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# Laser welding of thermoplastics: An overview on lasers, materials, processes and quality

quality are discussed.

Luis F.F.F. Gonçalves<sup>\*</sup>, Fernando M. Duarte, Carla I. Martins, Maria C. Paiva

IPC—Institute of Polymer and Composites, University of Minho, 4804-533 Guimarães, Portugal

ARTICLE INFO	A B S T R A C T
Keywords: Laser welding Thermoplastic Welds Quality	This paper offers an overview on the laser welding techniques for thermoplastic materials. The emphases are on four major aspects of the laser welding of plastics: (a) the laser systems, (b) thermoplastic materials, (c) process parameters and finally (d) characterization methods for materials and welded parts. The review focuses mainly in Laser transmission welding, including some technological variants. After an introduction to the laser welding fundamentals, the absorber-based laser transmission welding is described first. This is followed by absorber-free laser welding. The thermoplastic materials most used in laser welding are then reviewed and discussed, with focus on both welding parts of the same material and welding parts of dissimilar materials. Subsequently, the several laser welding processes or techniques are discussed. The next section discusses the main control variables or process parameters and their effects, including the effects of polymer compositions and pre-welding conditions on the laser welding process. Finally, several characterization techniques that are used to evaluate the weld

## 1. Introduction

Plastic welding is a process used in the production of many plastic devices to join different components in a single part. The surfaces to be joined are melted and the formed interfacial molten layer is subsequently solidified under pressure. There are many available techniques for welding thermoplastics, namely: hot-tool welding, in which the surfaces intended to be joined are heated by direct contact with a hot metallic plate [1], ultrasonic and linear vibration welding, in which heating is obtained by vibration and friction [2,3], resistive implant welding and dielectric welding, in which heat is generated from an electromagnetic heat source [4,5] and laser welding, in which a laser beam is used to melt the thermoplastic material in the joint region [6,7]. Laser welding presents many advantages when compared with the other welding methods. It requires a small amount of heat applied to a limited area and shows higher joint strengths as well as weld seam of higher quality. In addition, it enables the welding of complex shapes. Other advantages include no contact between the parts and the welding tools and no mechanical stress on the components. On the other hand, laser welding is more sensitive to polymer material, processing history, pigmentation and additives. This technique presents distinctive processing and performance features, allowing for a local and accurate

welding of films, sheets or moulded parts.

In general, there are 4 common steps on the laser welding process of thermoplastics (Fig. 1): i) Positioning of the parts to be welded. Depending on the technique to be used, the parts must be placed so that the part that is transparent to laser radiation is facing the laser beam. In addition, the parts must be in close contact; ii) Heating of the interface region where the joining will take place. The heating process may vary, depending on the part geometry, the desired end product and the materials used. iii) Application of pressure, intended to promote welding through molecular chain diffusion and formation of molecular entanglements at the interface. It can occur simultaneously with the heating phase and in a subsequent step. iv) Cooling phase. During cooling, the interface assumes the necessary stability so that the parts can be handled without compromising the joint. Typically, the cooling phase occurs while keeping the pressure at a lower value than that used in step iii).

Laser welding technology is strongly influenced by the interaction of laser radiation with the material. The radiation that strikes at the surface of a material can be reflected, transmitted and/or absorbed. However, with this technology the laser radiation must be efficiently absorbed in the precise location where the weld must be formed. The absorbed light is locally transformed into heat. The heat causes the polymer to melt, allowing segmental motion, intermixing of polymer chains and the

\* Corresponding author. *E-mail address:* luisf@dep.uminho.pt (L.F.F.F. Gonçalves).

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Fig. 1. Welding steps in laser welding.

formation of entanglements. Upon cooling, it solidifies forming the weld. The interaction of the radiation with the material surface depends on the type of material and additives content, and on the laser characteristics (wavelength, power, spot size or shape and beam quality). Laser radiation from far InfraRed (IR) laser sources (10600 nm) is readily absorbed by the surface and is normally used for direct welding [8,9]. On the other hand, most polymers show high transmission of light (i.e. low absorption) in the range 400-1600 nm. The laser radiation from laser sources operating within this range are mainly transmitted by the polymer and so, they can only be used for laser transmission Welding (LTW) [10,11,12,13]. In this case, an absorber must be added to one of the parts to be welded. The development of new types of lasers with a longer wavelengths (1500-2200 nm) has opened the possibility of welding transparent thermoplastics without the use of additives (absorbent layer or agents) due to the inherent absorption of radiation at these wavelengths by thermoplastics [14].

Laser welding finds increasingly new applications, ranging from medical industry (assembling of every sort of containers and filters for liquids, joining of bags, pipes, etc.) [15], electronics (assembling mobile telephones, keyboards, etc.) to automotive industry (from multimedia panels to headlight casings)[14,16]. By all that, laser welding of thermoplastics has become an independent segment of laser technology and has a significant scientific and industrial potential. There are some review papers on this specific subject, namely Hilton et. al. (2003) [17] and Moskvitin et. al. (2013) [14]. Amanat et. al. published a paper addressing the welding methods for joining thermoplastic polymers for the hermetic enclosure of medical devices [5]. More recently, Acherjee has published three reviews focusing specifically on LTW [18,19,20]. Dave et. al published a review addressing the LTW of semi-crystalline polymers and their composites [21]. Shin et al. published a more comprehensive paper covering aspects of laser technology other than just laser welding. The other topics covered include laser additive manufacturing, laser-assisted machining, laser forming and laser surface texturing [22]. Quazi et. al published a review covering the research accomplished in the field of laser welding of biomedical devices and implant materials [23]. Given the scientific and technological evolution on the subject, a new discussion encompassing lasers, materials, process and quality is justified in this review paper.

The aim of this paper is to review certain aspects of laser welding of thermoplastics, with main focus on laser systems, materials, process parameters and quality monitoring techniques. The paper begins with a general introduction to welding fundamentals (Section 1), stressing the main aspects and advantages relative to other welding methods, and is followed by sections focusing on various aspects of laser welding. The Section 2 begins by discussing the various laser systems used in laser welding of thermoplastics. Section 3 then focuses on LTW in particular, the most common laser welding technique used today. Some technological variants to the technique, such as the laser welding process using filler material, are also discussed. In the next section, the LTW technique without the presence of additives is discussed, a technique made possible by the advent of lasers emitting in the 1500-2200 nm range. Section 5 discusses the materials most used in laser welding of thermoplastics, reviewing and discussing several examples of their use. The first subsection deals with laser welding of parts of the same material, followed by laser welding of parts of dissimilar materials. This is followed by the discussion of the various LTW variants or techniques, namely contour welding, simultaneous welding, quasi-simultaneous welding, and mask welding, in Section 6. Section 7 discusses the main control variables or process parameters and their effects, including the effects of polymer compositions and pre-welding conditions (thickness) on the LTW process. Finally, is Section 8, several characterization techniques that are used to evaluate the weld quality are discussed.

#### 2. Lasers used for welding thermoplastics

The first demonstrations of laser welding appeared in the 1970s, especially for welding steel sheets or stainless steel [8,9]. However, only in the 1990s has become a widely used welding technique in several applications, due to the improvement in laser sources and methods. Currently, there are four main types of lasers used in welding of thermoplastic materials. They are described in Table 1 in terms of its wavelength, efficiency and beam quality. Fig. 2 shows their emission wavelengths positioned in the electromagnetic spectrum.

The first laser to be used in welding of plastics was the CO<sub>2</sub> laser in 1970 [24]. The  $CO_2$  laser radiation (wavelength of 10.6  $\mu$ m) is promptly absorbed at the surface layers of thermoplastic materials and the material will heat up and melt, making possible the joining. In CO<sub>2</sub> lasers, the beam is guided from the source to the workplace through mirrors. Only a few studies have been reported using CO<sub>2</sub> laser welding. Casalino et. al. [25] used CO<sub>2</sub> laser radiation to perform laser welding of polypropylene (PP), high density polyethylene (HDPE) and lower density polyethylene (LDPE) in a butt joint configuration to study the thermomorphologic and mechanical behaviour of moulded thermoplastic polymers during the laser welding. Another study involving laser welding of polymethyl methacrylate (PMMA) and HDPE using CO<sub>2</sub> laser radiation was reported by Sabah et. al [26]. Coelho et al. [27] also used CO<sub>2</sub> laser radiation in welding experiments of PP, HDPE and LDPE. More recently, Grififths et Dowding [28] performed transmission laser welding of polyethylene film to a polypropylene substrate using CO<sub>2</sub> laser source with wavelength 10,600 nm. The thermal activation of the adhesive in the polyethylene film was identified as the bonding mechanism. Besides welding, CO2 laser light is commonly used for cutting

Table 1	
Laser types used for welding plastics [8].	

	Nd:YAG	Diode	Fibre	CO <sub>2</sub>
Wavelength (nm)	1064	780–980	1000–2100	10,600
Efficiency (%)	3	30	20	10
Beam quality	High	Low	High	High

Efficiency is the percentage of the electrical power consumed by the laser that is emitted in the beam.

Beam quality is the ability to focus the beam to a small spot size with a high energy density.



Fig. 2. Lasers used in laser welding of thermoplastics.

plastic sheet with high speed and precision. However, due to many factors, such as a very limited sheet thickness (less than 0.5 mm), the  $CO_2$  lasers did not become widely used for welding of plastics. The main setbacks of these lasers systems are their large size, especially if they have gas flow systems incorporated, which limits the use of this technology in practical technological processes, and the large wavelength of the  $CO_2$  lasers radiation, which means that the radiation cannot be sharply focused [14].  $CO_2$  lasers are usually applied to thermoplastic laser welding using a butt joint configuration (Fig. 3), which is not ideal for laser welding these type of materials and limits the application in terms of part design.

