Wood Bonding by Mechanically-Induced in Situ Welding of Polymeric Structural Wood Constituents

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ABSTRACT: Mechanically induced wood fusion welding, without any adhesive, is shown here to yield rapidly bonding wood joints satisfying the relevant requirements for structural application. The mechanism of mechanically induced vibrational wood fusion welding is shown to be due mostly to the melting and flowing of amorphous cells-interconnecting polymer material in the structure of wood, mainly lignin, but also some hemicelluloses. This causes a partial detachment, the "ungluing," of long wood cells and wood fibers and the formation of an entanglement network drowned in a matrix of melted material which then solidifies, thus forming a wood cell/fiber entanglement network composite with a molten lignin polymer matrix. During the welding period some of the detached wood fibers which are no longer being held by the

INTRODUCTION

Assembly technologies with mechanical connectors or adhesives are common in joining solid wood in the furniture, civil engineering, and joinery industries. Both kinds of connections demonstrate several problems. With mechanical metal connectors rust stains may appear on the connectors and corrosion of the connector can and does occur. With adhesively bonded joints, liquid adhesives cause higher costs with regard to the upkeep and maintenance of the manufacturing chain. With adhesives the process is relatively longer unless large investments in adhesive materials and machinery (high-frequency or microwave systems) are made to speed up the hardening phase. Thermoplastic welding technologies, which are widespread in the plastic and car industries, have recently been applied also to wood joining by melting a thermoplastic polymer between the two wood surfaces to be joined. A variety of techniques such as ultrasound and mechanical friction^{1–5} have been used to melt the thermoplastic polymer in situ.

interconnecting material are pushed out of the joint as excess fiber. Crosslinking chemical reactions of lignin and carbohydrate-derived furfural also occur. Their presence has been identified by CP-MAS ¹³C-NMR. These reactions, however, are relatively minor contributors during the very short welding period. Their contribution increases after welding has finished, which explains why relatively longer holding times under pressure after the end of welding contribute strongly to obtaining a good bond. © 2004 Wiley Periodicals, Inc. J Appl Polym Sci 92: 243-251, 2004

Key words: wood welding; wood fusion; polymer melting; adhesives; lignin; joints

This paper, however, deals with the chance finding that the application of the same mechanical techniques at the interface of two solid wood surfaces in the absence of any thermoplastic material, or any other binder, yields joints of considerable strength. It deals with the optimization of the technology to achieve welded wood joints of structural strength level by welding at a very rapid rate. It deals also with the limits of the present technique and with the determination of the causes of the mechanically induced fusion of wood in the absence of any adhesive or welding thermoplastic.

Since the term "wood welding" is already used to define the use of a melted themoplastic resin as the binder of two wood surfaces, this paper refers to the novel process described here as mechanically induced wood fusion for lack of a better definition to distinguish it from traditional wood welding.

EXPERIMENTAL

The mechanical welding machine used was a Branson welding machine, Type 2700, 100 Hz, normally used to vibrationally weld metal.

Preparation of joints by mechanically induced wood fusion welding and their testing results

Specimens composed of two pieces of beech wood, each of dimensions $150 \times 20 \times 15$ mm, were welded

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together to form a bonded joint of $150 \times 20 \times 30$ mm by exercising a vibrational movement of one wood surface against the other at a frequency of 100 Hz. When the fusion state was reached on the joint interface, the vibration process was stopped. The clamping pressure was then briefly maintained until the bond solidified. The welded samples were conditioned for 1 week in a climatic chamber (20°C and 65% MC) before testing.

The parameters which were varied in the various experiments were the welding time (W.T. = 2 to 5 s); the contact holding time mantained after the welding vibration stopped (H.T. = 2 to5 s); the welding pressure exercised on the surfaces (W.P. = 0.4 to 2.3 MPa); the holding pressure exercised on the surfaces after the welding vibration stopped (H.P. = 1.33 to 2.7 MPa); and the amplitude of the shift imparted to one surface relative to the other during vibrational welding (A. = 1 to 3 mm). The frequency of welding was maintained at 100 Hz since initial experiments at higher frequencies (240 Hz) showed a too-high rise of the bond line temperature; two species of wood, beech wood (Fagus sylvatica), a hardwood, and Norway spruce (*Picea abies*), a softwood, were tried. The equilibrium moisture content of the samples was 12%. Water spray was used where indicated to evaluate whether it rendered bonding easier.

