COBEM-2017-2078
316L STAINLESS STEEL DIFFUSION BONDING OPTIMIZED PARAMETERS

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Abstract. Diffusion bonding is a process that joins two mating surfaces through the atomic diffusion of the material in the solid state. Comparing to conventional joining methods, such as brazing, the process is more useful for certain applications, due to the larger mechanical resistance achieved between the bonded surfaces. Other advantages are the absence of filler material that may cause corrosion problems, beyond the lower processing temperatures, which avoid the liquid phase formation and the deleterious microstructural modification associated.
These characteristics make diffusion bonding a suitable method for compact heat exchanger fabrication. The diffusion bonding is a result of the combination of three main parameters: heat, pressure and time. This paper provides an overview of the study conducted to determine suitable parameters for the bonding of AISI 316L stainless steel. Samples were diffusion bonded and from these bonded blocks, 40 specimens were machined for tensile tests as well as microstructural analysis, to define the best process parameters. It was concluded that the temperature is the parameter that most influences the quality of the bond, as expected, since it increases the mass transport. The temperature that yielded the best results was 1040ºC, regardless the pressure and time parameters evaluated, which resulted in similar mechanical property results. Therefore, those parameters will be later applied in the fabrication of 316L stainless steel cores of compact heat exchangers.

Keywords: Diffusion Bonding, Compact Heat Exchangers, 316L Stainless steel.

1   INTRODUCTION

Callister (2008) defines diffusion as the atoms migration from a lattice site to another, furthermore, it can only happen if there is an adjacent site and the atom have enough energy to break the atomic bond. According to the diffusion principle, it is possible to join two materials in the solid state if adequate heat and pressure are applied for a certain period of time. A practical application of this principle is the joining process called diffusion bonding. The Heat Pipe Laboratory (Labtucal/UFSC) develops compact heat exchanger technologies, where diffusion bonding is applied as the fabrication process of compact heat exchanger cores (Mortean et al, 2016). In the present work, a study of the best parameters to be applied during the bonding process of AISI 316L stainless steel samples has been performed. This joining technology has been chosen because of the superior characteristics of the resulting bond, such as high mechanical resistance (similar to the base material) and large temperature and pressure resistance during operation. Likewise, the lack of addition material avoids the occurrence of galvanic cell and embrittlement of the material (Paiva, 2008).

Accordingly to Schawartz (1969), the main parameters in diffusion bonding are temperature, mechanical pressure and time. Temperature favours the diffusion process by exponentialy increasing the atoms energy, as seen in eq. 1 (Callister, 2008) :

$$D = D_0 exp\left(-\frac{Q_d}{RT}\right) \tag{1}$$

where:
   D = diffusion coefficients,
   D_0 = a temperature-independent pre exponential (m^2/s),
   Q_d = the activation energy for diffusion (J/mol ou eV/atom),
   R = the gas constant, 8.31 J/mol-K or 8.62×10^{-5} eV/atom-K,
   T = absolute temperature (K),

The increase of the temperature increases the atoms energy, which makes atoms move more easily to vacant sites, thus increasing the mass transport kinetics through the interface, bonding the materials. Aditionally, the temperature used in the process is not supposed to reach the material melting point, so the formation of a liquid phase is not expected during the process.

Mechanical pressure is used mainly to ensure uniform contact between the pieces during the bonding cycle. In some cases, pressure is also necessary to break passive oxide layers present in some materials (Schawartz, 1969). Furthermore, the time of the bonding cycle is related to the diffusion length as seen in eq. 2 (Schawartz, 1969):

$$\delta = K\sqrt{dt} \tag{2}$$

where:
   δ = diffusion length,
   K = a constant,
   t = time.

The diffusion length is the average distance traveled by an atom during the diffusion process. The longer the diffusion length; the better is the bonding quality.

In the present paper, the best combination of the parameters for the diffusion bonding of AISI 316L stainless steel, within a set of chosen parameters will be investigated and presented. Those parameters are time, pressure and temperature, and after the conclusion of the proposed study, they will be used in the fabrication of compact heat exchanger cores.

