

# ENHANCING DIFFUSION BONDING

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## Abstract

The purpose of this project is to study the enhancement of diffusion bonding by mainly analyzing the effects of dislocations on atomic migration at the interface in 316L. Therefore, the relationship between dislocation densities and bonding quality was investigated.

It was observed that dislocations successfully enhanced bonding quality by reducing porosity, decreasing average pore size, and increasing grain boundary migration ratio at the interface. These results were characterized using an optical microscope and a scanning electron microscope. Furthermore, the importance of an optimal surface roughness was highlighted, as it enables enough contact area between the components, enabling dislocations to function effectively.

Finally, the effect of different temperatures and heating rates on grain coarsening and grain boundary density evolution was analyzed and quantified. Concluding that different heating rates resulted in a similar grain coarsening, whereas a lower temperature maintained the initial grain size in the material. Suggesting that a lower temperature could potentially further enhance bonding quality at the interface by facilitating faster atomic migration through the grain boundaries.

## Preface

I would like to express my sincere gratitude to everyone who has supported me throughout this project. Mainly to my mum, Ixiar, my dad, Migel, and my sister Alaitz, together with my entire family and boyfriend Mikel, this would not have been the same without their support.

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# List of Abbreviations

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Table 1: Abbreviations used in this study

| Abbreviation | Definition                        |
|--------------|-----------------------------------|
| DB           | Diffusion Bonding                 |
| GBM          | Grain Boundary Migration          |
| GB           | Grain Boundary                    |
| AR           | As Received                       |
| SS           | Stainless Steel                   |
| SEM          | Scanning Electron Microscope      |
| EBSD         | Electron Back Scatter Diffraction |
| KAM          | Kernel Average Misorientation     |
| Rx           | Recrystallization                 |
| OPS          | Oxide Polishing Suspension        |
| ND           | No deformation rate               |
| LDR          | Low deformation rate              |
| MDR          | Medium deformation rate           |
| HDR          | High deformation rate             |
| FMEA         | Failure Mode and Effect Analysis  |
| WBS          | Work Breakdown Structure          |

# 1 Introduction

---

## 1.1 Background

Diffusion bonding is a solid-state bonding process, where atoms gradually migrate from one component into another [1, 2]. Bonding of components happens at elevated temperatures and pressures over a specific bonding time. One of the key challenges in diffusion bonding is to achieve strong, defect-free joints with shorter bonding times and lower bonding temperature [1, 3].

The bonding process can be divided into three different stages. In the first stage, components undergo plastic deformation, here, the contact surfaces of the components deform plastically, increasing the initial contact area between the components to be bonded. During the second stage, the creep mechanism takes place, leading to interfacial pores' closure. In the last stage, atoms diffuse through the interface, which results in the final elimination of pores at the interface [4].

Surface dislocation density is a key factor in accelerating the diffusion bonding process. Dislocations are crystallographic defects in the material, and these provide fast diffusion pathways, increasing the atomic mobility in the components. Increasing the dislocation density can improve joint strength and reduce the bonding time [5].

This study aims to deepen the understanding of key bonding parameters and provide a more optimal approach for diffusion bonding. Therefore, apart from the conventional bonding parameters, the effect of dislocation density on the bonding quality has been analyzed, and guidelines for industrial applications have been provided.

## 1.2 Objectives

The main objective of this project is to study the effects of key parameters on diffusion bonding, with the most focus on dislocation densities. The aim is to determine whether diffusion bonding quality can be improved by introducing dislocations, while reducing the need for costly or time-consuming changes, such as longer bonding times, higher bonding temperatures, or improved surface finish. Therefore, sub-objectives have been defined. These are outlined in the following points:

- Identify bonding parameters to achieve high joint strength and minimal porosity for SS 316L in diffusion bonding.
- Develop a mechanical pre-treatment method to increase dislocation density (e.g., surface deformation, shot peening).
- Investigate the relationship between surface dislocation density and diffusion bonding quality.
- Quantify the effect of enhanced dislocation density on diffusion rates, microstructure evolution, and mechanical properties of bonded joints.
- Characterize the microstructure and mechanical properties of diffusion-bonded joints using characterization techniques (SEM, hardness testing).
- Investigate the benefits of dislocation density for production scale bonding cycles.
- Evaluate how the bonding cycle could be further optimized while still increasing or maintaining bonding quality.
- Provide guidelines for industrial application of dislocation-enhanced diffusion bonding.



### 1.3 Scope and limitations

The study has been limited to laboratory scale experiments, industrial-scale validations are outside of the scope. The study will just focus on a specific stainless-steel alloy.

Apart from this, characterization will be restricted to available techniques such as optical microscopy, SEM, or surface characterization. Environmental effects, oxidation, and contamination in the process will not be explored

The furnace used for the experiments was not designed for this purpose, therefore, instead of performing the tests in a specially built diffusion bonding furnace, a simpler vacuum furnace was used. Therefore, the bonding pressure was applied as weights on the samples before introducing them into the furnace. Moreover, the machines used for sample preparation in these experiments were not designed for this purpose, so sample preparation was not always repeatable.

Due to confidentiality requirements, the exact values of some of the key parameters used are not shown in this report. However, these parameters are still compared and discussed using qualitative descriptions, such as higher/lower, rougher/smooth, longer/shorter, faster/slower... and similar comparative terms. Therefore, this should not affect the understanding of the results presented in this study.

### 1.4 Activities

To complete this project, an initial project organization was first designed. Afterwards, research was done to study how diffusion bonding works, including principles, mechanisms, and key influencing parameters. Later, a detailed experimental plan was developed. The experiments were carried out together with continuous analysis of the results and meaningful conclusions. The report was written based on these conclusions. Lastly, some guidelines and suggestions were also given for future work.

The overall structure of the activities is summarized in a work breakdown structure (WBS), which can be seen in Figure 1, which provides an overview of the tasks and their interrelationships.