A major breakthrough in welding of plastics was the development of diode laser sources (wavelength between 780 and 980 nm) in the 1990s, enabling wide use of lasers in industrial applications for welding plastic modules [8]. Diode lasers operate in the Near-InfraRed (NIR) region, where thermoplastics have low intrinsic absorption (Fig. 2), meaning that the use of this type of laser is dependent on the presence of absorbent additives in thermoplastics [8,9]. Without the presence of this additive in plastics, the laser will penetrate the material without being absorbed.

Diode lasers generate radiation using a far more efficient process (30 %) compared to other laser sources and are available in several different options in terms of the radiation wavelength (e.g., 808, 915, 940 and 980 nm). Therefore, they are very adequate for welding a wide range of thermoplastics. However, as a result of some constraint designs (namely the design of the resonator), laser diodes do not possess the ability to efficiently adjust the radiation beam, meaning increased difficult in obtaining a sharp beam focus, and the laser spot size is larger for this laser compared to that of fibre lasers, resulting in inferior beam quality [8,14]. In addition, diode lasers display wider radiation spectrum (lower coherence) and cannot be used efficiently at long working distances [14]. However, these setbacks are largely compensated by the relatively low price and lower operating costs. The beam is usually guided through an optical fibre and often combined with a lens system in a single unit that can be easily coupled to a robot arm. Diode lasers are mainly used with LTW (see below). Many studies using diode lasers in welding of thermoplastics have been reported. Knapp et. al. used a diode laser ( $\lambda =$ 940 nm) to join a composite material made from a PP matrix reinforced with 40% glass fibres and a composite material made from a polyamide (PA) matrix reinforced with 40% glass fibres using a lap shear joint configuration and LTW technique [29]. Devrient et. al. also used fibre guided diode laser with a wavelength of 940 nm to join reinforced PP



Fig. 3. Butt joint configuration in a pipe.

and reinforced polyamide 6 (PA6) [30]. Kagan et. al. successfully applied the LTW technology to several commercial nylon 6 based thermoplastics using high-power diode with 808 nm and has demonstrated that the LTW process is efficient for laser welding of nylon [31]. Juhl et. al. reported laser welding of six common thermoplastics HDPE, PP, PMMA, polystyrene (PS), poly(butylene terephthalate) (PBT), and polvcarbonate (PC) using a Laserline 300 W diode laser (wavelength 808 nm) and lap-joint configuration. They varied laser speed and an optimum weld speed was determined for all 36 material combinations [32]. Other diode lasers using different wavelength were reportedly used by Chen et. al. (semiconductor diode laser with wavelength of 915 nm) [33], Zak et. al. [34] and Chen et. al. [10] (diode laser operating at 940 nm), Mamuschkin, et. al. (conventional diode laser with wavelength of 968 nm) [35], Visco et. al. (diode laser wavelength of 970 nm) [36] and Schkutow et. al. (fibre coupled diode laser of 980 nm) [37]. Zak et al. presented a technique for obtaining an energy flow distribution at the welding interface transversal to the beam scanning direction during laser welding. The technique is based on the measurement of the line width for a sequence of lines scanned on the surface of the polymer by a laser with progressively increasing power (for a given welding speed). The variation of the line width is then interpreted to obtain the distribution of the laser energy at the welding interface. The technique uses the idea of keeping the two parts slightly separated by thin shims in order to avoid their union during welding and to facilitate the quick examination of the joint interface [34].

Another major innovation in plastic welding was the development of Nd:YAG laser sources (1064 nm wavelength) [8]. The Nd:YAG laser is a solid-state laser that has found increasing industry applications in the last decades. Nd:YAG operates in the NIR region and is mainly used in LTW. Normally, the radiation from Nd:YAG lasers is far less readily absorbed by thermoplastics than CO<sub>2</sub> laser radiation. Therefore, the use of this type of lasers also depends on the presence of absorbing additives in the thermoplastic materials [9]. A silica optical fibre is normally used to guide the beam from the laser source to the work piece allowing simple flexible operation with robot manipulation. Nd:YAG lasers using flash lamps have a lower efficiency (3%) when compared to other laser systems, but the rise of diode pumped Nd:YAG lasers has increased both energy efficiency (10%) and beam quality. The superior beam quality enables the production of comparatively small spot sizes. The amount of energy absorption at the Nd:YAG laser wavelength (1064 nm) can be tailored by adding absorbers to the thermoplastic materials [38]. In the absence of absorbers in the thermoplastic, the laser will penetrate deeply into the material, thus preventing its absorption near the joining line and hinder the formation of the welding. This penetration is larger in amorphous polymers than in semi-crystalline polymers. There are available several reports that mention the use of Nd:YAG laser operating at 1064 nm to make welded joints using thermoplastics such as polyethylene, [11], polyamide [12,13] and epoxy resin [39]. Pereira et al. [13] first used a pulsed Nd:YAG laser, instead of the continuous wave laser commonly used, to perform welding of thermoplastics. Pulsed lasers tend to hamper the welding process due to the existence of cooling steps between the pulses, causing heating-cooling cycles in the same place, which can cause damage or defects. However, no welding defects were observed caused by the use of a pulsed laser, demonstrating that

this type of pulsed Nd:YAG laser, developed for welding metals, can be used in the welding of thermoplastics [13].

A recent improvement in laser welding was the introduction of fibre lasers. The laser radiation from fibre lasers (wavelength between 1000 and 2100 nm) can be readily absorbed by polymers, thus avoiding the use of absorbers [40]. Rare-earth doped fibre lasers show improved beam quality when compared to other previous laser sources and are regarded as alternatives for Nd:YAG lasers, with similar beam quality but more efficient. Taking into account the higher efficiency and high beam quality, fibre lasers are applied especially in precision welding, as well as in welding of sheets, films, moulded parts and fabrics [14]. Laser welding using a Thulium fibre laser at 1940 nm was used to join a variety of thermoplastics, such as HDPE [41], PA [42], PP [41] and PC [43], without the use of additives or prior processing, by taking advantage of the laser absorption of polymers at this wavelength. Absorber-free transmission welding of different thermoplastics such as PP [44,45] Acrylonitrile butadiene styrene (ABS) [44], PA [44], HDPE [45], Polymethyl methacrylate (PMMA) [37,45] and Polyoxymethylene (POM) [45] was also reported using thulium fibre laser radiation at the wavelength 2000 nm [44,37,45]. Other fibre lasers have been reported: Amanat et. al. [46] used a pulsed fibre laser with a wavelength of 1060 nm to determine the influence of laser intensity, scan speed, and material morphology on the lap-joint bond strength of poly-ether--ether-ketone (PEEK) joined by LTW. The results showed that the highest joint strengths were achieved using the two lowest welding speeds. The laser intensity had no significant effect on joint strength for the two intensities tested and the semi-crystalline PEEK joints were stronger than the amorphous PEEK joints regardless the welding speed or laser power [46]. Aden et. al. [47] used a fibre coupled diode laser (wavelength of 1530 nm) for LTW studies of PP and PC with carbon black (CB) and indium tin oxide (ITO) as absorbers. At similar concentrations the absorption coefficient of ITO showed to be an order of magnitude smaller than that of CB, resulting in a smaller penetration depth of laser radiation in the case of CB. Tensile shear tests of welded PP parts showed that PP filled with ITO required higher laser power to achieve a similar welding strength as for PP filled with CB [47]. Boglea et. al. [48] used an Erbium fibre laser (1550 nm) and a Thulium fibre laser (1908 nm) for laser welding of PMMA. The results showed that the high laser intensity and the intrinsic absorption of the laser radiation at 1908 nm by the polymer was sufficient to achieve high quality and nearly invisible weld seams at acceptable welding speeds for industrial applications. However, the high laser intensity turned the process more sensible to any variation of the welding conditions [48]. Kurosaki et. al. [49] welded overlapped polymer films of PC, PMMA and Polyvinyl Chloride (PVC) using a Tm:fibre laser (wavelength of 1904 nm) using an innovative laser welding method for thermoplastics assisted by a solid heat sink transparent to the laser beam in order to prevent thermal

Table 2
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Lasers used for welding plastics.

damages to the surface. The results confirmed the non-occurrence of superficial damages in the materials subjected to the welding process due to the use of a heat sink.

Table 2 summarizes the studies performed with the most used lasers for welding plastics.

#### 3. Laser transmission welding (LTW)

The LTW technique was patented in 1987 by Nakamata [92]. This technique is based on the fact that unpigmented polymers are able to transmit the NIR wavelength radiation, but with the addition of an absorber they are able to absorb the laser radiation enabling the local heating and melting of the polymer. The NIR lasers (Nd:YAG and diode lasers) enabled the use of laser welding in a different manner compared to the technique using far IR  $CO_2$  laser wavelengths.

The technique became popular given its simplicity: an upper lasertransparent part and a lower laser-absorbing part are overlapped and clamped together in a manner in which the laser-transparent part faces the laser beam. Clamping pressure is needed to assure proper contact between both parts. The laser energy passes through the upper lasertransparent part and is transmitted to the lower laser-absorbent part, where it is absorbed and is converted to thermal energy. Some of the generated thermal energy is transferred by heat conduction from the lower laser-absorbent part to the upper laser-transparent part at the interface of both parts. The polymer will melt at the interface between the two parts, and ultimately, through molecular diffusion, a weld seam will form after cooling. Fig. 4 illustrates the LTW process. In this process



Fig. 4. Diagram of laser transmission welding, illustrating the movement of the laser over a work piece.