2   EXPERIMENTAL PROCEDURES

2.1   Bonding Process

To achieve the results presented in this paper, 10 samples were produced using the high vacuum hot press furnace PVA Tepla MOV 653 HP, available at the Heat Pipe Laboratory/UFSC, presented in Fig. 1. The furnace has a useful volume of 0.3 m^3, supplies a maximum load of 250 ton and may operate at a maximum temperature of 1700 ºC.
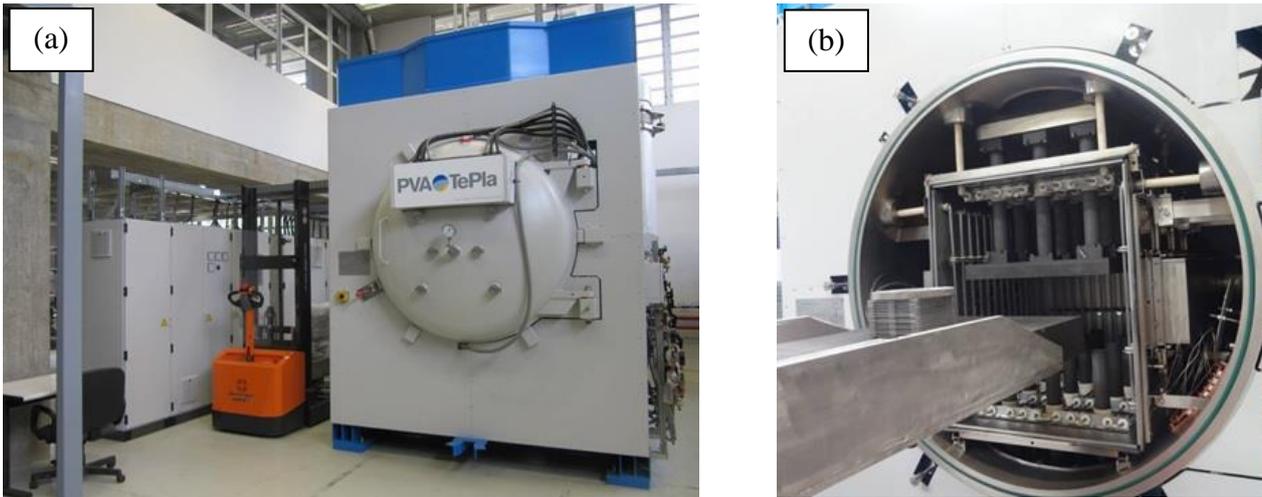
Figure 1 - (a) Hot press furnace and (b) chamber for diffusion bonding of the Heat Pipe Laboratory/UFSC

For the evaluation of diffusion bonding parameters, polished stainless steel (AISI 316L) blocks (47.5 x 105 x 105 mm) were cleaned in ethanol ultrasonic bath and then stacked two by two (Fig. 2-a), producing samples with dimensions of 95 x 105 x 105 mm. These samples were processed in the hot press furnace under high vacuum (less than 10^{-6} mbar), and subjected to different combinations of temperature, pressure and time, according to Tab. 1.

For comparison purposes, the sample identified as S10 (Fig. 2-b) was composed by only one block (105 x 105 x 100 mm), which was submitted to a thermal cycle similar to those used for bonding the two blocks but without applied pressure. This way, the effect of the thermal cycle could be disregarded when the mechanical properties were compared.

Table 1 – Parameters used to produce the samples.

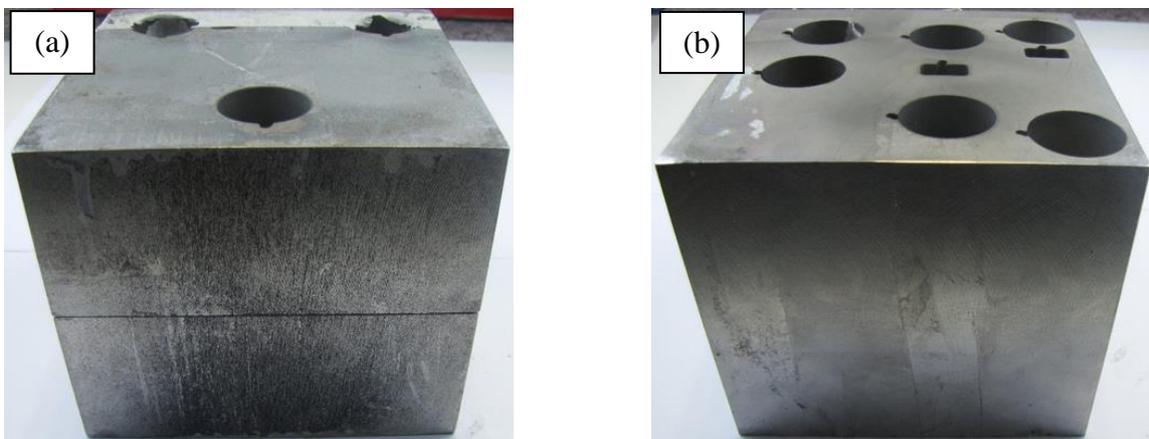| Sample	Temp. (ºC)	Pressure (MPa)	Time (min)
S0	-	As received	-
S1	945	8.75	105
S2	945	7.50	105
S3	945	10.00	105
S4	945	8.75	32
S5	945	8.75	179
S6	1040	9.51	60
S7	1040	9.51	150
S8	1040	7.98	60
S9	1100	8.75	105
S10	1100	-	105