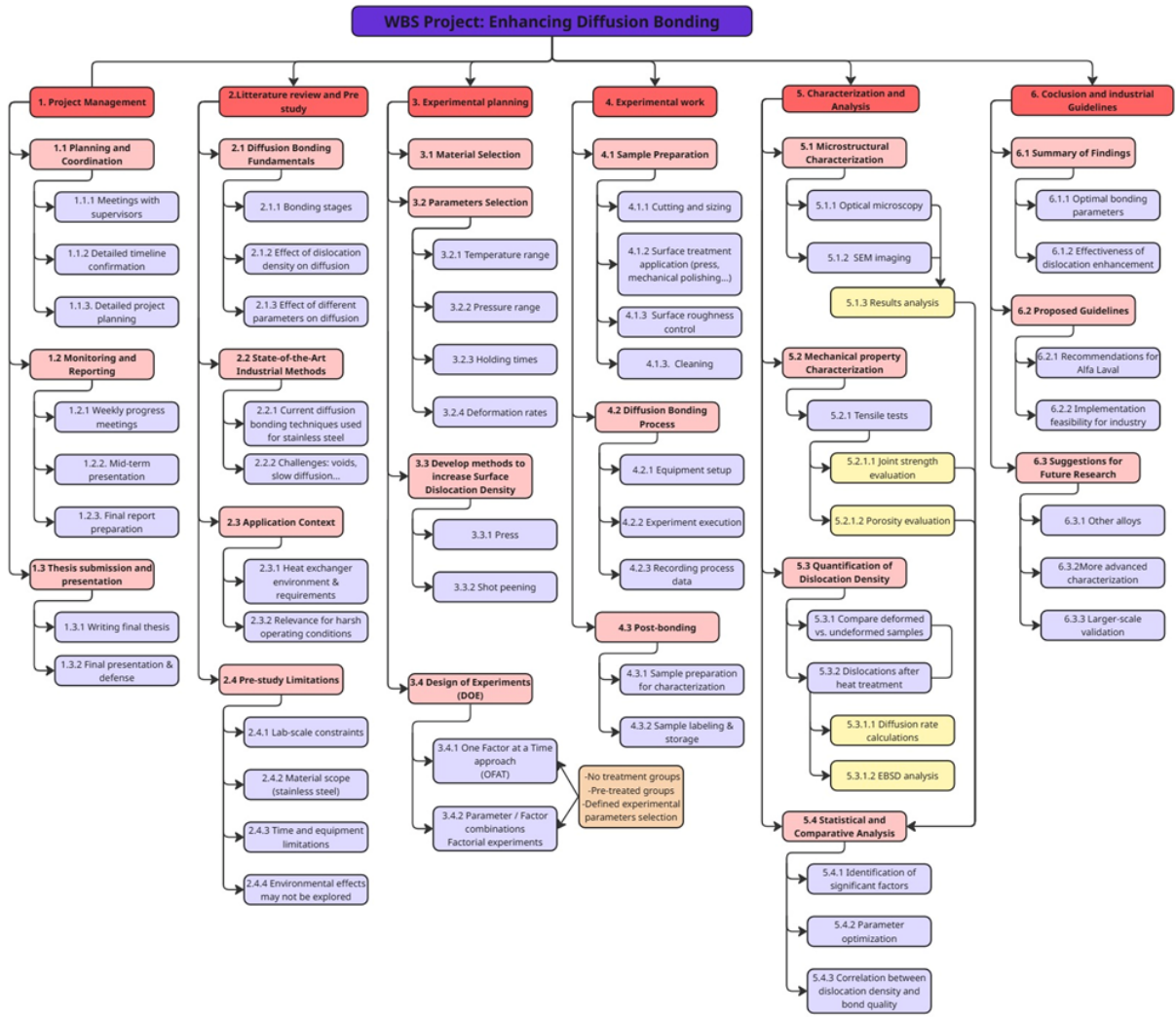


Figure 1: WBS.

The project started on January 19, 2026, and was finished on June 19 of the same year. The Gantt chart planning for the project is shown in Figure 2. The project ran for a total of 22 weeks, with the last two weeks reserved as a buffer. This Gantt Chart was developed based on the tasks previously described on the WBS, shown in Figure 2.

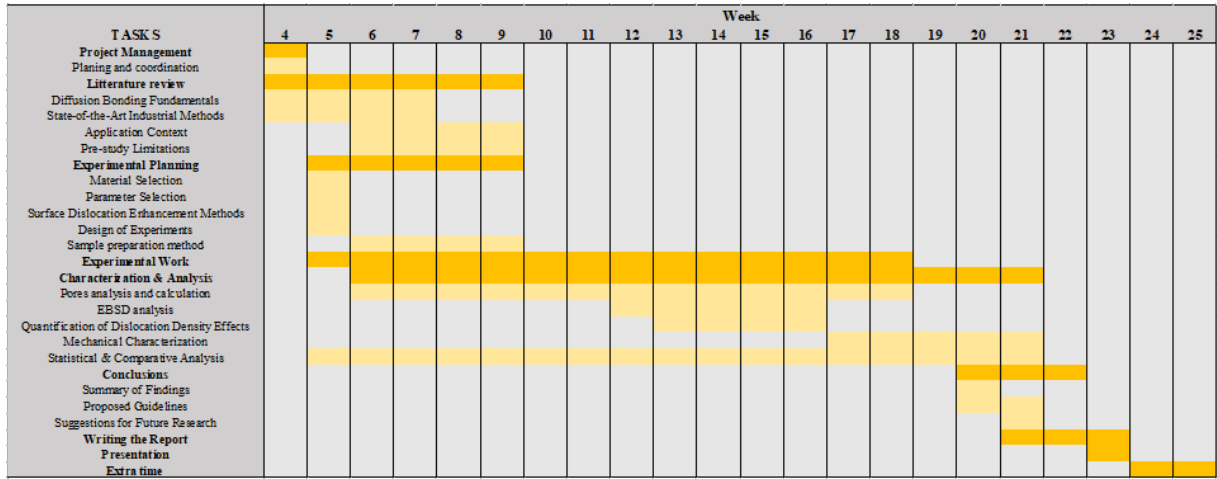


Figure 2: Gant Chart.

## 1.5 Resources and dependencies

This project depends on the availability of technical, material, and human resources. The availability of the bonding furnace, together with the hydraulic press, is essential to the process, directly influencing the experimental timeline and the number of bonding trials that can be performed. Apart from this, the study also relies on the availability of other machines, such as surface polishing and cutting machines, which are required for proper sample preparation.

Collaboration with company technicians and supervisors is necessary for proper training on laboratory equipment and guidance throughout the project. This will mainly be important at the initial stage of the project, but will later affect experimental success.

The identified risks related to this project are described in a Failure Mode and Effect Analysis (FMEA), in Appendix A). This approach is used to identify potential failure modes together with their causes, effects, and detection ratios. Furthermore, it also identifies the impact of these potential failure modes and suggests mitigation strategies [6].

## 2 Literature review

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Nowadays, solid-state joining methods are widely used in manufacturing to join both dissimilar and similar materials. These processes happen under the materials' melting temperature, in the solid state, and are based on atomic transport across the interface, resulting in atomic migration from one component to another. These solid-state joining processes include forge welding, friction welding, ultrasonic welding, explosion welding, cold welding, and diffusion bonding. [1]

In forge welding, components are heated and afterwards hammered. This is one of the oldest processes, and it is used to manufacture tools and structural components [1]. On the other hand, friction welding is used for mass production, mainly for the automotive and aerospace industries. In this case, the components rub against each other, and the heat is generated by the friction at the interface. When the temperature is high enough at the interface, more pressure is applied, and the components come together, forming strong joints [1, 7].

Ultrasonic welding is also a well-known joining process for thermoplastics or lightweight metals. Mechanical energy is transformed into thermal energy due to friction, and this will heat and soften the components at the joint line. This process is mainly used for medical, electronic, and automotive applications [1, 8]. Another used process is explosion welding, here, high velocity oblique collision is used to join the metals and is mainly used to join or clad tubes or pipes. Cold welding process produces defect free joints by applying very low heat and very high pressure to bring the joining surfaces to irrelevant nanoscale distances, so that the atoms can migrate from one surface into another [1].

Lastly, diffusion bonding is a solid-state process used in advanced engineering applications. Elevated temperatures, together with pressure, are applied to join the components. Using high temperatures, pressure, and holding time, atoms diffuse from one component to another. This is an advanced technology where excellent welding quality is obtained [1, 2].

It has been seen that solid state joining processes are very effective for joining materials such as aluminum alloys, titanium, stainless steel, and medium entropy alloys. These processes are used for getting strong joints together with cost reduction, energy savings, and enhanced material performance in different industrial applications such as automotive, aerospace, nuclear, marine, and electronics sectors. [1].