Lasers	Wavelength (nm)	Reference
CO <sub>2</sub>	10,600	[26,25,28,27]
Diode laser	808	[50,31,32,51,52,53,54,55,56,57,58,59,60,61]
	915	[33]
	940	[29, 30, 34, 10, 12, 62, 63, 7, 64, 65, 66, 67, 68, 69, 70, 71, 72, 73, 74, 75, 76, 77, 78, 79]
	970	[35,36]
	975	[80]
	980	[37,44,81,82,83,84]
Nd:YAG	532	[85,86,87]
	1064	[11,39,13,40,62,63,88,89,87]
Fibre laser	1060	[46]
	Yb:fibre laser 1070	[90]
	1530	[47]
	Tm:fibre laser 1904	[49]
	1550	[48]
	Tm:fibre laser 1940	[41,43,42,91]
	2000	[37,44,45]

the excessive laser heating may generate high temperatures at the joint inducing polymer degradation. Therefore, correct knowledge on the welding conditions applied are required, as discussed later.

The materials to be weld also play a role, as its structure influences the transmission of laser wavelength. A neat amorphous thermoplastic has little or no influence on the incident laser beam when used as lasertransparent part. However, semi-crystalline thermoplastics or thermoplastics loaded with reinforcements or certain additives may interfere with the incident laser beam, influencing by this way their transmission [35]. The absorption of laser radiation by the laser-absorbing part can be tailored by adding proper additives (pigments or dye absorbers). The absorber may be included as a discrete film, coating or absorber-rich surface layer at the interface or bulkily incorporated into the laserabsorbing part. It is also possible to adjust the absorber to the laser wavelength being used so that the energy is absorbed more efficiently, enabling the reduction of the amount of absorber needed [93]. One such example, that uses special absorbers, is ClearWeld® [94], in which any plastic material of any colour can be welded. It is only necessary that one side of the joint is transparent to laser radiation. The ClearWeld® process can also be used to weld components with black colour, as long as the laser-facing part is transparent to infrared radiation. There are not many reports in the literature referring to the use of ClearWeld® technology. Amanat et. al. [46] reported a study for determining the influence of laser intensity, scan speed, and material morphology on the lapjoint bond strength of PEEK joined using LTW and Clearweld® as the infrared absorbing medium at the interface. A pulsed fibre with a wavelength of 1060 nm was used. They observed, after Scanning electron microscope (SEM) examination, the presence of bubbles within the weld line, which have negative implications in fatigue strength (by acting as stress concentration points) and hermetic effect of the seal (compromised due to increased porosity of the material at the joint location). They raised the hypothesis that the possible source of the bubbles could be related to residual solvent remaining after Clearweld® application. Liu et al. [57] proposed a new LTW technique using a diode laser with wavelength of 808 nm as light source in which the dye is replaced with metal as the absorber. They welded polyethylene terephthalate (PET) sheet with a thickness of 1.5 mm and reported that the welding strength with a metal absorber with high thermal conductivity was higher and more stable than that with CB absorber, showing that for some cases, dye absorbers could be feasibly replaced with metal absorbers in clear plastic LTW [57].

For a good control of the LTW, the following requirements are needed:

- 1. The Top Layer or laser transparent part (layer that directly receives the laser radiation) must be transparent to the laser radiation, namely in the range 800-1060 nm. Most neat thermoplastic resins can be used in LTW since they transmit all or the most part of NIR radiation within that specific wavelength ranges. The colour of the thermoplastic parts does not affect the welding process, since the laser radiation sources are in the NIR and therefore, out of the visible light range that gives the visual colour sensation. For example, an entirely black or opaque plastic part can still transmit the NIR laser radiation. Some features, such as crystalline moieties, additives and fillers, have influence on the transmission of laser radiation, but they pose few obstacles to the process since only a small amount of transmitted laser radiation is needed to achieve welding. The remaining radiation is subjected to reflection, absorption and scattering. Nevertheless, higher transmission rates will permit a better control of the welding process. The part thickness also plays a major role, as the laser radiation only penetrates a few millimetres within thermoplastics [9]. Table 3 shows the radiation penetration depth of CO<sub>2</sub> lasers and diode lasers in some thermoplastics.
- 2. The laser-absorbing part must have a high absorptivity at NIR wavelength

Table 3

Depth of radiation penetration of  $CO_2$  lasers and diode lasers in some thermoplastics [9].

Material	a [mm] λ = 940 nm	a [mm] λ = 1064 nm	a [mm] λ = 1550 nm	a [mm] λ = 10.6 μm
Amorphous	s thermoplastics			
PC	22.82	23.04	18.94	0.070
PMMA	37.93	36.56	22.10	-
PVC	-	-	-	0.020
Semi-crysta	alline thermoplasti	cs		
LDPE	8.49	10.34	9.71	0.280
PA 6	5.06	5.06	3.01	0.040
PP	11.63	12.87	12.96	0.190

A laser absorbing part is responsible for absorbing the laser radiation that is transmitted by the top layer and convert it into heat. The higher the absorption, the higher the energy that remains at the surface or near bellow where it is necessary to achieve the weld, so melting of the material occurs at the surface or near bellow. Most thermoplastics are transparent to laser NIR radiation; thus they must be filled with absorbing additives [95]. The additive most used in LTW is carbon black soot (CB), since it is very effective and economical [53,52,47,33,12,54]. This filler offers excellent absorbing properties to any thermoplastic material in amounts between 0.2 and 0.4 vol.%.

## 3. The polymers to be joined must be compatible

The two thermoplastics must share some properties, namely chemical structure and melt-temperature of the polymer, in order to achieve good welding [9]. Therefore, similar thermoplastics are preferred as compared to dissimilar ones.

#### 4. Intimate contact between parts through clamping

The heat generated in the laser-absorbing component, must be transferred to the laser-transmission part in order to promote melting in that surface as well. Thus, intimate contact is needed during welding process, so that heat conduction can occur. Therefore, clamping pressure is essential to promote the mixing between the molten surfaces of both parts, enabling stronger joints [9,95].

A modified version of the LTW may be required when welding materials of different optical properties or when black colouring of the absorbing part is not desirable. The technique is a modified version of LTW with an intermediate layer [65]. In this technique, an absorbent layer is placed between the joining parts in the interface zone. This new layer melts completely due to the absorption of the radiation and, due to heat conduction, both joining parts are melted and joined upon cooling. The absorbent layer can be introduced in the form of an absorbing film (intermediate film technique) or injected at the interface during twocomponent injection moulding. Like in regular LTW, one of the joining parts must still be laser-transparent; the other joining part may have any colour. As it is not necessary for one of the parts to have an absorbent pigment, this process can be used to join unpigmented parts [65]. This process was used to successfully weld PA6 and PP using laser radiation. As this technique is strongly influenced by the thickness of the film, the use of films with a thickness greater than 50  $\mu m$  results in a significant reduction in welding resistance [65].

Another modification of the LTW is used for reinforced thermoplastic applications, especially those with carbon fibres. The use of carbon fibre reinforced thermoplastic is increasingly being used in many industrial applications. However, due to absorption properties of carbon fibres, this filler cannot be used to reinforce the upper part when using LTW. LTW using filler material provides a way to join two opaque thermoplastic parts and still continuing to use the laser transmission welding process [12,71]. This joining method using filler material is analogous to conventional LTW, but the joining part facing the incident laser beam does not need to transmit the laser radiation. In fact, both joining parts are absorbent for the applied laser radiation. The filler material that is used to join the two thermoplastic parts plays the role of the laser's transmissive part, and is transparent to laser radiation. The filler material is a monofilament made of the same matrix material of the two thermoplastic parts to be joined. The incident radiation is transmitted through the transmissive filler material and it is subsequently absorbed by the two thermoplastic parts that are to be joined. Like conventional LTW, the generated heat is transferred from the filler material to the joining parts by heat conduction resulting in the melting of the filler material and of both joining parts. After cooling, the two parts and the filler material are joined together due to intermixing of the polymer chains. Fig. 5 illustrate this technique. This process takes time and therefore the welding speeds are significantly lower than the conventional LTW using CB additives [12].

## 4. Absorber free welding technology

The most common procedure used in laser welding, LTW, relies on the use of standard materials. The upper part - transparent part - should be transparent to laser radiation and the lower part - absorbing part should absorb laser radiation. Normally, the lower part has to include an additive, pigment or dye to increase its absorptivity [96]. The most common absorber used is CB, which turns the part black in colour. This combination significantly limits the design possibilities, as at least one of the parts to be welded must be visually non-transparent or black, greatly restricting the areas of application of laser technology. Another issue associated with the use of absorbers that are used in laser welding to sensitize thermoplastics for laser radiation, besides colour or other aesthetic related aspects, is the possibility of chemical reactions of the absorbers with liquid contents in the case of laser welded containers used in certain biomedical applications [97]. With increasing demand for welding clear plastics, new and more reliable methods were pursued. A new welding process became available using higher wavelength lasers (1500–2200 nm) with high power (more than 100 Watts). This type of laser enables the welding of thermoplastics without the use of additional absorbing agents or additives due to the inherent absorption of such wavelengths by thermoplastics. Fig. 6 illustrates the process.