Figure 2 - a) Sample after bonding and machining processes, b) As received material after thermal cycle and machining processes

2.2 Bonding Characterization

2.2.1 Tensile test

After the diffusion bonding of samples, at least three specimens were machined from the blocks for tensile tests and microstructural evaluation, as shown in Fig. 3 and 4. The as received sample (S0) and the block submitted only to the thermal cycle (S10) were machined the same way as the other samples, so that their results could be compared. Therefore, forty-four specimens were manufactured; the S0 sample has only two tensile tests results because one specimen was damaged during the machining process.

The tensile tests were performed at room temperature, accordingly to the standard ASTM E8, and the properties evaluated were yield strength, ultimate tensile strength (UTS) and elongation at rupture. The raw data were further analyzed to obtain the toughness of the produced specimens.
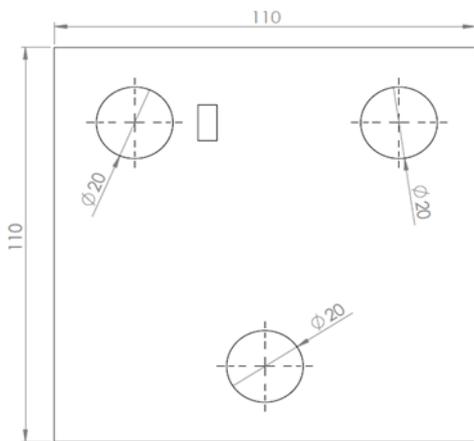
Figure 3 - Technical drawing for the water jet machining
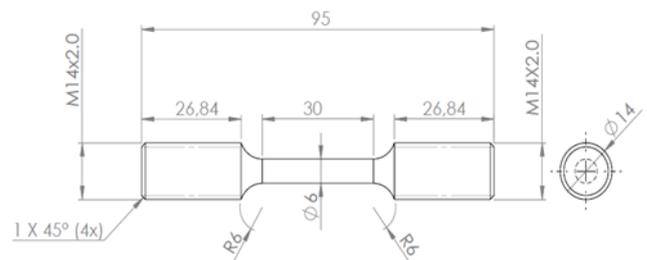
Figure 4 - Technical drawing of tensile test specimen

2.2.2 Microstructural analyses

After the water jet machining, for obtainment of specimens for microstructural evaluation, each specimen was sectioned in a cut-off machine in order to analyze only the bonded region. For the metallographic preparation, the sectioned specimens were hot mounted in bakelite resin, grinded using a sequence of sand papers (grind size 100, 220, 600, 1200 and 1500) and polished using a 1μm diamond paste.

In order to reveal the specimens microstructure, after the polishing process a chemical etch was performed by submerging the samples in a solution of glyceregia, which consists of a mixture of 10 ml HNO_3, 15 ml acetic acid, 10 ml HCl and 3 drops of glycerin. After etched, the microstructures resulting from each set of bonding parameters were analyzed under an optical microscope (OM - BX60, Olympus).

3 RESULTS AND DISCUSSION

3.1 Tensile test

Yield strength, ultimate tensile strength, elongation at rupture and toughness of all specimens were compared to determine the best set of chosen parameters for the fabrication of compact heat exchangers using the stainless steel AISI 316L. For a visual comparison, bar graphs related to the properties reached by each specimen were plotted with the corresponding average of the base material (black line - S0) and the standard deviation (gray lines).