### 2.1 Diffusion bonding principle

In this specific project, diffusion bonding is the process that has been studied. As said, in this solid state process, atomic diffusion happens through the interface. To achieve this bonding, high temperature and pressure are applied to the components to guarantee that the interfaces are in close contact.

Diffusion bonding process can be explained as follows in Figure 3 [9]:

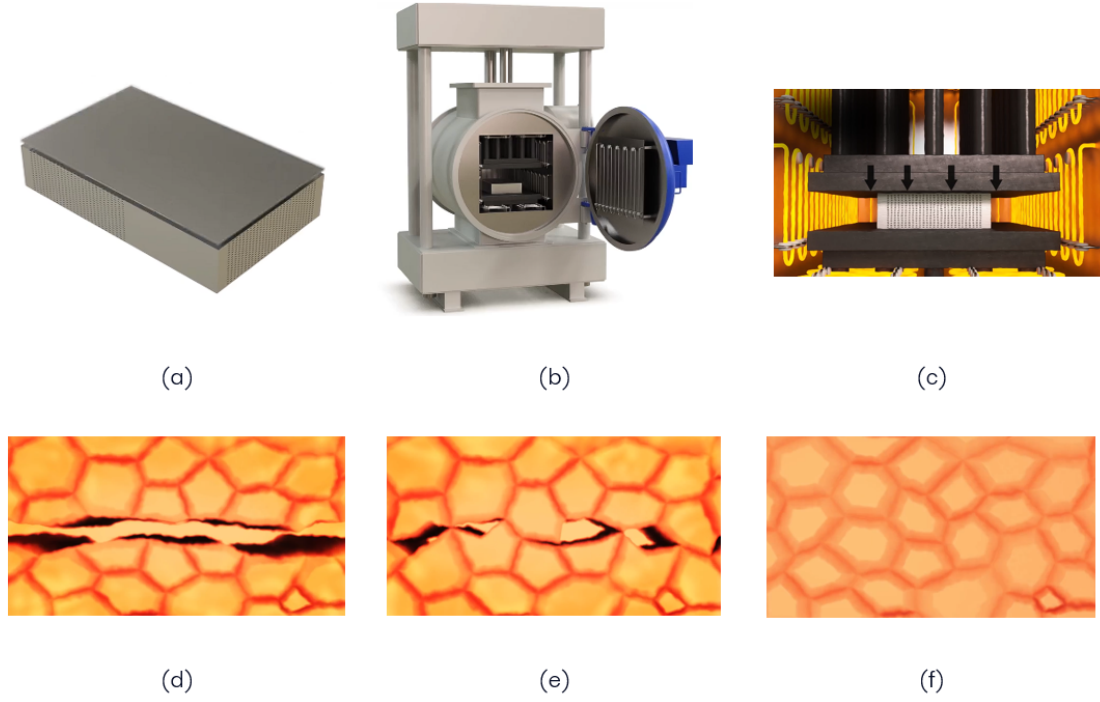


Figure 3: Bonding process. [9]

First, the layers that are intended to bond are assembled one on top of the other. Afterwards, these are taken into a special vacuum furnace, where heat is applied. When the desired bonding temperature is achieved, a bonding pressure is applied against the piled layers. (As can be seen in Figure 3 (a)(b)(c) [9]). This pressure is applied during a specific holding time. The atomic diffusion causes crystalline interpenetration, and atoms diffuse from one component into the other, creating very strong joints. (Figure 3 (d)(e)(f) [9])

Figure 4 is a schematic representation of what occurs in the vacuum furnace during diffusion bonding. It shows that an initial constant low pressure is applied from the beginning while the furnace is heated at a specific heating rate. When the desired temperature is achieved, it remains constant for a certain bonding/holding time. During this time, a higher constant bonding pressure is applied to the components. Atoms diffuse across the contact surfaces during this holding time, forming indistinguishable atomic bonds and creating a metallurgical join. After this holding time at a high constant bonding pressure and time, the bonding pressure is released, and the furnace cools down at a certain cooling rate. [10, 11]

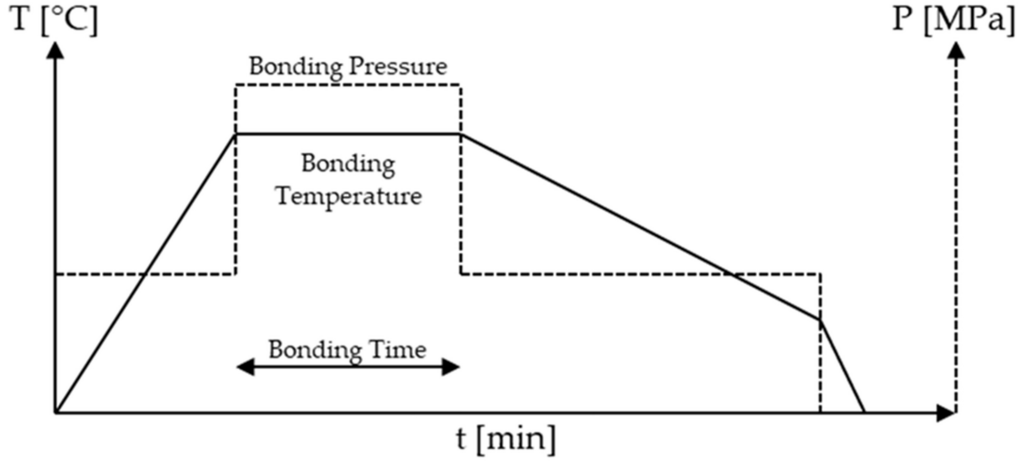


Figure 4: Diffusion Bonding furnace schematic representation. [10]

The evolution of the surfaces in contact during the bonding can be seen in Figure 5. There is an initial surface contact area, which mainly depends on the quality of the surfaces. Once bonding pressure and temperature are applied, a first stage deformation and boundary formation start to happen. Deformation in this stage is dominated by plastic deformation, and it also results in the closing of the initial interfacial voids. After a certain time, the secondary stage takes place, where creep happens together with pore closure. In the last stage, pore closure and elimination occur. Here, grain boundary migration happens, and the initial bonding line (interface) disappears, resulting in a diffusion bonding joint. In summary, initial voids are formed in the first stage, then in the following stages, these are closed and eliminated [4, 11].

In order to ensure close contact between the contact surfaces, a high enough pressure has to be applied. If the applied pressure is insufficient, voids at the interface may stay unfilled, which will increase the porosity ratio and weaken the interface strength or joint quality. On the other hand, the holding time and the applied temperature are also key factors to guarantee effective bonding. [4]

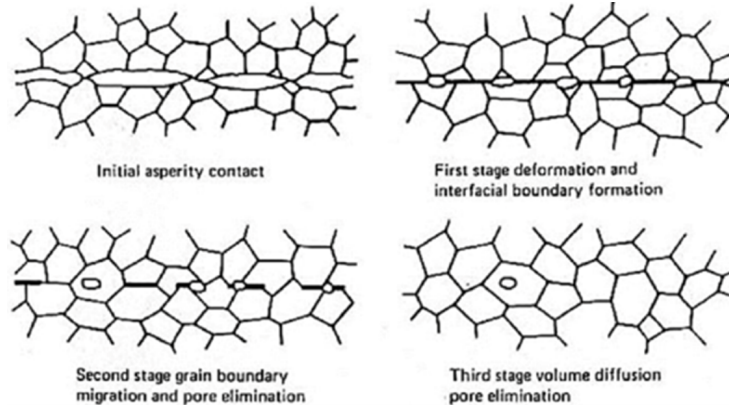


Figure 5: Evolution of the contact area during the bonding. [11]

This process has numerous benefits. As mentioned in the previous section, similar and dissimilar materials can be joined. Apart from this, since it is a solid state process, there is no melting of materials. A high-strength joint is produced, and it can withstand high pressure and temperatures, similar to those of the parent material, making it suitable for hazardous environments. In addition, it's a self-generating process, just one single material can be used, providing better corrosion resistance. However, the disadvantages are that special furnaces are needed and that it is a time-consuming process, which increases the process cost [3].