Scanning electron microscopy of joints obtained by mechanically induced wood fusion welding

Scanning electron microscopy (SEM) of (1) the surface of the open joints after mechanical testing, (2) the surface of the joints after one of the wood surfaces had been microtomed away, (3) the side of still closed joints, and (4) the waste fiber that accumulated on the surface of the joints during welding was analyzed after metallizing with gold-palladium using a Hitachi *S*-520 at magnifications of 300 : 1, 1000 : 1, 2500 : 1, and 7000 : 1.

Solid state ¹³C-NMR of joints obtained by mechanically induced wood fusion welding

The surfaces of the welded joints obtained were analyzed by solid state CP MAS ¹³C-NMR: (1) The surface of the open joints after mechanical testing, (2) the surface of the joints after one of the wood surfaces had been microtomed away, and (3) the side of still closed joints were tested. Furthermore, (4) the waste fiber that accumulated on the surface of the joints during welding was also analyzed. Spectra were obtained on a Bruker MSL 300 FT-NMR spectrometer. We obtained the solid state CP-MAS ¹³C-NMR spectra of the bonded interface reduced to powder by sanding of the control beech wood powdered by either sanding or grinding and that of the ground excess fiber waste



Figure 1 Schematic indication of linear oscillating friction of two solid wood surfaces during wood fusion welding.

coming out at the joints sides at a frequency of 75.47 MHz and at a sample spin of 4.0 kHz. The impulse duration at 90° was of 4.2 μ s, contact time was 1 ms, the number of transients was about 1000, and the decoupling field was 59.5 kHz. Chemical shifts were calculated relative to TMS for NMR control. The spectra were accurate to 1 ppm.

Thermomechanical analysis (TMA) of joints obtained by mechanically induced wood fusion welding

The wood fusion joints obtained were cut progressively into joints of dimensions suitable for testing by thermomechanical analysis. Thus, samples of $21 \times 6 \times 1.2$ mm were obtained.

Triplicate samples of these beech wood joints for a total sample of $21 \times 6 \times 1.2$ mm were tested in nonisothermal mode between 40 and 220°C at a heating rate of 10°C/min with a Mettler 40 TMA apparatus in three points bending on a span of 18 mm exercising a force cycle of 0.1/0.5N on the specimens with each force cycle of 12 s (6 s/6 s). The classical mechanics relation between force and deflection, $E = [L^3/(4bh^3)][\Delta F/(\Delta f)]$ allows the calculation of Young's modulus *E* for each case tested. Since the deflections Δf obtained were proven to be constant and reproducible,^{6,7} and these are inversely related to the values of the modulus, the values reported in the tables are often the values of the deflection (in micrometers).

RESULTS AND DISCUSSION

The equipment used for frictional welding of wood is the same type of equipment used for frictional welding of metal. Figure 1 shows the characteristic frictional movement imparted by an industrial mechanical metal welding machine as well the frictional shift and force applied to the two pieces of wood during welding. The applied welding results were obtained by varying some of the parameters that influence welding of metal with the same type of equipment. Thus, the influence on the final bond of the vibration welding time, the contact holding time after the welding vibration stopped, the welding pressure exercised

TABLE IParameters Used for the Optimization of MechanicallyInduced Wood Fusion Welding: Variations of VibrationAmplitude, Welding Time, and Welding Pressure andResults Obtained by Test in Tensile of Bonded Joints

	Welding	Welding	Joint tensile
Amplitude	time	pressure	strength
(mm)	(s)	(MPa)	(MPa)
Beech wood			
1	3	0.4	_
1	4	0.4	
1	5	0.4	
3	3	0.4	_
3	4	0.4	1.43
3	5	0.4	3.48
1	3	1.3	_
1	4	1.3	_
1	5	1.3	_
3	3	1.3	5.43
3	4	1.3	6.96
3	5	1.3	6.74
1	3	2.3	_
1	4	2.3	
1	5	2.3	
3	3	2.3	5.86
3	4	2.3	7.61
3	5	2.3	5.37
Spruce wood			
1	5	2.3	0.5
3	3	0.8	1.5
3	4	0.8	2.0
3	5	0.8	1.7

on the surfaces, the holding pressure after the welding vibration stopped, and the amplitude of the shift imparted to a surface relative to the other during vibrational welding was tested. The frequency of welding was maintained at 100 Hz as initial experiments (not reported here) at higher frequencies (240 Hz) showed a too-high rise of the bond line temperature and lower bond strength due to excessive degradation of some of the materials at the interface. Two different species of wood were compared, beech wood (a hardwood) and Norway spruce (a softwood). The majority of the experiments were done with beech wood because the bonding results on Norway spruce were less encouraging. This was due to the bonding interference caused by cell wall collapse that is such a characteristic and well-known problem in this wood species.