When the UTS of specimens were compared, in the graph of Fig. 5, it can be observed that three samples presented a superior behavior when compared with the base material (S0), such specimens were produced using the diffusion bonding parameters S6, S7 and S8 described in Tab. 1. It is also possible to notice that the base material after the thermal cycle (S10) had its ultimate tensile strength lower than the material as received (S0).
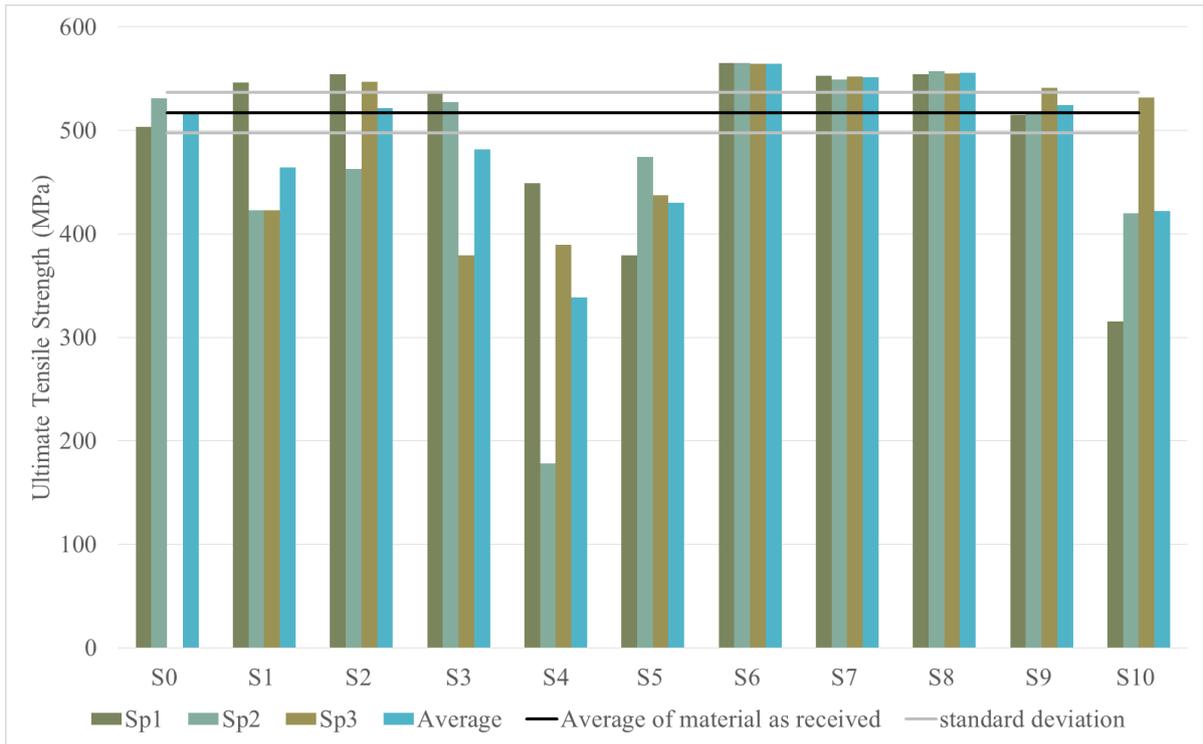
Figure 5 - Plot of sample's ultimate tensile strength