Unlike conventional LTW, there is no need for an absorbing lower layer and either one or both parts need to be semi-transmissive to the laser radiation. The only condition is the large difference in the absorptivity of the radiation wavelength of the working laser between the pair of parts to be welded. The major difference, between conventional LTW and the new absorber-free technology, is that in conventional LTW the laser energy is confined at the joint interface, while in the absorberfree technology the laser energy is spread out. In this way both parts are bulky heated. Therefore, the main issue, when welding optically equal thermoplastics without absorbers, is the heat-affected zone spreading outside of the interface zone and bulkily heating both parts, with some of the laser energy going through the back of the joint [43]. This method was first applied for joining two visually black thermoplastic materials and soon was extended to a large number of combination of pairs, including white materials [8]. As increasingly new laser welding





**Fig. 6.** Progression of radiation intensity in classical laser transmission welding (A) and absorber-free laser transmission welding laser (B).

systems are being developed, new results can be obtained by proper selection of laser radiation with the wavelength at which the welded thermoplastics have the required optical transmittance and absorption of laser radiation [98]. For example, Boglea et. al. [48] analysed the typical absorption peaks for PMMA and selected three lasers emitting in the range between 1550 and 1908 nm (Erbium fibre laser 1550 nm; InP diode laser 1700 nm; Thulium fibre laser 1908 nm) with the aim of determining the general process performance of welding with no additives. Results using 1700 nm diode laser showed weld seams without any signs of thermal damage and with high weld strength tested through tensile tests, while when using 1908 nm a higher laser intensity and the sufficient intrinsic absorption of the laser beam at that wavelength enabled the achievement of high quality and nearly invisible weld seams. Other studies approached the absorber-free laser transmission welding. Mamuschkin et. al. [43] used Thulium fibre laser (1940 nm) for welding PC samples without absorbers. By irradiating the welding path quasi-simultaneously, exploiting in this way the poor heat conductivity of polymers, he studied in detail the influence of the irradiation regime on the weld seam formation and the length of the irradiated contour. His results showed that the energy deposition could be significantly improved when the welding contour length does not exceed a critical length determined by the capability of the welding system. Schkutow et. al. [37] performed welding experiments on extruded PMMA sheets using 2000 nm laser radiation. He found out that the application of this laser radiation could help reducing stress-cracking-susceptibility compared to conventional laser transmission welding with wavelengths of  ${\sim}1\,\mu m$  due to lower thermal gradients between the melt pool and the colder transparent joining partner. The intrinsic absorption properties of the polymer and the use of suitable laser wavelength could allow for a direct heating of the transparent part, helping by this way to reduce these residual stresses [37].

#### 5. Laser welding materials

Laser welding is a widely used technological method to produce high-quality welded joints of thermoplastics materials. The most common is the welding of similar thermoplastics, in which both parts to be welded are of the same material. That provides a better polymer chain intermixing during heating because of the same melting point and chemical structure, which facilitates the welding process. The laser welding of similar thermoplastics tends to show better results in terms of weld quality, originating weld joints that are more stable and stronger [14]. Table 4 summarizes the thermoplastic materials that have already been used in laser welding applications, specifically when both parts to be welded were of the same material. Common plastics and engineering plastics are the ones of great interest and the most reportedly used. Marginal attention is given to the high-performance plastics, given its thermal characteristics and high costs.

Laser welds of dissimilar thermoplastics are produced using the same

#### Table 4

Polymers used in weldings of the same material.

Thermoplastic Material	References
Polyethylene, HDPE, LDPE, UHMWPE	[26,27,36,11,41,45,85,64,32,69,80,99,100,87]
Polymethyl methacrylate (PMMA)	[26,32,37,49,41,45,52,53,55,56,66,101,59]
Polyoxymethylene (POM)	[49,45]
Polypropylene (PP)	[27,29,30,32,33,47,49,41,44,45,65,69,102,103,100,104,105,106,77,78]
Polycarbonate (PC)	$[32,\!10,\!35,\!39,\!49,\!41,\!43,\!44,\!54,\!7,\!69,\!81,\!83,\!101,\!107,\!108,\!100,\!109,\!97,\!60,\!105,\!106,\!106,\!110]$
poly-ether–ether–ketone (PEEK).	[46,61,15]
Polyvinyl chloride (PVC)	[49,50,51]
Nylon (Polyamide 6.6), Polyamide 6 (PA6)	[29, 30, 31, 34, 10, 12, 39, 13, 40, 49, 41, 42, 62, 63, 65, 67, 69, 71, 72, 81, 111, 112, 100, 90, 106, 77, 113, 84, 78]
Polyphenylene sulfide (PPS).	[62,114,115,74]
Polystyrene (PS)	[32,41]
Acrylonitrile butadiene styrene (ABS)	[39,41,44,52,33,6,106]
Poli(L-lactic acid) (PLLA)	[41]
Polyethylene terephthalate (PET), Polybutylene terephthalate (PBT)	[49,44,45,32,57,73,75,77,110,78]
Polyetherimide (PEI)	[68,79]
Polytetrafluoroethylene (PTFE)	[91]

process that are used for similar thermoplastics. However, they pose some challenges due to the following restrictions. First, the two materials to be welded must have similar melting temperatures (semi crystalline polymers) or similar glass transition temperatures (amorphous polymers) and their viscosities must not differ significantly, otherwise the required intermixing of the molten polymers will be hampered [65]. In addition, the polymers to be joined must be chemically compatible, so that intermixing of the molten polymers can be achieved [65]. In the case of incompatible polymers, the molecules are unable to intermix in molten state, originating, instead, separate phases.

There are some examples of laser welding of dissimilar thermoplastics in the literature. They are summarized in Table 5. For example, an experimental investigation of LTW using diode laser, between PMMA and ABS has been reported [52]. The precise adjustment of laser parameters enabled the control of the welding process and the obtaining of good quality welds. Bhattacharya et. al. [101] studied the effect of process parameters, namely, laser power and welding speed, on the formed lap joints obtained by LTW of PC and acrylic and were able to obtain strong welds with tailored weld width by adjustment of the laser processing parameters. Acherjee developed a three-dimensional finite element model of LTW of dissimilar plastics [58]. The model was tested by laser welding PC to ABS with a moving volumetric heat source. The

#### Table 5

Some examples of laser welding of dissimilar thermoplastics.

Thermoplastic materials	Reference
Polycarbonate and Polymethyl methacrylate	[32,88,89,101]
Polymethyl methacrylate and Polypropylene	[32,45]
Polypropylene and Low density polyethylene	[45]
Polymethyl methacrylate and Acrylonitrile butadiene styrene).	[52]
Joining thermosetting composites to thermoplastics	[39,116,76,116]
(Polycarbonate, Polyamide 66, Polyetherimide and	
Acrylonitrile butadiene styrene)	
Polypropylene and High density polyethylene	[28,32]
Polystyrene and High density polyethylene	[32]
Polycarbonate and High density polyethylene	[32]
Polymethyl methacrylate and High density polyethylene	[32]
Polystyrene and Polypropylene	[32]
Polystyrene and Polycarbonate	[32]
Polymethyl methacrylate and Poly(butylene terephthalate)	[32]
Polymethyl methacrylate and Polystyrene	[32]
Poly(butylene terephthalate) and Polycarbonate	[32,117]
Poly(butylene terephthalate) and Polystyrene	[32]
Poly(butylene terephthalate) and High density polyethylene	[32]
Poly(butylene terephthalate) and Polypropylene	[32]
Polyamide 6 and Polypropylene	[65]
Polypropylene and Acrylonitrile butadiene styrene)	[33]
Polymethyl methacrylate and Acrylonitrile butadiene styrene /	[66]
Polycarbonate alloy	
Polyamide 6.6 and Polycarbonate	[81]
Acrylonitrile butadiene styrene and Polycarbonate	[118,58]
Polyamide 66 and Polyvinyl chloride	[82]

results showed that the melt width for both materials at the weld interface is not the same due to differences between the glass transition temperatures of PC and ABS [58].

In some cases, due to large differences in chemical compatibility and melting point between polymers, it is difficult to weld those directly using laser techniques. One way to overcome this problem is to use chemical modification technologies. Liu et al. [82] successfully joined PA66 and PVC using magnetron sputtering technology to deposit a 20  $\mu$ m-thick layer of aluminum film on the PA66 surface. By this way, they increased the surface chemical activity of PA66 and the welding compatibility with PVC. X-ray Photoelectron Spectrometer (XPS) analysis used to detect the chemical bond formation revealed that the increasing of coated PA66 surface free energy and the generation of new Al-Cl chemical bonding were the key factors for the success of the weld [82].

There are some general tables that list the compatibility of dissimilar thermoplastics in laser welding, but they should be viewed with care due to the large variety of thermoplastic resin types. For each type of thermoplastic there can be variations in some properties like melt-flow index, melt temperature or other thermal or chemical properties due to variations in chemical structure or chain length distribution. Table 6 illustrates the compatibility of dissimilar thermoplastics in laser welding.

## 6. Processes used in laser welding of plastics

Various methods for laser welding are available, which differ regarding the relative movement of the laser beam and the welded parts. These methods can be grouped in five main processes: contour, simultaneous, quasi-simultaneous, mask welding and hybrid welding.

In contour welding, a robotic arm is normally used to control de laser motion. The laser radiation beam moves relative to the part making a single passage over the joint (Fig. 7). Contour welding is very flexible and especially suitable to large parts with complex three-dimensional weld geometries [70]. However, it has slower cycle times compared to the other laser welding processes.

In the case where a great number of identical weld seams are needed, simultaneous welding can be used instead. In this case, a group of diode lasers can be assembled in the pattern of the weld seam in order to irradiate and weld the entire joint simultaneously (Fig. 8). This method is mainly used when high volume runs are required because welding times can be shortened. The main drawbacks include expensive tooling targeted to a single application, intensive maintenance and the use of multiple laser sources [8,9,95].