### 2.1.1 Void closure

At the first stage of diffusion bonding, when surfaces come into contact, initial voids are created. It is known that the initial size of these voids is mainly affected by the surface characteristics, rougher the contact surfaces, larger are the generated initial voids. Therefore, smoother surfaces enhance the pore closure ratio [12, 13].

During this bonding stage, the initial voids are relatively big, as a result, the length of the actual contact area at the interface is small, indicating that a high pressure is concentrated on these contact areas [13]. When the interfacial stress value is greater than the base material's yield strength, plastic deformation occurs, reducing the size of these initially created voids and creating larger contact areas [13, 14]. The Johnson-Cook (Equation 1 [14]) model describes the stress-strain relationship during this plastic deformation [14]. The degree of connection by plastic deformation can be studied by using Johnson-Cook's theoretical analysis [13, 14].

$$\bar{\sigma} = (A + B\bar{\epsilon}^n) \cdot \left[ 1 + C \ln \left( \frac{\dot{\bar{\epsilon}}}{\dot{\bar{\epsilon}}_0} \right) \right] \cdot \left[ 1 - \left( \frac{T - T_{\text{room}}}{T_m - T_{\text{room}}} \right)^m \right] \quad (1)$$

In Equation 1,  $\bar{\sigma}$  is the Von-Mises equivalent to plastic stress,  $\bar{\epsilon}$  is the equivalent plastic strain,  $T_{\text{room}}$  is the room temperature during the experiment,  $T_m$  is the used material's melting point temperature,  $T$  is the used temperature in the experiment.  $A$ , is the yield strength of the used material,  $B$  is the strain hardening coefficient of the material, and  $C$  is the strain rate sensitivity coefficient [14].

The relation between the applied bonding pressure and the plastic deformation depends on the matrix's yield strength, making the material's strength a key parameter. As the temperature rises, the material's strength decreases. Therefore, the combined influence of bonding pressure and temperature will initiate the plastic deformation mechanism in this initial stage [13].

During this initial contact stage, the highest point of the voids (tips) experiences the highest stress. These highest points are the ones that make first contact between the two surfaces and experience plastic deformation. Plastic deformation at these tips continues until the contact stress becomes lower than or equal to the material's yield stress [4, 14]. These tips suffer transient stress values, from higher to lower stresses. As the contact area grows, the pressure on the connecting interfaces lowers. This area grows because of the reduction of the void sizes caused by plastic deformation [4, 14, 13]. The reduction of the void sizes and increase of the contact area will also increase the bonding ratio [4]. Pressure changes are known to have the biggest impact on the micro convex contact, it directly affects the initial void shape and consequently the diffusion bonding process [14].

Once the surface micro convexes come into contact during the plastic deformation phase, the creep stage begins [12, 14]. The creep stage is a time-dependent deformation under a constant stress and is divided into three different stages: decelerated transient creep, steady-state creep, and accelerated creep. [14]. The steady-state creep stage is the most significant one for the bonding ratio. In this stage, rather than having just high stress concentrations in the void's contact tips, stress affected zones are expanded above the neck and tip of the voids during the creep. This stress distribution expands over time. Therefore, the stress values at the tips and at the neck of the void decrease over time. In this creep stage, material flow happens from the contact area to the neck of the pore, as a result, the void size reduces, and the contact area at the interface continues to grow, therefore also reducing the stress in the contact area [12, 14]. The relationship among the stress, time, and strain ratio during a constant temperature in the steady-state creep stage is described by Norton's creep law in Equation 2 [4]. Here,  $\dot{\epsilon}_c$  represents the strain rate,  $A_c$  represents the material's parameter,  $\sigma_c$  represents the applied stress,  $n_c$  represents the stress sensitivity index,  $t$  represents time, and  $m_c$  represents the time sensitivity index [4].

$$\dot{\epsilon}_c = A_c \cdot \sigma_c^{n_c} \cdot t^{m_c} \quad (2)$$

During creep, the void closure happens at varying rates, and void size presents a closing trend with a decreasing changing ratio [4, 14]. This can be seen in the simulation results shown in Figure 6, where void closure happens faster at the initial stage of creep and decreases with time. Results in this simulation also show that the void closure is mainly influenced by the creep stage rather than by plastic deformation. Finally, in these results, the influence of temperature on void closure can also be seen; void height reduces faster at higher temperatures [14].

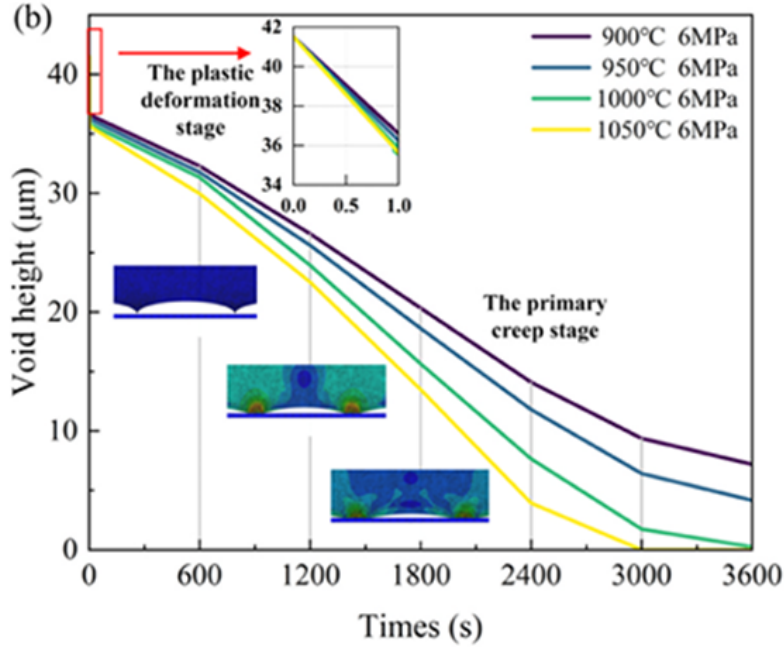


Figure 6: Void height closure as a function of time during diffusion bonding. [14]

The main process for interfacial void closure is bulk deformation (creep and plastic deformation). If the bonding time is prolonged, the bonding ratio keeps increasing up to a certain point where the growth stabilizes because of decreasing creep rate [14, 15].