Bonding results

The initial bonding experiments reported in Table I compare the influence on bonding strength of welding time, welding pressure, and vibration amplitude. The results in Table I indicate the following:

1. Joint tensile strength depends on vibration amplitude, with the joint presenting no bonding strength at all for 1-mm vibrational amplitude and some good bonding results for -mm vibrational amplitude.

- 2. Joint tensile strength depends on welding pressure: the higher the welding pressure in the range used, the higher the bond strength of the joint. The only exceptions to this are the last cases for beech wood at 1.3 and 2.3 MPa in Table I, where the joint strength starts to decrease for the pressure used. This is due to temperature-induced incipient degradation of the bond.
- 3. Joint tensile strength depends on welding time. This dependence, however, appears to be less intense than that of welding pressure. At each pressure, while bond strength in general increases with welding time, the increase is not too marked, with good results obtained at 3-, 4-, and 5-s welding times, at partity of the other conditions.
- 4. While it appears from Table I that softwoods can be frictionally welded, the problem of cell wall collapse characteristic of the softwood species chosen renders it impossible to arrive at a definitive conclusion regarding the potential of softwood welding.

Furthermore, the initial experiments reported in Table I indicate that wood welding is potentially very promising, with the best average reported result of 7.61 MPa not very far from the 10.0-MPa strength value for a structural wood joint required by the relevant European Norm EN 205-D1.⁸

The series of experiments and results reported in Tables II and III shows the results obtained using the combination of different welding times, holding times after the end of vibration welding, and holding pressures after the end of welding. The influence of the presence or absence of water sprays on the wood surfaces was also tested. The influence of some of these parameters appeared to be determinant in obtaining joint strengths comparable to and higher than the requirements of the relevant standard. Thus, the best results were obtained at the shortest welding time (3 s) used but at a longer holding time (5 s). In this case the results are much better than just relying, as in Table I, on a longer welding time (4 s) but a much shorter holding time (2 s). Two of the average results reported in Table III actually satisfy the requirements of the relevant EN 205-D1 specification, while at least three other sets of conditions yield results very close to the requirements of the standard. Increasing welding times decreased joint bond strength under the range of conditions used. The higher of the two welding pressures, at 2 MPa, yielded the best results. Higher welding pressures than this, such as 2.3 MPa in Table I, yielded poorer results due to incipient bond degradation. The presence or absence of water sprays did not appear to affect the result. Initially, water spray were

		1		5		0
Series	Welding time (s)	Welding pressure (MPa)	Holding time (s)	Holding pressure (MPa)	Water spray (g/mm ²)	Number of specimens tested
3a	3	1.3	2	1.3	No	10
3b	3	1.3	2	2	No	10
3c	3	1.3	5	1.3	No	10
3d	3	1.3	5	2	No	10
3e	3	1.3	5	2	Yes	10
4a	4	1.3	2	1.3	No	10
4b	4	1.3	2	2	No	10
4c	4	1.3	5	1.3	No	10
4d	4	1.3	5	2	No	10
4e	4	1.3	5	2	Yes	10
5a	5	1.3	2	1.3	No	10
5b	5	1.3	2	2	No	10
5c	5	1.3	5	1.3	No	10
5d	5	1.3	5	2	No	10
5e	5	1.3	5	2	Yes	10

 TABLE II

 Parameters Used for the Optimization of Mechanically Induced Wood Fusion Welding

tried to see whether increased moisture content would further favor lignin flow. This did not appear to be the case, with the 12% equilibrium moisture content appearing to be more than sufficient. This can be understood from the diagram of temperature increase as a function of welding and holding time shown in Figure 2. The temperature on the glue line reaches 170°C during welding: no increased moisture content will be maintained in such a hot interface, and as such water sprays have no effects. It must be noted that while on the bondline the maximum temperature is 170°C, in the wood just 1 mm away from the bondline the temperature is equal to ambient temperature(20–