Quasi-simultaneous welding may be viewed as a mixture of simultaneous and contour welding. A single laser beam is guided by special mirrors at very high speeds several times along the part that is to be welded (Fig. 9), gradually heating and melting the entire weld seam at

#### Table 6

Summary of compatibility between thermoplastic resins in laser welding of similar and dissimilar thermoplastic. Adapted from Ref. [9].



Fig. 7. Principle of contour laser welding. Adapted from Ref. [9].

Work piece

the same time [60]. Simultaneous heating results in overall welding process stability, especially in the case where the weld collapse is critical. Quasi-simultaneous welding is the most flexible method, has fast cycle times and is best suited for serial production, mostly for two-dimensional parts [8,9,95]. This variant is used whenever a hermetically sealed welding joint is to be obtained [119].

In the mask welding process, a metallic mask is positioned between the laser and the parts to be welded (Fig. 10). The mask is used so that the laser beam can reach only the part specific areas to be welded through specially designed cut-outs. This process allows production of weld seams with great precision, although the precision and the dimension of the weld seam is greatly influenced by both the quality of the laser beam and the quality of the mask [70]. The process is very flexible and enables the production of different weld seam assemblies on a single part simply by using different masks. The major disadvantage is the need of a specific mask for a specific shape of the weld joint, with the consequent greater cost and lower flexibility. This process is applied mostly in electrical engineering, namely in circuit boards, sensors and

Fig. 8. Principle of simultaneous laser welding with four direct emitting laser sources. Adapted from Ref. [9].

other types of electronic components.

Work piece

Hybrid welding is basically a variant of contour welding with the additional use of high-powered halogen lamps for heating the zone to be welded. The use of pre-heating requires less laser energy to melt the thermoplastic material, resulting in reduced cycle times, increased seam strength as well as reduced part stress due to temperature fluctuations [8,9,95].

The welding process has great influence on the geometry of the weld seam, particularly when laser welding fibre reinforced thermoplastics [62]. Jaeschke, et. al. [62] applied two different strategies, contour and quasi-simultaneous welding, to study the influence of glass fibre reinforcement within the laser transparent part as well as the carbon fibre reinforcement within the laser absorbing part on the weld joint formation of PA6 composites by laser welding. Laser power as well as energy per unit length were varied. They reported that an almost five times higher energy input had to be applied when using quasi-simultaneous welding compared to contour welding, in order to generate the same



Fig. 9. Principle of quasi-simultaneous laser welding. Adapted from Ref. [9].



Fig. 10. Principle of mask laser welding. Adapted from Ref. [9].

weld joint width due to differences in thermal conductivity between carbon fibres and the polymer matrix [62]. Wippo et. al. [67] used contour and quasi-simultaneous welding to demonstrate a new pyrometric-based temperature measurement method to be applied to different LTW techniques. Mamuschkin et. al. [43] performed quasisimultaneous laser welding of PC using fibre laser of 1940 nm to study the influence of the irradiation regime on the seam formation and showed the potential of this technique to obtain a more favourable heat affected zone compared to contour welding. Ghasemi et al. [105] studied the meltdown phenomena during quasi-simultaneous laser welding of PC and PP and correlated the degree of meltdown with different process parameters, namely laser power, welding speed, clamp pressure among others. Acherjee developed a three-dimensional thermal model to study the transient heat transfer phenomena during the quasisimultaneous welding process [60]. The effect of welding parameters on weld pool temperature was also investigated and the model was able to predict the time-dependent temperature history in three-dimensional space, which could be further used to predict the weld seam dimensions and for process optimization.

## 7. Control parameters used in laser welding of plastics

Common to all plastics welding techniques, temperature, time, and pressure are the three most critical process parameters [8]. In laser welding, these parameters are controlled by laser power, welding speed, laser spot size, irradiation time, laser work distance, clamping pressure and type and concentration of laser absorber additive if present. The energy density in laser welding is determined by the laser power, the laser spot size at the joint, and the irradiation time (simultaneous welding), or welding speed (contour welding) [8,9]:

Energy density = Laser Power  $\times$  Irradiation Time/ Laser Spot size

or

## Energy density = Laser Power / (Laser Spot size x Welding Speed)

Since the polymer diffusion process necessary for welding requires a certain period of time, problems will arise if welding speed is too high or the laser power too low. Low energy density means insufficient heating resulting in a weld of lower strength than the required. On the other hand, higher energy density means excess heating, which can degrade the polymer at the joint, resulting in porosity and in a weaker weld [113]. The type and concentration of the absorber included in the laser-absorber part or present at the joint interface is also critical. Higher absorber loads will allow faster weld or lower energy density required. A clamping system is often used to ensure the pressure that needs to be applied in order to achieve a good weld. Insufficient pressure will prevent intimate contact, hampering heat conduction and intermix of molten polymer chains across the joint interface resulting in poor weld seam [95].

Temperature is the factor that most affects the weld joint quality [118,25]. Several studies have demonstrated that the weld seam strength tends to increase with laser energy input until a critical value, after which it starts to decrease. This decreasing in joint strength is mainly due to material decomposition and is therefore strongly dependent on the decomposition temperature of the used thermoplastic. This means that the joint strength can be improved by increasing the laser energy up to the polymer decomposition temperature [118]. Laser energy can be controlled by changing laser power, welding speed or irradiation time [107]. In most cases, the process control variables are tested together in order to determine which set of parameters allows to obtain the best results in terms of weld quality. Several models can be used. Acherjee et al. [59] reported an artificial neural network (ANN) model for laser welding of thermoplastic sheets that was able to establish a relationship between the laser parameters, such as welding speed, laser power and clamp pressure, and joint features such as strength and weld seam length of the joints. The same ANN model was used by Wang et al. [109] in the LTW of PC to predict the relationship between optimum laser parameters and the joint quality, and by Mehrpouya et al. [75] to estimate the weldability of dissimilar PET and PET/Poly(ethylene-vinyl acetate) (PEVA) using a wide range of welding conditions. This model was successfully employed as a prediction tool to estimate the optimum welding conditions based on several laser parameters. Another method commonly used is response surface methodology (RSM). Acherjee et al. [120] carried out experimental investigation and process optimisation of LTW of dissimilar plastics using RSM and flower pollination algorithm (FPA) integrated approach. RSM was used to plan the experiments and to develop the parametric models of the process responses, namely weld strength and weld width. FPA was applied to search for the optimal process parameters combinations that satisfy single objective as well as multiple objectives using the developed RSM models. The second-order equations developed by RSM were able to predict the values of the responses with significant accuracy [120].

In industrial practice, the process parameters (laser power, welding speed and irradiation time) as well the weld seam quality are described by the energy density. The combination of the process parameters determines the welding temperature, which influences the weld seam quality. Lakemeyer et al. [117] designed a set of experimental examinations for quasi-simultaneous LTW to determine whether the energy input or the welding temperature has higher influence on the weld quality. They determined the influence of the energy input on the weld quality in PBT and PC and calculated the welding temperature for every

design point to analyse its influence on the weld strength. A correlation analysis was performed, in order to compare the influences of the two factors, welding temperature and energy input. The analysis showed a higher influence of the welding temperature on the weld strength compared to the energy input. For industrial processes the welding temperature is more suitable for the characterization of optimal process parameters, although the energy input may be used as well [117].

Table 7 summarizes the main process parameters commonly used in laser welding. According to the literature, the most relevant and referred parameters are the laser power, welding speed and clamping pressure. They are described in detail next.

## 7.1. Laser power

The most used form of controlling the weld temperature is through the laser power. Many studies have been reported concerning the effect of laser power on weld/joint quality. Jaeschke et. al. [62] used Nd:YAG and a diode in contour welding experiments on polyphenylene sulfide (PPS) with Carbon Fibres. Among other effects, the average weld seam width was studied by changing the laser power. The weld seam width increased with an increase of laser power, as more matrix material is molten and the weld seam gets wider due to a change of the intensity distribution in the focal point [62]. Acherjee et. al. [52] reported an experimental study on diode LTW of dissimilar thermoplastics in which the effect of the laser welding parameters such as laser power, welding speed, stand-off distance (distance between laser source and parts to be welded) and clamp pressure on weld strength and weld width was investigated using response surface methodology (RSM). They concluded that laser power, among other parameters, has a strong interaction effect on weld strength and weld width, by controlling the heat input to the weld zone and thus the quality of the weld. Quadrini et. al. [64] used a high-power diode laser with a 940 nm wavelength to weld HDPE sheets. Laser-welded joints were fabricated with different values of laser power and welding speed. The results showed a dependency of the laser power and welding speed on the tensile strength of the welded joints, with higher maximum stress achieved by increasing the amount of energy per length unit. However, the surface temperature increased with the irradiation time and laser power and at high values of laser power or irradiation time the samples deformed significantly and started to burn.

## 7.2. Welding speed

Welding speed is another major parameter used to control the weld joint of thermoplastics. Many studies are reported in the literature showing the effect of welding speed on the weld joints. Van de Ven et. al. [50] reported LTW experiments involving PVC that explored the interaction of laser power and welding speed. Welding speed was ranged between 0.04 m/s and 0.07 m/s in combination with laser power (between 16 W and 19 W). They observed that welds obtained at high laser power and low welding speed resulted in decomposition while the welds obtained at low laser power and high welding speed resulted in thin or tapering welds. Schkutowa et. al. [37] studied the influence of the feed

rate on the formation of stress cracks at the edge of the welds created with a 980 nm diode laser in commercially available extruded PMMA sheets. They observed that lower feed rates required much higher laser line energy so that successful weld could be created, since much of the heat was lost due to thermal conduction to the surrounding material. The minimization of thermal losses in surrounding areas, by selecting high feed rates and high laser powers, proved beneficial compared to lower feed rates and lower laser powers [37]. Bhattacharya, et. al. [101] reported the effect of laser power and welding speed, in the LTW of PC and PMMA in a lap joint configuration. Through empirical models using RSM and analysis-of-variance (ANOVA) they correlated the input parameters with responses and checked the significant parameters in the process. They found out that power and welding speed have significant influence in controlling the weld width.