Furthermore, the yield strength results presented in the graph below (Fig. 6) indicates that the bonded samples S6 and S7 achieved an increased average yield strength when compared with sample S0 (S6 about 16%). On the other hand, the same property was detrimentally affected for the samples S4, S5 and S9. Recurrently, it is possible to notice that the sample S10 also had this property affected, with an average 5% lower than the as received material without the thermal cycle. The yield strength was calculate for all samples as 0.2 percent of the unstressed length.
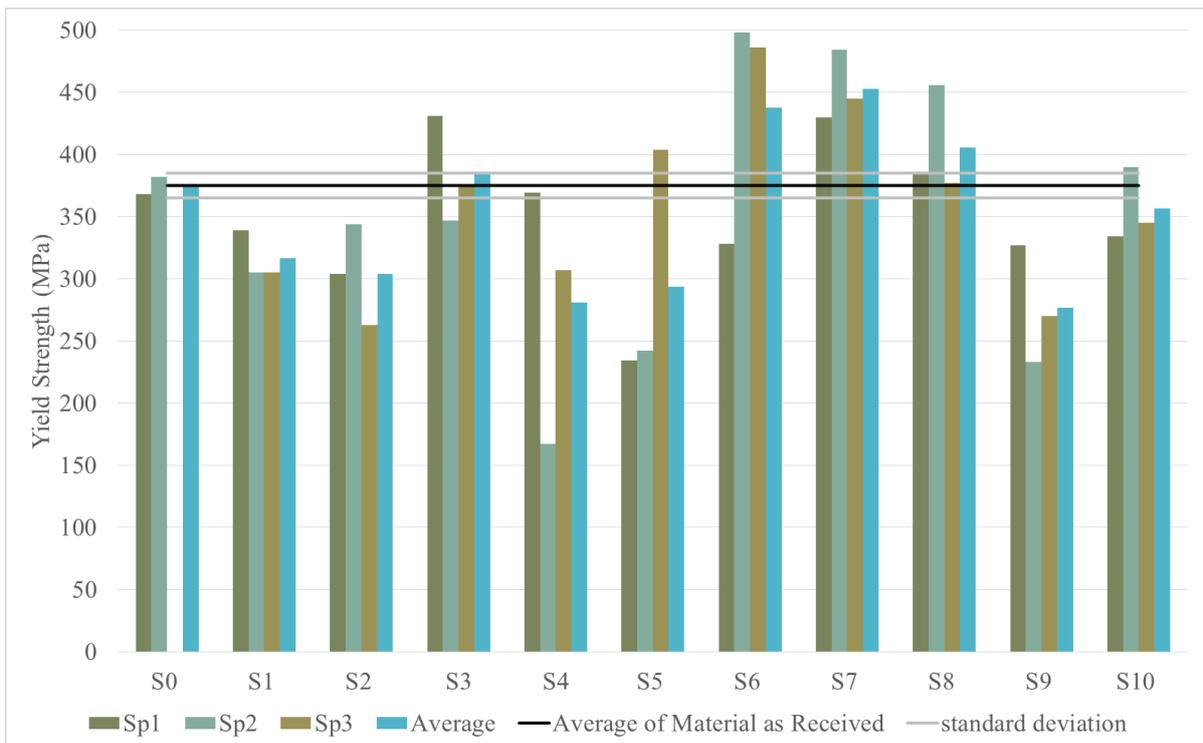
Figure 6 - Plot of sample's yield strength

Figure 7 compares the percentual elongation of specimens after rupture, in order to evaluate the behavior of each bond up to the rupture. These bonds can be either more ductile or brittle, presenting an increased or decreased elongation respectively, in relation to the material as received (S0). The plot in Fig. 7 indicates that most samples (S1-S5) presented a brittle behavior. The sample with closer result in comparison to the base material was S9, albeit slightly more brittle. Moreover, the samples S6, S7 and S8 were more ductile than the material as received, and sample S10 did not lose its ductility since its average elongation value is similar to the material as received.
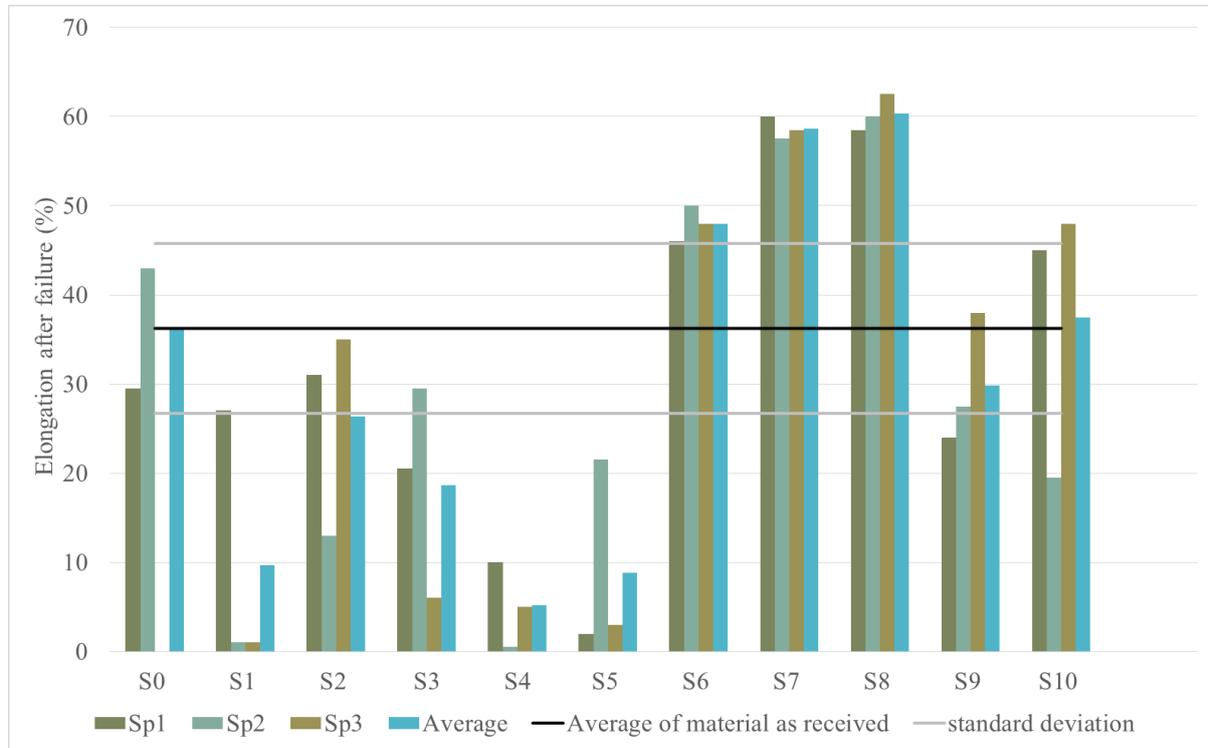
Figure 7 – Plot of sample's elongation after rupture