TABLE III Results of Tensile Strength Tests According to European

Norm EN 204 of Mechanically Induced Wood Fusion Welding Specimen Cases Outlined in Table I

Series	Tensile strength (MPa)	SD^{a}	SD (%) ^b	SEc
3a	3.64 ± 1.3	1.7	47.9	0.6
3b	2.26 ± 1.8	2.2	98.8	0.8
3c	9.40 ± 1.2	1.3	14.0	0.5
3d	10.45 ± 0.9	1.1	10.2	0.4
3e	10.37 ± 1.0	1.3	12.8	0.4
4a	1.18 ± 0.4	0.4	37.4	0.2
4b	0.86 ± 0.2	0.3	32.2	0.1
4c	8.78 ± 0.8	1.1	12.0	0.4
4d	6.41 ± 1.2	1.7	25.8	0.6
4e	8.47 ± 0.8	1.1	12.6	0.4
5a	0.82 ± 0.3	0.3	41.1	0.1
5b	0.51 ± 0.2	0.3	51.1	0.1
5c	7.54 ± 0.8	0.9	12.4	0.3
5d	4.55 ± 1.3	1.7	36.9	0.6
5e	4.44 ± 1.2	1.5	34.6	0.5

^a SD, standard deviation.

^b SD%, standard deviation %.

^c SE, standard error (standard deviation from the mean).

25°C). This maintenance of a remarkably low temperature in the body of the joint explains why the high temperature reached (exclusively at the interface) does not distort the joint, as has been observed.

Results not reported here have shown that the strong joints obtained are not capable of satisfying any relevant specification for exterior joints since they present very poor resistance to water.⁹ These joints can then be only considered for interior applications, such as for furniture and interior grade joinery.

Once it is established that mechanically induced wood fusion welding can and does yield bonding results satisfying the relevant standard, the question that remains to be answered is "why"? A series of analysis of the joints by SEM, solid state ¹³C-NMR, FT-IR, and TMA was carried out to understand what occurred.

Figure 3 reports the CP-MAS ¹³C-NMR spectrum of the wood of the bondline (less than 0.5 mm on each side of the bondline) of a wood fusion-welded beech



Figure 2 Bondline temperature increase during wood fusion welding time and holding time.



Figure 3 Solid state CP-MAS ¹³C-NMR spectrum of beech wood material in the bondline. Sample powdered by fine sanding.

joint welded for 3 s and held for 5 s. The sample was obtained by fine sanding. This spectrum did not show any major differences in its carbohydrate region in relation to the control samples. The spectra shown in Figure 3 show that not too many differences regarding the situation of the wood carbohydrates appear to exist in relation to untreated wood. The only noticeable difference regarding the bondline sample in relation to the controls is a slightly higher proportion of amorphous carbohydrates in the former: the 83.29ppm shoulder is more intense than in the controls. The main differences among the four spectra are, however, in the smaller signals in the region of 120-175 ppm, characteristic of some of (but not only) the signals of lignin. To facilitate observation of the differences in this region of the spectra their details are compared in Figure 4. In Figure 4 one can see a main peak at 153.4/153.9/154.1 ppm characteristic of the carbon of an aromatic ring to which is bound a —O—, be it an -OH or an -OCH₃; the main lignin aromatic carbon to which a $-OCH_3$ methoxyl group is bound is noticeable at 161.2 ppm. The absence of this latter peak in the excess fiber and in the sanded beech control indicates that lignin in the glue line does not appear to be too heavily demethoxylated, while the fibers expulsed during welding contain heavily demethoxylated lignin. The number of peaks in this region represent aromatic ArC-O- presenting different substitution patterns of the aromatic ring. Of particular interest is that in the samples from the welded bondline and from the welding excess fibers there are pronounced peaks at