Even if the bulk deformation is the primary mechanism for bonding, diffusion mechanisms also contribute to void closure, this includes interface source and surface source diffusion mechanisms. When it comes to interface source mechanisms, atoms migrate across grain boundaries or dislocations at the interface, enabling void closure and the interchange of the atoms at the interface. These dislocations can be eliminated in the later stage of the bonding as the temperature rises. On the other hand, the surface source mechanisms happen at the very beginning, and it fills small interfacial voids by fast migration of the atoms across the materials' surfaces [14, 15]. The influence of creep, surface source diffusion, and interface source diffusion on the bonding ratio over time can be seen in Figure 7 [15].



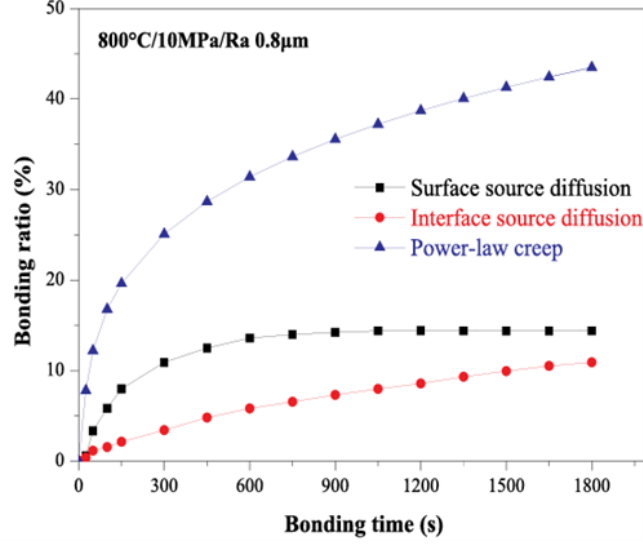


Figure 7: Bonding ratio as a function of time during diffusion bonding. [15]

#### Effect of pores on mechanical properties

It is known that as porosity or the average pore size at the interface decreases, tensile strength, micro-hardness, and overall ductility increase, increasing the overall mechanical properties of the joint [16]. The negative effect of porosity on tensile strength can be seen in Figure 8(a).

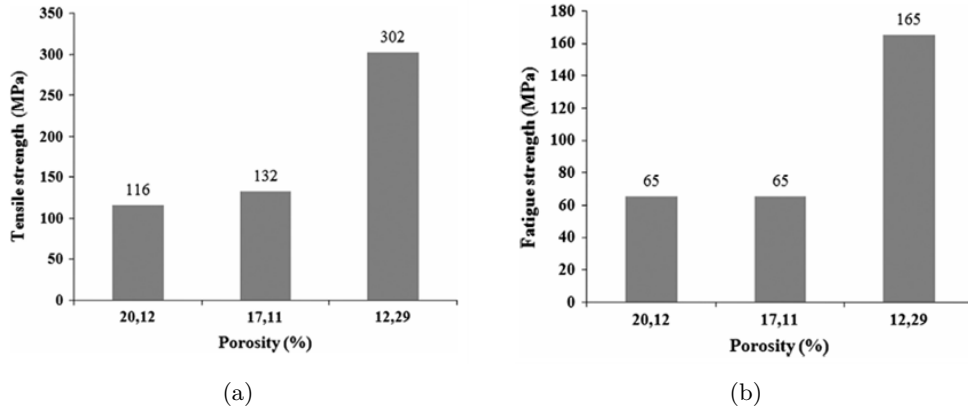


Figure 8: Tensile strength and Fatigue strength as a function of Porosity. [16]

A low porosity ratio is generally associated with smaller and more spherical pores, resulting in a more ductile structure and enhanced overall fatigue strength. In contrast, larger average pore size results in the formation of irregular pores and weaker internal bonds, which reduces the bonding strength and increases the chances of easier fracture breakage. Furthermore, fatigue fracture initiation is also more likely to happen with irregular pores at the interface. As said, increasing the porosity ratio results in more irregular voids, and the effect of these on the fatigue strength can be seen in Figure 8(b) [16, 17].

Areas with voids at the grain boundaries suffer localized stresses, therefore, crack initiation is typically reported where these pores are placed. Once a crack occurs, due to stress concentrations, the crack spreads into the surrounding material, and it grows following the stress path [16, 17]. In addition, the size of the voids also affects the distribution of stresses at the interface, and larger voids suffer higher stresses. Therefore, crack initiation is more likely to happen when the pores are larger due to these higher stress concentrations [16].

### 2.1.2 Quantifying join quality in diffusion bonding

Joint quality can be assessed using different quantification analyses, these include the interface bonding ratio, porosity ratio, average pore size, and grain boundary migration ratio, all of which are explained in this section.

#### Interface Bonding and Porosity ratio

The interface bonding ratio is defined by the size and amount of the voids at the interface. This ratio shows how much of the interface has been effectively bonded and is used to assess the joint quality. A higher bonding ratio indicates a higher bonding quality at the interface. This value is obtained by following Equation 3. Here, the void length is represented by the total sum of every void's horizontal length at the interface, as can be seen in Figure 9, in this case,  $L_n$  is the sum of  $L_1, L_2$  and  $L_3$  [12].

$$\text{Interface Bonding ratio (\%)} = \frac{\text{Total interface length (L)} - \text{Void length (L}_1 + L_2 + L_3 + \dots + L_n)}{\text{Total interface length (L)}} \times 100\% \quad (3)$$

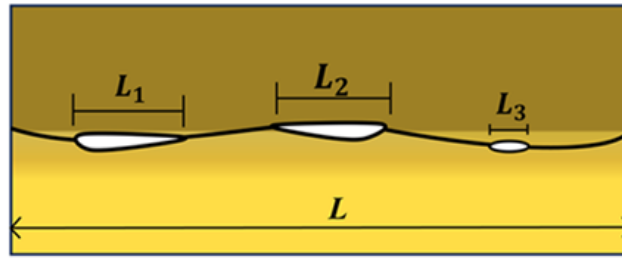


Figure 9: Illustration for porosity ratio calculation. [12]

On the other hand, the porosity ratio is calculated using Equation 4. The higher the porosity ratio, the worse the quality of the diffusion bonded joint [12]. In this project, the porosity ratio will be used to assess the quality of the joint.

$$\text{Porosity ratio (\%)} = \frac{\text{Total void length (L}_1 + L_2 + L_3 + \dots + L_n)}{\text{Total interface length (L)}} \times 100\% \quad (4)$$

#### Average pore size

In general, the size and the number of pores at the interface are related. For a given porosity ratio, the average pore size is inversely related to the total number of pores at the interface. In other words, for the same porosity ratio, a lower average pore size is related to a smaller (length) but a higher number of pores. While a higher average pore size is associated with a larger but lower number of pores. When the number of pores is higher, the defects are more uniformly distributed and, therefore, stresses are more evenly spread. These small pores distribute stress among all the corners at the interface, consequently creating lower peak stress values but more stress concentration points, which lowers the material's Young's modulus and makes it less stiff. However, a larger average pore size will increase Young's modulus, resulting in a stiffer structure. A larger average pore size also increases the standard deviation of the pore size (bigger pore size variation), increasing the maximum pore size value [18].

The average pore size and the maximum pore size values are also related to the amount of average historical energy that the material can absorb. As interfaces with lower average pore size have more evenly distributed voids, being more capable of higher energy absorption before fracture [18]. Therefore, interfaces with higher average pore size values can absorb less energy and make the structure more sensitive to early stage cracking, enhancing fracture breakage, as previously shown in Figure 8 (previous page) [18, 16].

