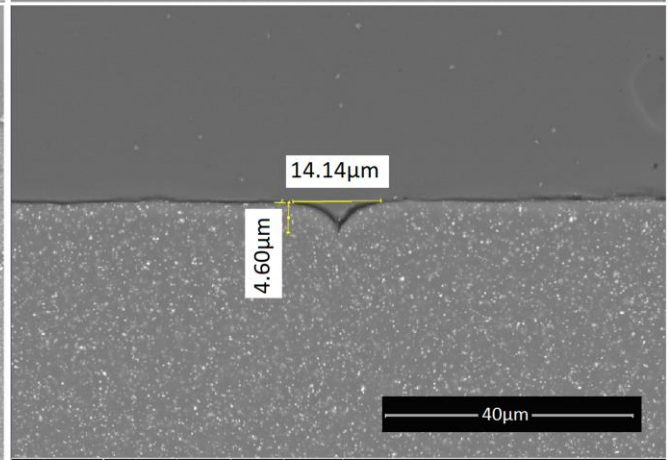
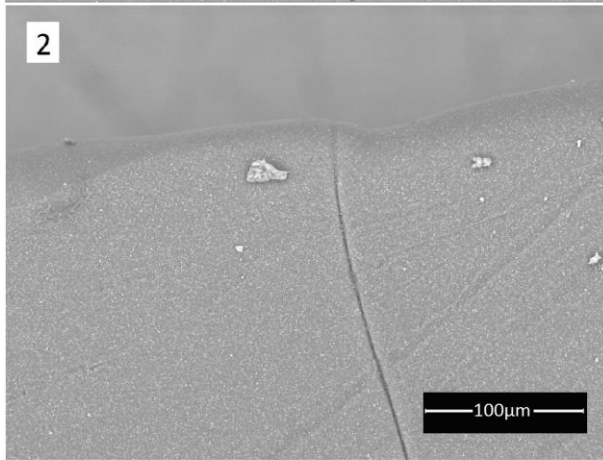
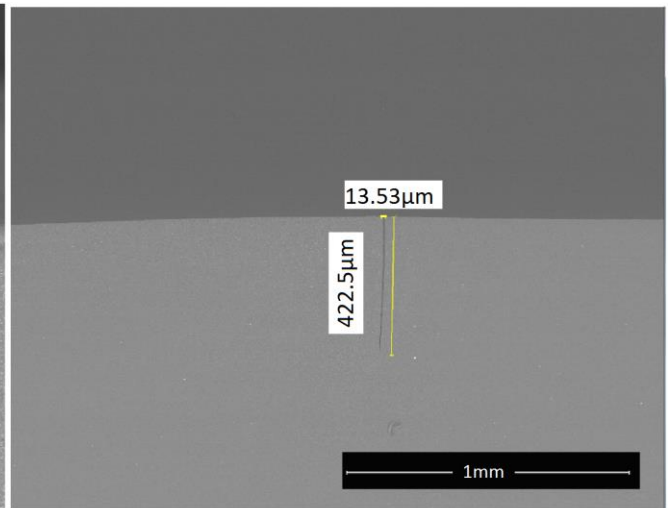
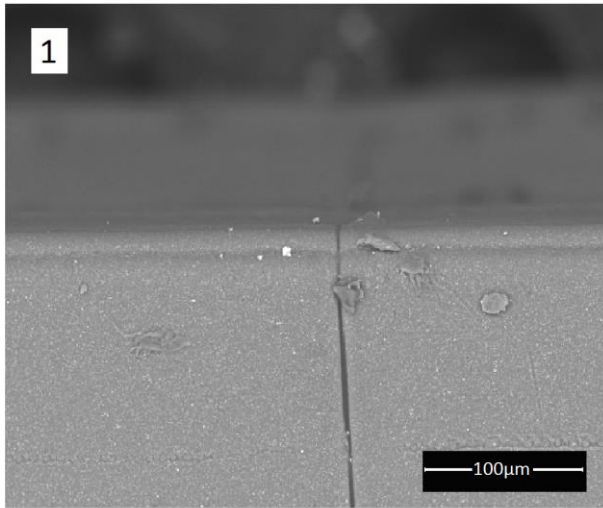


Fig.9. SEM micrographs of weld line from ultrasonic micro-moulding (1,2,3) and injection micro-moulding (4) processes

The effect of weld line on tensile strength of polyphenylsulfone (PPSU) in

Table 1. General property of PPSU

Properties	Values
Density (g/cm ³)	1.30
MFR (g/10 min)	17
Tensile stress (MPa)	70
Heat deflection temperature (°C)	207 (1.82 MPa, unannealed)
Thermal expansion coefficient (μm/m °C)	56
Glass transition temperature (°C)	220
Thermal conductivity [W/(m K)]	0.35
Dielectric strength (kV/mm)	14

Table 2. Processing parameter values for ultrasonic micro-moulding

Parameters	Unit	Values		
		1	2	3
Amplitude	%	80	90	100
Ultrasonic exposure time	s	5.5	4.8	4
Force	N	2000		
Mould temperature	°C	145		
Frequency	kHz	30		
Cycle time	s	20		

Table 3. Processing parameter values for micro-injection moulding

Parameters	Unit	Values
Injection pressure	bar	1000
Injection time	s	1
Packing pressure	bar	1000
Packing time	s	3
Mould temperature	°C	145
Melt temperature (nozzle)	°C	375
Cycle time	s	25

Table 4. Average tensile strength values and dispersion

Technology	Set No.	Average tensile Strength [MPa]	Standard deviation (s)	Variance (s²)	Mean absolute deviation (MAD)
Ultrasonic micro-moulding (US)	1	63.27	8.03	64.63	6.41
	2	69.47	1.80	3.26	1.44
	3	69.53	2.23	4.98	1.89
Injection micro-moulding (IM)	4	71.27	0.45	0.20	0.39