149.1 and 148.2 ppm, which are almost completely absent in the sanded beech control and are very small in the ground beech control (Figure 4). Of these the 149.1-ppm peak corresponds to an aromatic carbon bound to a nonaromatic carbon, namely (Ar)C-C or other than the normal bonds of this type present in lignin or of the same type present in lignin but in considerable excess of what is normally found. This indicates that a substituent has linked onto the aromatic ring, indicating that a reaction on the aromatic ring of lignin has occurred. This is also shown by the number of peaks visible in this crowded region: one passes from 7 to 8 visible in the sanded beech control to the 10 present in the sanded glue line sample and on the excess fibers (Figure 4). This increase in the number of peaks could just be due to part of the lignin being demethoxylated. Most interesting is the peak at 148.2 ppm characteristic of the carbons of a furan ring¹⁰ (Figure 4). Furfural, methyl furfural and other furanic materials are well known to be obtained from wood carbohydrates,¹¹ possibly the hemicelluloses, by thermal treatment. The high temperature reached during welding (Figure 2) indicates that the formation of furfural in the glue line is quite likely and the presence of its 148.2-ppm peak confirms its presence both in the bondline and in the excess fibers. Its slightly higher proportion on the excess fiber (Figure 4) is logical if one thinks that this is the material which has undergone the most marked mechanical treament. It is not present at all in the sanded beech control, and it is present to a very much smaller extent in the ground



Figure 4 Comparison of detail of 120- to 175-ppm regions of CP-MAS ¹³C-NMR spectra of finely sanded and finely ground control beech wood samples, of finely sanded beech wood material in the bondline, and of finely ground excess fibers issued during welding from the bondline.

beech control (Figure 4). However, the lack of a signal of an aldehyde function indicates that it is this material that may have self-polymerized as well as possibly having reacted on sites of the aromatic ring of lignin. The clear –COO- acetic acid peaks at 173.5 and 173.9 ppm belonging to beech hemicelluloses, which can be observed in the two controls, decrease markedly in bondline and excess fiber samples and resolve in a series of smaller peaks, some of them with shifts characteristic of levulinic acid, obtained with furfural from the same type of reaction.¹¹ This indicates that welding has heavily deacetylated the hemicelluloses and that it is this free acid that has helped to catalyze at higher temperature the formation of furanic compounds during wood welding.

In short, the results of the NMR analysis indicate the following:

 —A certain amount of lignin demethoxylation has occurred during welding;



Figure 5 Comparison of NMR proton mobility spectra for the materials in Figures 6 and 7.

- The proportion of amorphous carbohydrates appears to slightly increase during welding;
- —A reaction of autocondensation of lignin on its aromatic ring appears to have occurred;
- —Furfural appears to have been produced during welding and to have self-polymerized or reacted with lignin aromatic nuclei, or both;
- —Some deacetylation of hemicelluloses appears to have occurred during welding, with the free acetic acid possibly contributing to the occurrence of the preceeding reaction in this list.

Notwithstanding the indication that some self-condensation and/or crosslinking reactions involving lignin and furfural clearly seem to occur, their limited extent does not appear to justify joint bond strengths in excess of 10 MPa as obtained in wood fusion welding (Table III). These reactions are surely a contributory factor, but they are unlikely to be the main cause of the bonding results obtained in mechanically induced wood fusion welding.

A first indication that the main cause of wood fusion welding might be a physicochemical effect rather than just a crosslinking chemical reaction comes from the proton mobility NMR spectra of the four main samples shown in Figure 5. These shows weak interactions of water with wood in three of the samples with the intensity of the interaction increasing as the peak becomes less sharp, hence passing from the sanded beech control to ground beech control and to the bondline. The interaction of water with the excess fibers has been considerably stronger as shown by the much broader proton peak in Figure 5. The higher the water interaction the lower the T_g of hemicelluloses and lignin¹² and the easier it is to melt these two materials or for these two materials to flow.^{12,13} This means that, particularly in the excess fibers and to a lesser extent in the bondline, melting of hemicelluloses and lignin appears to have taken place during welding, an occurrence that has clearly not taken place in the two controls.