The average pore size at the interface is calculated using Equation 5

$$\text{Average pore size} = \frac{\text{Total void length } (L_1 + L_2 + L_3 + \dots + L_n)}{\text{Number of voids}} \quad (5)$$

### Grain Boundary Migration (GBM)

Apart from the bonding ratio and the average pore size, Grain Boundary Migration (GBM) is also another factor that significantly affects the joint quality and strength. As the bonding process progresses, grains start to bulge, this is known as GBM, and this is also used to quantify joint quality. As shown in Figure 10, this parameter is measured by dividing the sum of the horizontal length of the migrated grain at the interface ( $L_1 + L_2 + L_3 + L_4 + \dots + L_n$ ) by the total bonding length of the interface ( $L$ ), using Equation 6. There is no distinction between the triple junction ( $L_1$  and  $L_2$ ) or the strain induced migrations ( $L_3$  and  $L_4$ ) to measure GBM ratio [19, 20]

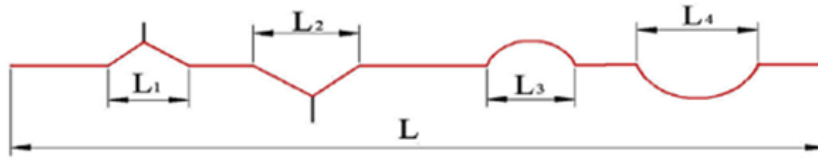


Figure 10: Illustration for GBM. [20]

$$\text{GBM ratio (\%)} = \frac{\text{Migrated GB length } (L_1 + L_2 + L_3 + L_4 + \dots + L_n)}{\text{Total interface length } (L)} \times 100\% \quad (6)$$

Higher bonding temperature or holding time is directly related to enhancing the GBM ratio. As bonding temperature increases, a higher percentage of GBM occurs, effectively leaving the voids in the inner regions of the grains (intragranular voids), instead of in the grain boundaries (intergranular voids) [21].

When the grains do not migrate through the interface, voids remain in the grain boundaries, and under tensile stresses, a heterogeneous plastic deformation and large plastic flow occurs around the voids. However, when the pores are left inside the grains, stress concentrations in the pores are reduced, and no significant local plastic deformation occurs around these voids, this effect is comparable to the benefit of a physical void closure. In this case, the fracture generally spreads through the material matrix rather than through the interface. The local plastic deformation behavior at the interface is directly related to the distance between the void and the migrated grain boundary [21, 22].

GBM lowers crack propagation along the initial bonding interface, a larger GBM ratio improves the shear strength of the joint. In addition, impact toughness is also generally increased with GBM. The relationship between GBM ratio and impact toughness of the joints can be seen in Figure 11 for different holding times (a) and bonding temperatures (b) [19]. However, even if higher temperatures enhance GBM, excessive temperatures, instead of enhancing mechanical properties, will result in grain coarsening and will ultimately reduce the overall mechanical properties as well as the impact toughness on the joint, irrespective of GBM [19].

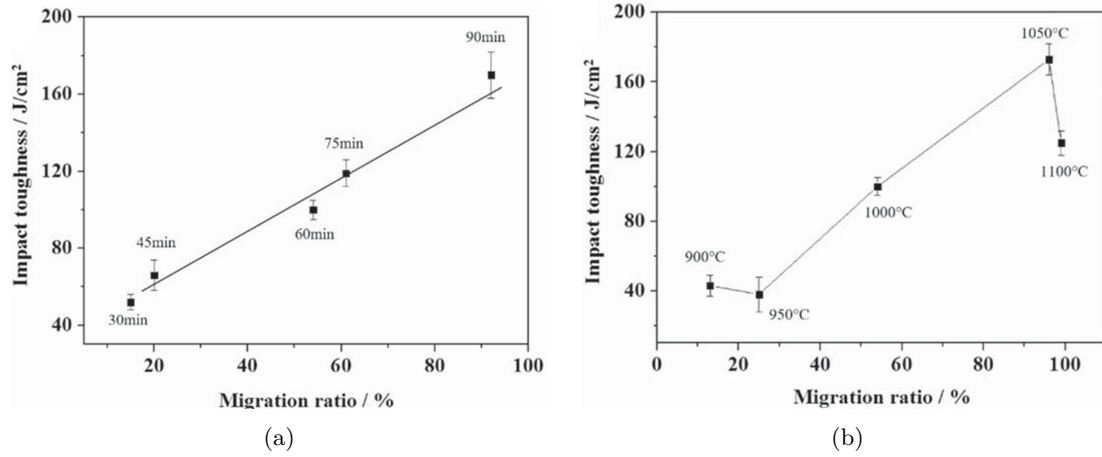


Figure 11: Impact toughness as a function of GBM ratio for different bonding durations (a) and temperatures (b). [19]

### 2.1.3 Important parameters that influence diffusion bonding

Many parameters significantly affect the bonding quality, and these are interlinked. The initial void size is directly related to the applied pressure, which affects the diffusion behavior. In addition, increasing the applied pressure also enhances the plastic deformation that regions that come into contact exhibit, improving the bonding ratio [14].

Moreover, temperature controls atomic diffusion rate, which is crucial for material transport or atomic mobility over the interface. Additionally, process stages are also dependent on the temperature, at lower temperatures, plastic deformation occurs, whereas at higher temperatures, creep and diffusion dominant processes take over [14, 21].

Bonding ratios also grow progressively as the holding time gets longer. But after a certain time, this growth slows down, suggesting that bonding time enhances bonding to a certain extent [4, 14].

When high bonding pressure and low bonding temperature are applied, bonding is mainly influenced by the plastic deformation mechanism. However, when applying lower bonding pressure and higher bonding temperature for a longer holding time, creep and diffusion mechanisms are the driving forces [15].

Apart from these three main parameters, many other crucial factors influence the bonding ratio and the joint's quality. Some of these include; induced dislocation density, surface roughness, and grain size. All these parameters, together with the required temperature, pressure, and holding time, are analyzed in this section [12, 5, 23].

#### Required temperature

Since temperature directly affects atomic diffusion, deformation behavior, and microstructural evolution in the interface, it is one of the most important factors in the bonding process. Elevated temperatures facilitate creep mechanisms and enhance atomic mobility or diffusion after plastic deformation [12]. How temperature increases the bonding ratio as a function of different applied pressures can be seen in the simulation shown in Figure 12 [24].