## 7.3. Clamp pressure

The thermoplastic parts to be welded must be in close contact at the weld interface in order to obtain good welded joints. Close contact is crucial for heat conduction from the laser-absorptive part to the lasertransmissive part and intermix of molten material from both parts. Heat conduction is particularly important to ensure that both parts are getting enough energy for melting to occur. The best way to attain close contact is to apply external clamping pressure. Clamping also aids overcoming part tolerances, gaps and warping. Van de Ven et. al. [50] reported several laser transmission welding experiments with PVC exploring the influence of clamping pressure on weld quality. Clamping pressure was ranged between 0.5 MPa and 4 MPa, while keeping other parameters, such as laser power and welding speed constant. They observed that visual consistency of the welds is dependent on the clamping pressure. At levels of lower pressure, a greater percentage of welded samples exhibited a tapering weld. Furthermore, the weld width was found to vary with clamping pressure, with a maximum width found at a clamping pressure of 2.5 MPa.

Acherjee et. al. [55] investigated the effects of process parameters, including clamp pressure, on the lap-shear strength and weld-seam width for LTW of PMMA, using a diode laser system with an wavelength of 809.4 nm. RSM was employed to develop mathematical relationships between the welding process parameters, such as clamping pressure, and the output variables of the weld joint, namely tensile strength and weld-seam width, to determine the welding input parameters that lead to the desired weld quality. They observed that clamping pressure had slight positive effect on the lap-shear strength and contributed positively with statistically insignificant effect on the weld-seam width. This effect was attributed to the much smaller molten area achieved compared to the width of overlap of the plaques, with consequent resistance against the applied pressure as most of the overlapped area is not affected by heat.

## 7.4. Irradiation laser time

Irradiation laser time is another parameter frequently used to control the welding process. Torrisi et. al. [85] studied the welding effect of

#### Table 7

Control parameters used when performing laser welding.

Laser power Welding speed	$[28, 29, 31, 33, 34, 35, 37, 12, 46, 41, 43, 62, 63, 88, 89, 50, 52, 53, 54, 55, 56, 57, 64, 65, 6, 70, 71, 72, 82, 83, 101, 102, 114, 112, 73, 58, 90, 91, 75, 109, 59, 117, 105, 120, 116, 84]\\[28, 29, 31, 33, 37, 12, 46, 49, 41, 43, 62, 63, 88, 89, 50, 52, 53, 54, 55, 56, 66, 67, 70, 71, 72, 82, 83, 101, 102, 114, 112, 73, 58, 90, 91, 75, 59, 109, 117, 105, 120, 116, 84]$
Clamping pressure	[29,31,63,50,52,54,55,57,6,72,83,59,109,105,120]
Irradiation time	[11,85]
Laser beam size	[28,33,41,31,63,55,56]
Additives	[10,35,36,11,40,62,57]
Welding	[43,42,62,65,67,115]
technique	
Thickness	[10,40,56,65,100]
Laser work	[31,52,54,64,59,109,120]
distance	

Ultra High Molecular Weight Polyethylene (UHMWPE) and linear low density polyethylene (LLDPE) polymer sheets with Carbon nanotubes as a function of the laser irradiation time, among other parameters, using pulsed Nd:YAG visible laser. They observed that, for long irradiation times, some joints appeared highly deformed, as a consequence of the too high temperature, and large amount of gas bubble generated at the joint zone. Another example that shows the importance of the irradiation time is given by Visco et. al. [11], in which UHMWPE and UHMWPE/nanocarbon nanocomposites polymeric sheets were irradiated by Nd:YAG laser (1064 nm) with different irradiation times (30 s, 60 s and 90 s). The load-strain curves showed that mechanical strength of the polymeric sheet joint changes with exposure time, the highest value reached after an exposure time of 60 s. The mechanical tests confirmed that the quality of the joint depends of the proper laser exposure time since lower irradiation time produces only slight weld, while higher irradiation time results in material decomposition [11].

## 7.5. Laser spot diameter

The quality of the weld joint can also be controlled through the laser spot diameter. Kagan et. al. [31] reported a study to determine the optimized LTW processing conditions of nylon based thermoplastics. Several LTW parameters were evaluated during the experiments, including laser beam focus (laser beam size/diameter at the font of transmitting plastic) among other parameters. They reported that at optimized processing conditions, the tensile strength of laser welded plastics is close to tensile strength of hot plate welded joints. Chen et. al. [33] used a set of Taguchi experiments to evaluate the characteristics of weld joints, using different welding parameters (laser spot diameter, laser power and welding speed), in terms of molten pool area and shear strength. Mathematical expressions between molten pool area, shear strength and welding parameters were fitted out to determine optimal welding parameters. They observed that shear strength of welding joint can reach up to 42.0 MPa when applying  $0.44 \text{ J/mm}^2$  of laser energy per unit of area. In addition, under high laser energy density, there are several signs indicating thermal decomposition of the polymer due to excessive laser energy per unit area. The overall results showed that weld joints with good mechanical strength can only be obtained with suitable welding parameters; too low or too high laser energy input can result in weld damage.

#### 7.6. Other effects (sample thickness and effect of additives)

In LTW, the welding efficiency depends greatly on laser operating parameters but also on part features such as additives and part thickness. The thickness of the laser-transmissive part has great effect on the energy density and laser beam width that reaches the joint interface [40]. The effect of thickness in laser welding is often evaluated by measurements of laser transmission and absorbance, at the laser wavelength used, through thermoplastic samples or plaques. The laser transmittance depends greatly on the thermoplastic part thickness in the case of semicrystalline materials [40]. Some thermoplastic materials have a lower intrinsic absorbance in the NIR region compared to the IR region. By this way, the use of NIR lasers in welding, as they present less transmission losses due to the lower thermoplastic absorption of NIR radiation, allows the welding of parts with greater thicknesses [40]. Kagan et. al. [40] evaluated the thermoplastic sample thickness and fibre-glass reinforcement of PA and observed that transmission decreases monotonically with increasing thickness of the plastic part. He also observed that laser energy transmission decreases (from 70% to 20%) with increasing fibreglass content from 0 to 63 wt.%. Hopmann et. al. [65] used high-power diode lasers that emit laser radiation at a wavelength of 940 nm to study the effect of sample thickness, among other effects, in the laser welding of dissimilar thermoplastics, namely PA6 loaded with CB (absorbing joining member) and PP. They observed a significant decrease in the range of values for welding parameters, which allow successful welding,

when increasing film thickness. Films with thickness  $d = 50 \mu m$  enabled a maximum welding speed of 290 mm/s, while films with thickness d = 150 µm allowed only a maximum welding speed of 35 mm/s [65]. With increasing film thickness, the amount of material to be melted also increases, requiring more energy for welding. In addition, due to low heat conductivity of thermoplastics, a longer welding time is necessary, resulting in an overall reduction of the process velocity with increasing thickness. Simultaneously, the laser power must be decreased to avoid thermally damaging the material, namely in the surface [65]. Hubeatir [56] performed laser welding of PMMA transparent parts with semiconductor laser (808 nm) using different part thickness and several laser welding speeds. The results revealed that the welding seam width and depth were greatly dependent on the welding speed whereas the transparent part thickness had little effect. Changing thickness had only a slight effect on the transmittance of the transparent part, which was used by the authors to explain the slight effect of part thickness in the results [56].

Besides thickness, the composition of the laser-transmissive part has also a strong effect on the energy density and on the laser beam width at the joint interface [40]. The amount of additives must be appropriately selected to guarantee good welding joints and avoid polymer degradation. Low absorption rate and low laser irradiation time result in weld joints of poor quality, while high laser irradiation time and high absorption rate could permanently degrade the polymer [11]. Kagan et. al. [40] measured the influence of some thermoplastic additives such as fibre-glass, impact modifiers, mineral fillers and colour versions on the NIR laser transmission properties of some nylon based thermoplastics. He reported that for PA6 thermoplastics reinforced with short fibre-glass (GF), the laser energy transmission decreased (from 70% to 20%) with increasing GF load. In addition, he added that mineral fillers (MF) showed increased effectiveness in energy reduction compared to fibreglass (laser transmission was five time less than for GF with the same wt.%). Regarding Impact modifiers, he reported that these additives could reduce laser transmission by 50%. In respect to colorants, the authors stated that plastics of different colours showed some differences in the transmission of the laser energy. Yellow and green colours could reduce the transmission by 75-85%, while red colours transmission are close to natural colour. Carbon black extremely reduces transmission of laser light but non-carbon black colorants allow levels of laser transmission similar to yellow and green colorants. The type of colorant used has great influence on the scattering effect. While organic pigments dissolve, originating homogeneous solutions with the polymer, inorganic pigments do not dissolve, originating agglomerates of small sizes with similar behaviour to mineral fillers, increasing by this way particulate scattering [40]. Other reports concerning the effect of amount of filler in the quality of laser welding were presented by Visco et. al. [11,87]. They checked the mechanical strength of the welded joints of UHMWPE containing different amounts of carbon nanomaterials (0.2%, 0.5%, and 1.0%) using Nd:YAG laser operating at 1064 nm. The results showed that good welding joints could be obtained with 0.2 wt.% filler load and a maximum exposure time of 60 s. Both higher filler loads and higher laser exposure times degraded the mechanical properties of the welding joints due to intense damage made by the laser beam. High amounts of filler (0.5-1.0 wt.%) with only 30 s of laser exposure originated degradation shown by visible holes and burn areas [11]. Table 8 shows some reports of fillers commonly used in laser welding of plastics.