Another analyzed data was toughness up to the ultimate tensile strength, or the ability of a material to absorb energy and plastically deform without fracturing. Those values were calculated through the integration of the strain-stress curve for each specimen tested. Comparing the values obtained, graph presented in Fig. 8, it is possible to observe that the samples S8 and S9 have their toughness values similar to the base material (S0). Another important observation is that sample S10 did not maintain this toughness property, presenting an average value 67.5% lower than that of the as received material, and such behavior is being further investigated.
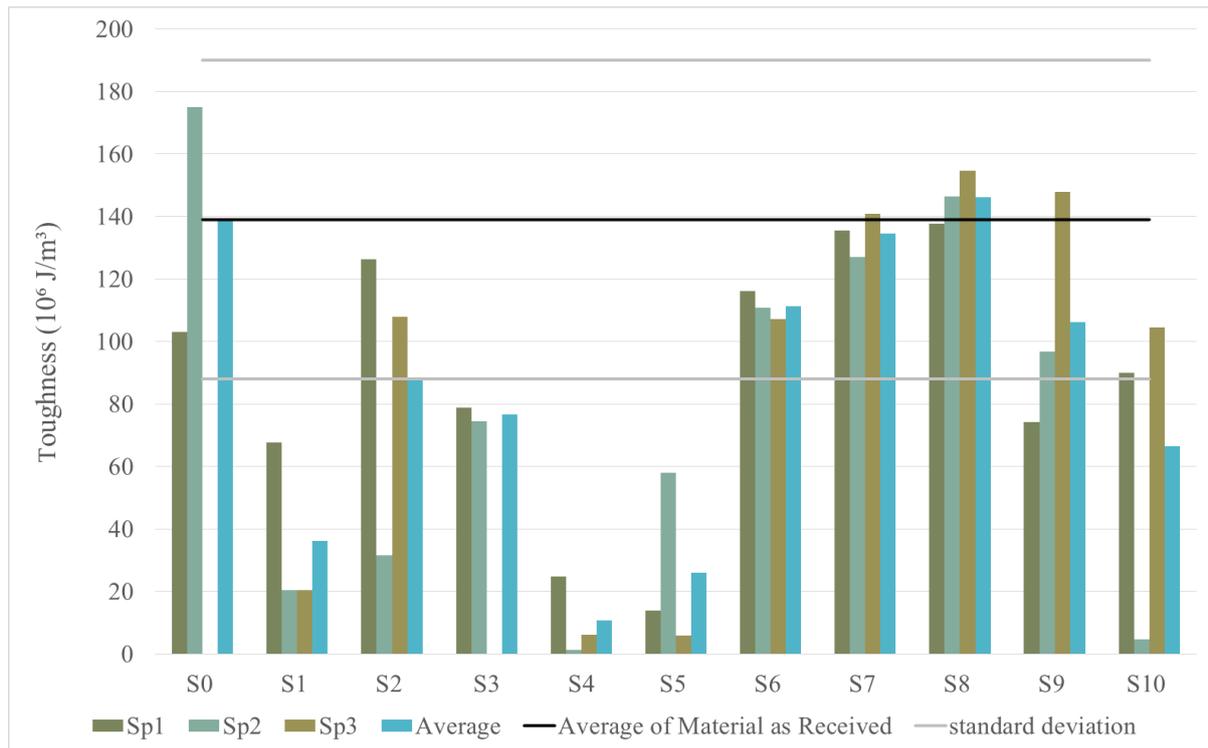
Figure 8 - Plot of the sample's toughness