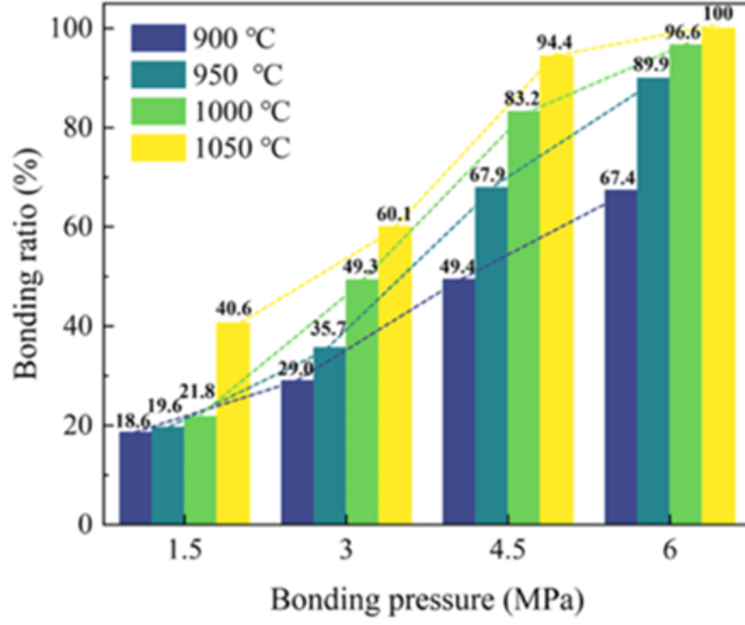


Figure 12: Bonding ratio as a function of bonding pressure, for different temperatures. [14]

Atoms can only diffuse from one side of the component into the other when the temperature reaches a certain level, usually between 0.6 and 0.8 times the material's melting point ( $T_m$ ) [24]. Therefore, atomic diffusion across the interface will only happen if the bonding temperature is sufficiently high. Higher temperatures significantly increase the number of atoms crossing the interface, and this results in a thicker interfacial region (higher diffusion distance from the interface to the migrated atoms). However, if the bonding temperature is insufficient, diffusion will be insignificant. [24, 12].

As the temperature gets higher, the plastic deformation stage also gets shorter. This reduction can be explained by the fact that material becomes more vulnerable to plastic deformation because the material's yield strength decreases at higher temperatures, promoting faster plastic deformation and therefore reducing the duration of this stage. Therefore, the transition from the plastic deformation stage to the creep mechanism stage happens. This stage is defined by grain boundary sliding and dislocation climbing. Diffusion mechanisms take over at even higher temperatures, while the contribution of bulk deformation (plastic deformation and creep) drops. At these high temperatures, thermal activation energy increases and materials lattice resistance reduces, enhancing atomic mobility and the ability of dislocations to overcome lattice resistance and slip, resulting in an increase in the diffusion ratio [14].

Higher temperatures promote larger contact area expansion and therefore enhance void closure. However, even after long bonding durations, insufficient temperatures can lead to partial void closure [14]. Both atomic diffusion and creep rate are the two principal mechanisms contributing to void closure, and these are temperature promoted processes [21, 14]. The effect of the bonding temperature in the void closure (void length reduction) can be seen in the simulation results shown in Figure 13 [14].

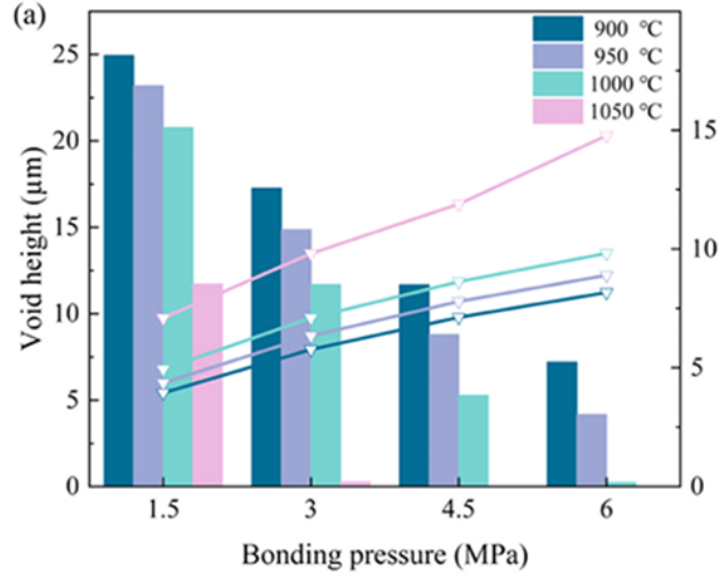


Figure 13: Void height as a function of bonding pressure, for different temperatures. [14]

At a microstructural level, temperature controls recrystallization and grain evolution. Under constant pressure and constant holding time, raising the temperature facilitates grain growth and the formation of a more uniform interface structure. It has been seen that at 900 °C, the grain size remains small when using 304 austenitic stainless steel [14]. Here, since the bonding temperature is not enough for recrystallization, the main aspect of microstructural evolution is that dislocations rearrange and subgrains are formed. As the bonding temperature increases, the grain size shows a coarsening tendency. When the temperature reaches 950°C, deformation storage energy is released, causing subgrains to merge and increasing recrystallization. When the temperature rises to 1000°C, the interface structure becomes uniform. After recrystallization happens, grains grow fast (grain coarsening). In addition, GBM accelerates as the temperature rises due to an increase in bulk diffusion [14]. Apart from this, grain growth provides sufficient GB velocity, promoting GBM. At elevated temperatures, fast dissipation of stored energy happens, causing insufficient nucleation driving force and increasing grain size [21, 14]. Microstructure evolution at 900°C, 950°C, and 1000 °C for 304 austenitic stainless steel can be seen in Figure 14, it also shows that GBM happens at higher temperatures (950°C and 1000°C in this case).

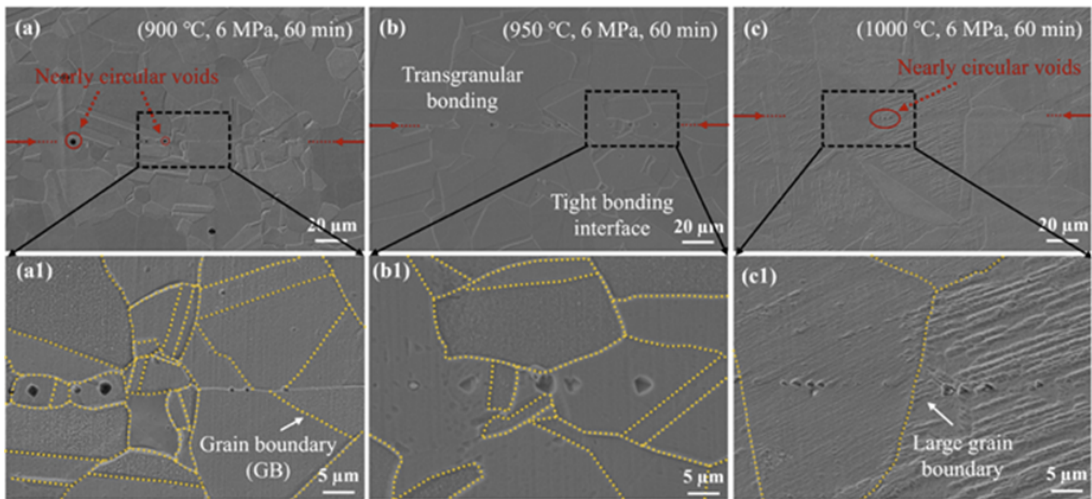


Figure 14: Microstructure evolution at 900°C (a-a1), 950°C (b-b1) and 1000 °C (c-c1) for 304 austenitic stainless steel. [14]

As mentioned, void closure, GBM, and Recrystallization (Rx) are temperature dependent mechanisms, and the influence of temperature on these parameters can be seen in Figure 15 [21].