#### 8. Characterization techniques to evaluate materials and joints

Laser welding offers many possibilities for joining thermoplastics that are sensitive to thermal and mechanical stress, through the controlled precision of the welding energy that can be applied locally at the joint interface. To control the welding process, several techniques can be used to evaluate the quality of the welding. These techniques can range from simple observation of the welding joint by using a magnifying glass, to the use of more sophisticated equipment such as

Tal	ble	8
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Examples of thermoplastic fillers used in laser welding.

Fillers	References
Carbon black	[32,33,34,10,35,12,40,47,62,63,52,53,54,55,65,67,70,83,114,90,91,104,74,59,76,106,87,113,79]
Glass fibre	[29,30,33,10,40,62,7,63,69,72,81,115,100,76,79]
Carbon nanoparticles	[36,11,85,57]
Titan oxide	[35,47]
Indium Tin Oxide	[47]
Fe <sub>2</sub> O <sub>3</sub>	[85,86]
ClearWeld	[46]

thermography or SEM analysis. They can also be divided into destructive methods, such as mechanical testing, optical microscopy and SEM and nondestructive methods such as Infrared thermography. In this review, we will briefly discuss the techniques to assess the optical properties of thermoplastics that have influence on the welding process and then focus on the techniques that are used to assess the weld joint after its formation.

## 8.1. Transmission and absorption of laser radiation

Different physical analyses can be employed in order to characterize the properties of the thermoplastics that are relevant to the welding process. Transmitance is a crucial parameter in laser welding, as it controls the energy that actually reaches the interface of the two thermoplastics, where the welding takes place. Other laser-polymer interactions, which may interfere with the amount of radiation that reaches the welding interface, namely reflectance, absorbance and scattering, may be measured. Transmittance is the portion of incident light that is transmitted through the polymer and reflectance is defined as the portion of the incident light that is reflected at the polymer surface or interface. Finally, the absorbance may be defined as the logarithm of the ratio of the incident to transmitted light through the polymer. Light scattering is the process where light is forced to deviate from a straight line due to non-uniformities in the polymer area through which it passes. Normally, the absorption and light scattering of neat amorphous polymers, such as PC, is negligible at the NIR wavelength range of 800-1064 nm [10]. On the other hand, light scattering is significant for semicrystalline polymers such as neat PA6 and neat PP. Many reinforcements or multiple phases present within polymers may also cause light scattering [10]. Polymers are filled with additives in order to absorb specific wavelengths of radiation. When absorbance occurs, the radiation is converted into heat inside the part.

There are several techniques to measure the reflection, transmission and absorption characteristics of polymers, including spectroscopy [40,121,122] infrared thermal imaging [123] and the use of power meters [40,69]. Spectroscopic methods resort to a spectrophotometer equipped with integrating spheres that allow the measurement of the light reflection and light transmission over a wide range of radiation wavelengths. Power meters can also be used to obtain the light transmission and light reflection of a laser source striking a thermoplastic surface. IR thermal imaging is used to measure the surface temperature of the laser-transparent part in the laser entrance and exit zones. Subsequently, the measured temperature can be used to estimate the power flux. Azhikannickal et al. [123] developed a technique for laser reflection measurements based on IR thermal imaging. The reflected energy is absorbed by a black plastic plate and an IR camera is subsequently used to measure the surface temperature of the plate.

Wang et al. [121] used spectroscopy techniques to study the glass fibre content and sample thickness on light scattering and absorbance of several polymers, namely PP, PC and PA6. They observed that thickness had strong effect on transmission of light and that scattering increased with increasing glass fibre content in PA6. The scattering in the wavelength range from 600 nm to 1800 nm increased up to 50 % with increasing glass fibre content from 0 to 50 wt%. Chen et. al. used the Bouguer–Lambert law and an apparent absorption coefficient to describe the total laser energy reduction in unloaded and CB loaded semi-crystalline polymers [10]. Their model was validated by studding the transmittance dependence on part thickness of unreinforced PA6 and PC using a power meter that measured the laser power after going through the polymer part. Results showed that PC with 0.05 wt% CB, PA6 with 0.1 wt% and 0.2 % CB have absorption coefficients of 4.1, 6.0 and 11.3 mm<sup>-1</sup> respectively. Torrisi et. al. [85] measured the polymeric transmission curve of the visible radiation (532 nm) for several thermoplastics as a function of the thickness using a Nd:Yag laser as beam source and a fast Joule-meter as transmitted beam detector. The wavelength was selected to match with the wavelength of the Nd:YAG laser used. The technique is especially useful to compare the transmittance of thermoplastics with different types of additives and serves to determine the amount of laser energy that in fact reaches the interface of the termoplastcis to be joined. Ruotsalainen et. al. [42] measured the absorption, transmission and reflection percentage of 1 mm thick Thermoplastic elastomer (TPE) in the spectral range between 400 and 2500 nm. He used IPG Thulium fibre laser with a wavelength of 1.94  $\mu m$ in welding tests and at this wavelength, the TPE showed an absorption of 29% and a transmission of 65%. The reflection was nearly 8%. Genna et al. [80] developed a simple procedure to estimate the rates of absorbed, transmitted, reflected and scattered laser power irradiated by a diode laser. The transmitted and reflected energies were measured by power meter and by applying the Beer-Lambert law to samples of different thickness respectively. The absorbed ratio was estimated by IR thermal images and the scattered ratio was obtained by simple energy balance, as the difference between the incident energy and the other ratios.

## 8.2. Mechanical tests

Mechanical tests, especially shear strength tests, are widely used to measure the mechanical performance of the weld joints. The two most used geometries in laser transmission welding samples are lap-joint and T-joint. The chosen geometry has repercussions on the used method of testing weld strength: T-joint geometry enables the use of a direct tensile strength test while lap-joint geometry demands a lap-shear test [50]. Other configurations used may be scarf-joint [13] and a different type of lap-joint design [32]. Shear strength of the joints is often evaluated in a typical lap-joint shear strength test that has been used as a reference test for welding strength. Torrisi et al. [85] reported measurements of shear strength of UHMWPE / UHMWPE -Carbon nanotubes joints obtained using different irradiation times in order to determine the best welding conditions of the samples. Visco et. al. [11] used Nd:YAG laser operating at 1064 nm to make UHMWPE joints and also used mechanical tests to check the mechanical strength of the joints welded with different laser exposure times and containing different loads of filler.

#### 8.3. Optical microscopy

Optical microscopy allows inspecting the welding area after the detachment caused by the tensile tests. By proper sample preparation, it can also be used to inspect the welding seam without previous detachment. The laser spot can be observed and be used to evaluate the weld joint as reported by Visco et. al. [11] for UHMWPE samples in which he

associated its homogeneous morphology to perfectly distributed laser energy absorption in all areas of laser spot and its highly irregular shape with great voids in the joints with higher filler amounts. They also used this technique to inspect the heat affected zone (HAZ) of some welded joints reporting several distinct parts including an outer HAZ, a low and damaged inner welded ring (which appears yellow-orange) and a innermost welded zone (which appears dark orange). In this last section, the energy absorbed by the laser caused major damage and visible morphological changes that indicates that optical microscopy could be used to assess laser damage that sometimes is not visible without the aid of a microscope [11]. Hubeatir et. al. [56] used optical microscopy to inspect the weld joint and measure the width and depth of the joints. These measured parameters were then related to welding speed to determine the best operational conditions to achieve good weld joints. Ruotsalainen et. al. [42] used images obtained by optical microscopy to measure the weld width and depth and relate them with laser power.

#### 8.4. Thermographic analysis

IR thermography is among a non-destructive and contact-free method for real-time monitoring of surface temperature measurements. It has been reportedly used in process optimization and weld quality control in laser welding of PMMA and ABS with a diode laser with wavelength 940 nm [66]. As internal defects cause an increase in temperature due to the difference of thermal properties of materials in welds of dissimilar materials or filled/reinforced systems, the IR thermography may be applied in on-line monitoring of the welding process. A recorded temperature profile obtained for optimal welding parameters can be used as a reference and continuously compared to on-line measured IR thermography profile for the purposes of weld joints quality control monitoring in real time [66]. The heat distribution obtained by IR thermography can give detailed information about the behaviour and the influence of different process parameters in laser welding using filler materials. Berger et. al. [12] carried out laser welding experiments by using a diode laser emitting at a wavelength of 940 nm and using filler material as adhesive promoter for different material combinations comprising unmodified PA6 natural, filled with CB, as well as carbon fibre reinforced PA6 natural and containing CB. They reported that, besides material and its optical properties, one of the most important factors influencing the welding process is the thermal properties of the materials, as carbon reinforced and unreinforced polymers have different thermal conductivities. Wippo et. al. [67] used an on-axis temperature detection method by an on-axis pyrometer for the LTW and reported the temperature profiles of different kinds of defects in the weld seam by comparing contour and quasi-simultaneous welding. The temperature measurement was executed by a high-speed pyrometer connected to an optic scanner. The method proved its applicability in detecting small defects in welding. Villar et al[61] determined the temperature distribution at the weld interface along the thickness of the sample during the welding process of PEKK by recording the heat field using IR thermography with a camera sensor perpendicular to the lasersheet and to the welded interface. The device layout enabled the measurement of the temperatures at the welded interface and along the thickness of the specimens.