If melting of some of some of the major structural, polymeric wood constituents occurs, this should be observed by SEM. Figure 6a shows one of the characteristic bands on the surface of fusion welded wood. One can see fibers, long wood cells, or tracheids immersed in a mass of molten polymer. Since the cells do not appear to be greatly damaged this means that melting has occurred mainly in the intercellular connecting tissue or *middle lamella*. Wood middle lamella is particularly rich in lignin, more so than any other of the wood's anatomical features. A more magnified detail in Figure 6b of the same type of surface confirms the presence of a mass of entangled long wood cells immersed in a matrix of amorphous, fused intercellu-



Figure 6 Scanning electron microscopy images of a lignin fusion band with cellulose fibers and wood cells (tracheids) immersed in it obtained during welding at (a) $25 \times$ magnification showing the general appearance of the band and (b) detail of the same at $100 \times$ magnification showing the entangled and detached tracheids, a fused intercellular lignin mass, and tracheids and fibers immersed in the fused lignin matrix.

lar material, almost certainly composed of lignin, possibly including also some hemicelluloses. Figures 7 a and b, which show detail of the excess fibers expelled from the bondline during welding, confirm this. In Figures 7a and b the fiber is clearly an entire, undamaged, long wood cell and the material that is seen on its surface in patches is definetely part of damaged, melted cells-interconnecting material, hence mainly lignin from the wood middle lamella.

The SEM investigation then confirms visually what was partly inferred by the CP-MAS ¹³C-NMR spectra. The mechanism of mechanically induced vibrational wood fusion welding is due mostly to the melting and flowing of amorphous cells–interconnecting polymer



Figure 7 Scanning electron microscopy images of filaments of detached wood cells (tracheids) from the excess fibers issued during welding from the bondline at (a) $100 \times$ magnification and of (b) a single wood cell detail at $800 \times$ magnification. Note the residual fused amorphous residual material clinging to the tracheid's surface which is the residue of the intercellular bonding material holding wood cells together.



Figure 8 An example of the curve of the increase of the modulus of elasticity (MOE) as a function of time and temperature obtained by thermomechanical analysis, at a constant heating rate of 10°C/min, of a beech wood joint obtained by mechanically induced wood fusion welding. Note the further increase in MOE with the temperature indicative of residual crosslinking reaction after welding and limited holding.

material in the structure of wood, mainly lignin, but also some hemicelluloses. This causes the partial detachement, the "ungluing," of long wood cells, and the formation of an entanglement network drowned in a matrix of melted material which then solidifies, in short a wood cell entanglement network composite with a molten lignin matrix. During the welding period some of the detached wood cells which are not being held anymore by the interconnecting material are pushed out of the joint as excess fiber.

This is not, however, the complete story. The CP-MAS ¹³C-NMR spectra indicated that some crosslinking chemical reaction of lignin and carbohydrate-derived furfural also occurred. This contributed to the mechanical resistance of the joint, although to a minor extent. In this regard, the results of constant heating TMA testing of welded bondline joints yield an increase in the modulus of elasticity (MOE) of the joint at increasing temperatures (Figure 8). The increase occurs in two distinct steps indicating a distinct series of chemical reactions which go to completion only after welding has occurred. A distinct series of chemical crosslinking reactions contributes further to the strength of the joint, but the contribution comes to bear only after welding has finished. This might well be one reason why the longest holding time under pressure after the end of welding has such a strong contribution to a good bond (see Table III). After all, the increase in MOE with heating in Figure 8 is quite marked. These reactions might well be the completion of the initial reactions, which have been observed by NMR, hence an increase of strength due to some increase in crosslinking due to reactions of lignin and/or furfural.

CONCLUSION

Mechanically induced wood fusion welding, without any adhesive at the interface, yields rapidly bonding joints satisfying the relevant requirements of structural wood joints. Notwithstanding that the joints are acceptable only for interior use as they are not weather or water resistant, this technique opens the door to effective and inexpensive gluing of solid wood without adhesive for furniture and interior joinery. The mechanism of mechanically induced vibrational wood fusion welding is due mostly to the melting and flowing of amorphous cells-interconnecting polymer material in the structure of wood, mainly lignin, but also some hemicelluloses. This causes the partial detachment, the "ungluing," of long wood cells and the formation of an entanglement network drowned in a matrix of melted material which then solidifies, in short a wood cell entanglement network composite with a molten lignin matrix. During the welding period some of the detached wood cells which are not being held anymore by the interconnecting material are pushed out of the joint as excess fiber. Crosslinking chemical reactions of lignin and of carbohydrates-derived furfural also occurred but are minor contributors during the very short welding period. Their contribution increases after welding has finished, explaining why relatively longer holding times under pressure after the end of welding contribute strongly to obtaining a good bond.

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