When analyzing and comparing the ultimate tensile strength, yield strength, elongation after rupture and toughness, it is possible to state that the best bond parameters were the ones that were used for fabricating the samples S6, S7 and S8. Therefore, when observing the Tab. 1, it is possible to affirm that the best temperature (within the chosen parameters) for bonding 316L stainless steel is 1040 $^\circ$C, since all three samples were bonded under this temperature. Considering that those samples were bonded with different pressures and times, it is also possible to state that the main parameter that influences the bonding quality is the temperature.

Furthermore, samples S0 and S10 can be compared since both samples were not bonded and sample S10 passed through a thermal cycle. Comparing the results, one can see that sample S10 had lost some mechanical properties, as its values of UTS, yield strength and toughness were lower than "as received" material (S0). This means that the thermal cycle also influences the modification of mechanical properties of the material.

3.2   Microstructural analysis

Another method to evaluate the quality of bonding is through microstructural analysis, which allows a qualitative evaluation, unlike the tensile test, which is a quantitative analysis. By observing the samples under an optical microscope, it was possible to analyze the bonding in details and obtain the following images presented in Fig. 9.

The sharpness of the bonding line can be associated with the quality of the bond: the sharper is the line, the inferior is the bonding quality (continuous grains were not formed in the contacting surfaces) and vice-versa. When comparing the micrographs presented in Fig. 9 a to i, it is possible to visualize the difference in the sharpness of the bonding line, among the produced samples using different diffusion bonding parameters.
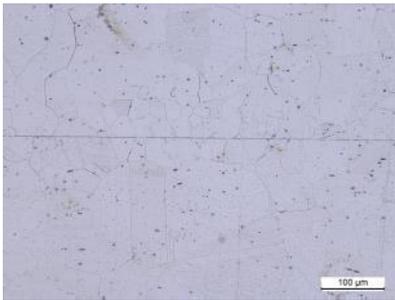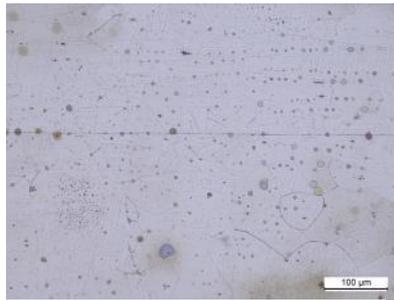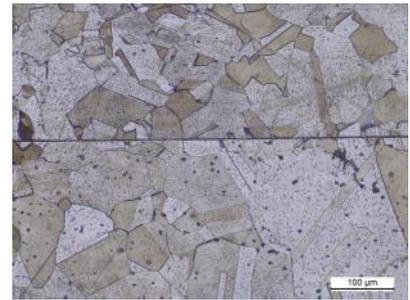
Figure 9a-Sample S1

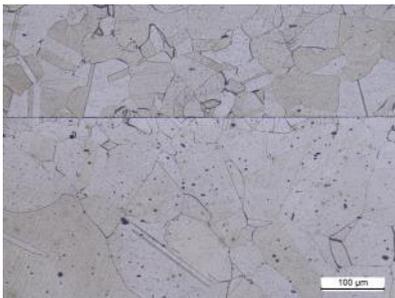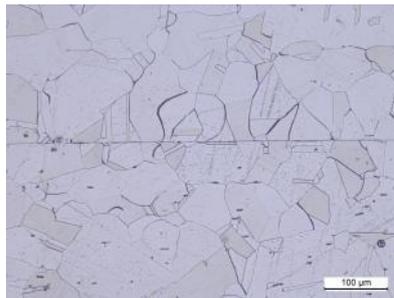Figure 9b- Sample S2