The effect of different process parameters on the temperature in the joining zone can be investigated by using a scanner-integrated pyrometer. This technique enables not only the development of a non-destructive technique to control the welding process, but also facilitates the part and process design as well as quality check in mass production. Schmailzl et al. [90] used a 3D-scanner with integrated pyrometer to measure the temperature during quasi-simultaneous LTW of PA6 taking into account the spectral filtering of the laser radiation in the semi-transparent emitter upper polymer. Process simulations were performed to compare the temperature field with the measured temperature signal. The temperature signals during welding were found to be in good agreement with the computed mean temperature inside the

detection spot, located in the joining area. They also found that more than 90 percent of the detected heat radiation comes from the joining area [90].

#### 8.5. Physicochemical polymer analysis

The knowledge of thermoplastics thermal transitions and their correlation with the materials' weldability is important for a greater understanding of the welding process. Differential scanning calorimetry (DSC) analysis may be used to study the influence of additives on the calorimetric behaviour of the thermoplastics after being subjected to a welding process. For example, Visco et. al. [11] used Nd:YAG laser operating at 1064 nm to make UHMWPE joints using different amounts of carbon nanotubes additive and showed that a lower percentage of additive (0.2%) corresponded to a higher melting temperature (132.5 °C) after irradiation than before irradiation (131 °C). With a higher percentage of additive load (1%), the calorimetric characteristics deteriorated because the melting temperature dropped from 131.5 °C to 130.83 °C. These results were used to demonstrate that the percentage of carbon nanotube additives influenced the characteristics of the material. A 0.2 % load of carbon nanotubes was sufficient to produce good welding joints, while an increase in the additive load led to a deterioration of the joint properties [11].

Pelsmaeker et. al. [41] used a Thulium fibre laser (1940 nm) to join a variety of thermoplastics, including ABS copolymer, HDPE, PA6 and PC, and subsequently used exclusion chromatography to analyse mixtures of welded material and the underlying substrate. They showed that the temperature at the welding interface did not exceed the degradation temperature as no significant change in molecular weight or dispersivity was observed for those samples. Furthermore, Pelsmaeker et. al. [41] used DSC and Thermogravimetric analysis (TGA) to measure thermal transitions in order to better understand the welding process and to determine their correlation with the materials' weldability. They also compared the weldability of the samples with the TGA data and reported that polymers with aromatic backbones are not suitable for welding applications as degradation was favoured over welding, due to the intrinsic higher chain rigidity in aromatic polymers that opposes the formation of entanglements. DSC can also be used to measure the degree of crystallinity as crystalline domains, like chain rigidity, may oppose the efficient welding of two thermoplastic parts [41]. The reported results showed that the successfully welded polymers are aliphatic and are amorphous or have a low degree of crystallinity with glass transition temperature higher than 50 °C [41].

Herthoge et al. [91] performed DSC measurements on material harvested from the welding pattern after welding and failure to examine degradation and/or the existence of newly formed compounds after welding experiments on PTFE samples. DSC measurements were used to give an estimation of the average molecular weight ( $M_n$ ). Although the results should be interpreted qualitatively, as pointed out by the authors, they showed a decrease of more than 30 % on  $M_n$  compared to that of the unwelded material, clearly indicating that degradation has effectively occurred in the weld joint.

#### 8.6. SEM

Torrisi et. al. [85] used mass quadrupole spectrometry (MQS) and SEM, among other techniques, in order to evaluate the mechanical resistance of weld joints of UHMWPE made using Nd:YAG laser, operating at 532 nm. The MQS spectroscopy was used to detect the gas species released from the polymer materials during laser irradiation. The SEM images were used to show that observed ring areas were due to hydrogen gas emission, released to the joint interface zone, which generated an interface pressure that subsequently caused the detachment of the faces at the centre of the hot polymeric weld joint. In addition, the microscopic analysis of the surface changes of the welding zone in the two separated parts after tensile tests enabled to confirm the intimate melting and intermixing of the two polymer surfaces [85]. Amanat et. al. [46] also used SEM to perform post-failure characterisation of the bond interface of PEEK joints and reported the presence of bubbles within the weld seam, which have negative impacts regarding the fatigue strength of the seal. The size of the bubbles was associated to the welding speed, with higher speeds resulting in large and superficial bubbles, and lower speeds resulting in widespread fine bubbles. These authors also used SEM images to distinguish between interfacial and substrate failure after tensile tests, by signalling the presence of pores that are plastically deformed in the direction of applied stress [46]. Visco et. al. [36] used SEM images to show how the typical smooth surface of the nanocomposites become irregular and rough with fragmentations and holes characteristic of and intense melting process due to laser radiation absorption. In the SEM images, several zones were easily identified, such as HAZ, where the highest changes in surface roughness are observed due to the contact with the laser beam, a deep hole and a groove in the contact point of the laser beam with the polymer and the presence of ablated-melted material redistributed in the neighbouring area. [36]. Table 9 shows some of the techniques that are reported in the literature to assess and monitor the welding process.

#### 9. Conclusions

The increasing replacement of metals or other materials with polymers in many applications has demanded the development of new techniques for joining thermoplastics. Many techniques have been developed over the years, but laser welding has been gaining a prominent position and hence, has increasingly becoming an area of interest for research and development. Laser welding of thermoplastics is a highly specific technique for joining plastic parts in applications that require high-speed welding and is especially adequate for mechanical fragile components and modules demanding sterile conditions. The laser welding provides several technological and practical advantages in comparison with other welding techniques. These advantages include ease of joint fabrication, higher joining quality, minimal resulting flash, lower stress to the thermoplastic parts, the ability to weld complex shapes, all this combined with advantages in terms of costs. Such advantages make laser plastic welding a prominent technique for many joining applications, specifically, within the medical, packaging, automotive, hermetic containers and consumer electronics industries.

This paper reviewed several aspects of the laser welding of thermoplastics focusing mainly in lasers, materials, process parameters and quality monitoring techniques.

Several types of laser sources available for welding of thermoplastics, including Nd:YAG, diode and fibre lasers, were discussed. They can be selected based on the operating wavelength that is more appropriate for

LTW of polymers and their composites. In the most common procedure, thermoplastic components can be welded by transmission of a laser beam through a laser-transparent part and by subsequent heat generation at the interface between transparent and absorbent parts. Additives such as CB or other colourless infrared absorbents are normally used as the method to generate heat and localised melting. Many reported works demonstrate the successful application of this technique in a wide range of situations and materials. The process only affects the area very close to the joint and the welds produced are aesthetically attractive.

The advent of fibre lasers in the range of 1500–2200 nm has allowed the joining of thermoplastics dispensing the use of absorber additives. In this absorber-free LTW technique, as both polymer parts absorb laser energy in the range of the laser used (1500–2200 nm) the radiation penetrates more deeply in both parts to be welded, compared to conventional LTW with absorbers, originating a deeper heat-affected zone at the weld interface.

Many studies were reported addressing the use of different laser systems to weld different thermoplastics and combinations of dissimilar thermoplastics. The welding of similar thermoplastics provides a better polymer chain intermixing during heating facilitating the welding process. Laser welding of dissimilar thermoplastics uses the same basic principle, however, the two materials to be welded must have similar viscosities and melting temperatures, otherwise the required intermixing of the molten polymers will be hampered.

The impact of welding technique (laser beam delivery: contour, simultaneous, quasi-simultaneous and masked), the process parameters and other welding conditions on the weld quality of the joints has been addressed. Depending on the purpose, the right welding technique is chosen. Optimised laser parameters like laser power, welding speed, stand-off distance and clamp pressure are normally selected based on modelling and simulation software. The proper selection of process parameters can improve the weld seam mechanical strength and overall quality.

Several techniques used to characterize the quality of the welds were discussed. These techniques ranged from destructive methods, such as mechanical testing, optical microscopy and SEM, to nondestructive methods such as IR thermography. Several techniques are used in prewelding stages, such as measurement of the absorption/transmittance of radiation by polymers. Others may be used during real-time, such some tomography techniques, while others are used to assess the laser welding process at a post-welding stage to check the weld quality. Weld evaluation is a valuable aid to the part design stage, which, when combined with the proper process monitoring, can be effectively used to reduce the weld failings, enhance the overall weld quality and reduce the production costs.

#### Table 9

Techniques used in characterization of the laser welded materials and joints

Technique	References
Material character	rization (prior to welding)
Absorption or	[10,35,40,47,49,41,44,45,85,63,56,7,6,61,69,80,83,90,97,106,87,77,84]
transmission of	
laser radiation	ntion
Optical	aluun 190 22 26 11 12 30 12 42 42 45 55 62 99 00 52 52 54 55 56 57 7 66 91 92 101 59 01 07 104 15 120 76 116 97 112 94 79 701
microscopy	[27,33,30,11,12,37,13,43,42,43,03,03,03,03,27,32,33,34,37,300,01,02,101,305,71,77,104,13,120,70,110,07,113,04,70,73]
Scanning	[36,85,29,46,63,57,59]
electronic	
microscopy	
(SEM)	
Mechanical tests	[26,28,29,31,32,33,35,36,11,12,39,13,46,47,49,41,43,42,45,85,62,63,88,89,50,51,52,53,54,55,64,65,66,6,68,71,81,82,91,104,74,59,109,120,116,87,113,78]
(tensile tests)	
Differential	[32,11,41,91]
scanning	
calorimetry	
(DSC)	[61 66 67 102 00 74 76]
Thermography	[01,00,07,102,90,74,70]

## **Declaration of Competing Interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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