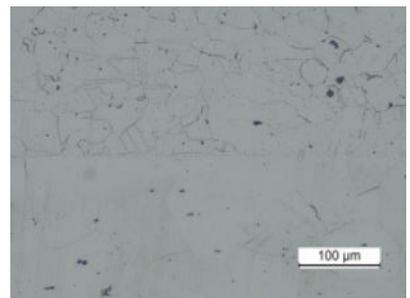Figure 9c- Sample S3

Figure 9d- Sample S4

Figure 9e- Sample S5
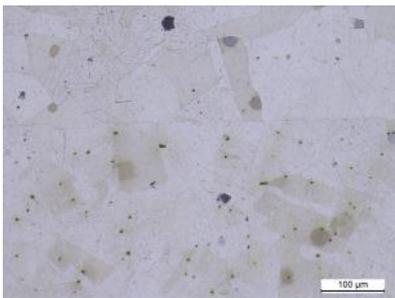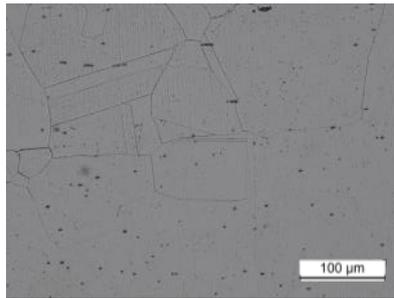

Figure 9f- Sample S6

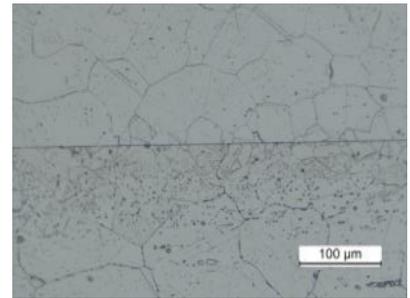

Figure 9g- Sample S7

Figure 9h- Sample S8

Figure 9i- Sample S9

One can see that samples S1 to S5 and S9 have sharper bonding lines when comparing to sample S6 to S8. This means that samples S6 to S8 had larger material continuity in the bonded region, compared to the other samples. Another comparison made from the microstructural analysis is between samples S0 and S10, possible by observing Figure 10, where it is possible to observe a qualitative increase in grain size. This effect was expected, as the temperature increase tends to increase the size of the austenite grains (Chiaverini, 1996).
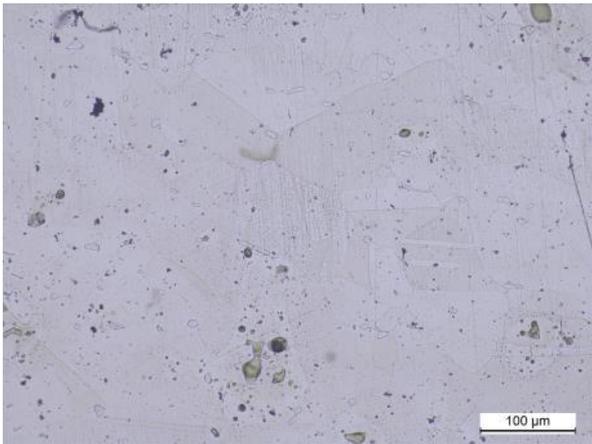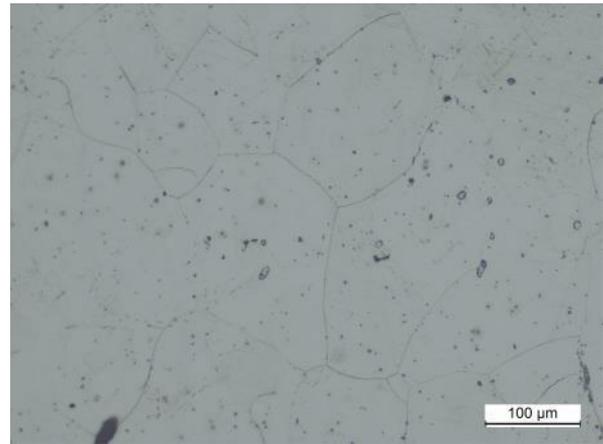
Figure 10a- Sample S0

Figure 10b- Sample S10

4   CONCLUSION

In this study, the best parameters for 316L stainless steel diffusion bonding process were identified, within the investigated set of parameters. Based on the described experimental procedure, it was established that the parameter that most influences the quality of the bond is the temperature, as expected since it increases the mass transport kinetics and atomic mobility through the interface. Furthermore, the temperature that yielded the best results was 1040ºC, applied in the bonding of samples S6, S7 and S8. Comparing those samples, one can see that they were produced with different pressure and time parameters, but they presented similar property results. Therefore, those parameters will be later applied in the fabrication of 316L stainless steel cores of compact heat exchangers.

Another conclusion from this study is that the temperature can influence the mechanical properties of the material. Comparing samples S0 and S10, one can see through the tensile test results that there was some loss in mechanical properties in the base material, after the thermal cycle. This is possibly caused by the grain size increase, observed in the microstructural analysis.

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