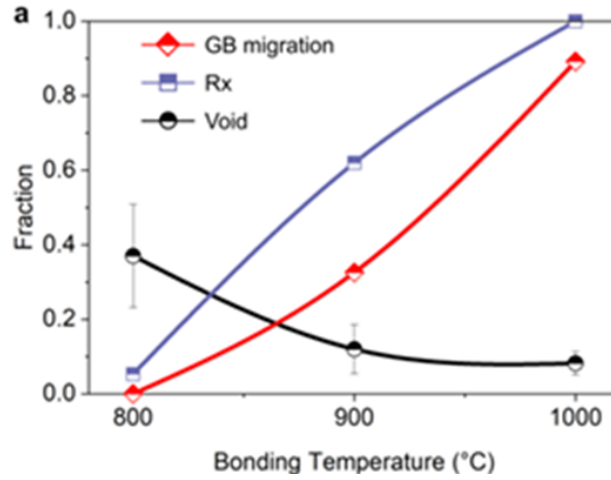


Figure 15: GBM, Recrystallization and void closure as a function of temperature. [21]

As said above, higher temperatures promote void closure and GBM, which will significantly improve joint quality. However, at the same time, it also promotes grain coarsening, which reduces yield strength [14]. According to the Hall-Petch relationship, tensile strength decreases with increasing grain size because larger grains have fewer grain boundaries that impede dislocation movement [19]. Coarse grains experience significant plastic deformation under low stress, eventually causing deterioration and reducing joint performance [14]. Therefore, the final mechanical properties of the bonded joint are directly influenced by the bonding temperature [14].

As shown in Figure 16, for a 316L stainless steel material, at first, when increasing bonding temperature, tensile strength also increases, but beyond 1000°C, it starts to drop, due to grain coarsening. However, ductility typically increases with temperature due to GBM. Ductility is represented by the elongation ratio. Moreover, there is no significant improvement in the bonding ratio above 1000°C [19]. All these phenomena can be seen in Figure 16.

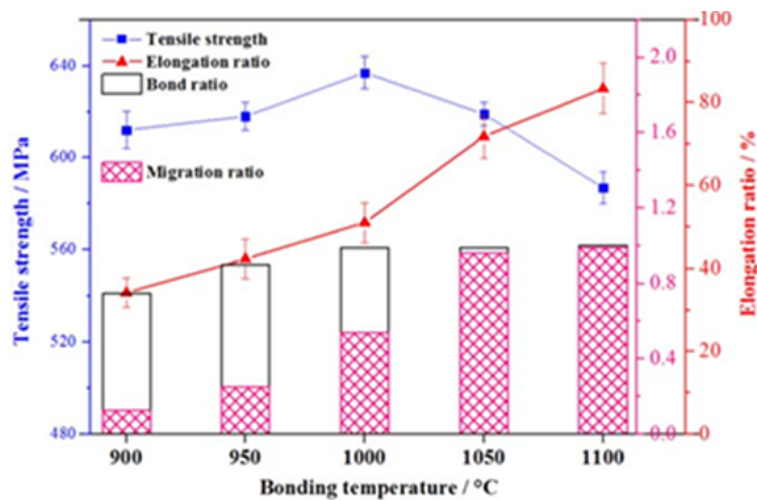


Figure 16: Tensile strength, Elongation ratio, Bonding ratio, and GBM as a function of bonding temperature. [19]



In conclusion, an adequately selected bonding temperature, typically between 0.6 and 0.8 times the  $T_m$  of the bonding material, is necessary for efficient diffusion bonding. To promote void closure and GBM, without excessive grain coarsening that can negatively impact mechanical properties [24, 19].

### Applied Pressure

When bonding pressure increases, atoms can more easily migrate through the interface, generally from the neck region towards the void. As the pressure increases, the tip of the void tends to deform in the opposite direction to that of the applied axial pressure. As a result, the axial deformation rate increases and the void's height shrinks. [14])

The optimal combination of pressure and temperature can lead to an effective void closure. Figure 17 shows a simulation of the effect of different pressure and temperature combinations in the void closure and stress distributions one hour into the bonding process at temperatures between 950 and 1050°C with bonding pressures between 1.5 and 6 MPa (for 304 stainless steel material). Once pressure is applied, convex regions (tips) first come into contact and then these experience plastic deformation. These regions exhibit the highest stresses at the initial contact. Figure 17 shows that stress distribution lowers in the opposite direction of the applied axial pressure [14].

Both the bonding pressure and temperature are crucial parameters that enhance the contact area at the interface. However, as can be seen in Figure 17, the decrease of stress concentrations in the tips is more significant, and stress distribution gets more pronounced when increasing temperature rather than increasing pressure. This suggests that pore closure happens faster when the temperature increases rather than when the pressure increases. As an example, a more significant stress distribution can be observed while maintaining a constant pressure at 3MPa but increasing the temperature from 950°C to 1050°C 17(a2-b2-c2) rather than having a constant temperature at 950°C and increasing the pressure from 3MPa to 6MPa 17(a2-a3-a4).

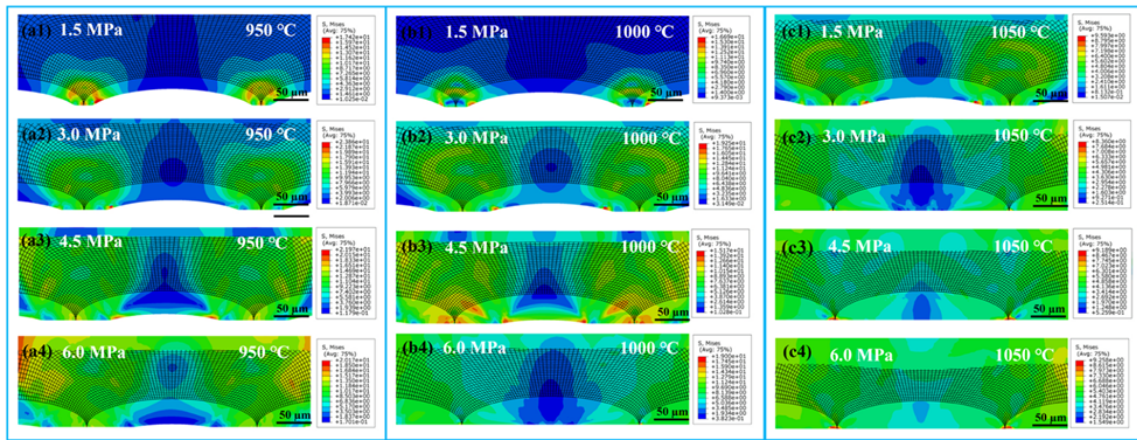


Figure 17: Simulation of stress distribution during void closure, under different bonding pressures (between 1.5 and 6 MP) and temperatures (between 900 and 1050°C). [14]

Apart from this, the simulation in Figure 18 represents the axial deformation rate and interface displacement in the contact area after 60 minutes of holding time for 1.5 MPa, 3 MPa, 4.5 MPa, and 6 MPa pressures at 950°C, 1000°C, and 1050°C (for 304 stainless steel material) [14]. It can be seen that the tip of the void exhibits the lowest displacement, while the neck shows the highest displacement toward the interface line, while the bonding zone grows, closing the voids.

Increasing both the applied pressure and bonding temperature enhances displacement at the interface and variation in the void height. However, higher interface displacement and axial deformation rate are observed when increasing the bonding temperature from 950°C to 1050°C (at 3MPa) rather than increasing the bonding pressure from 1.5MPa to 6MPa (at 950°C) [14].



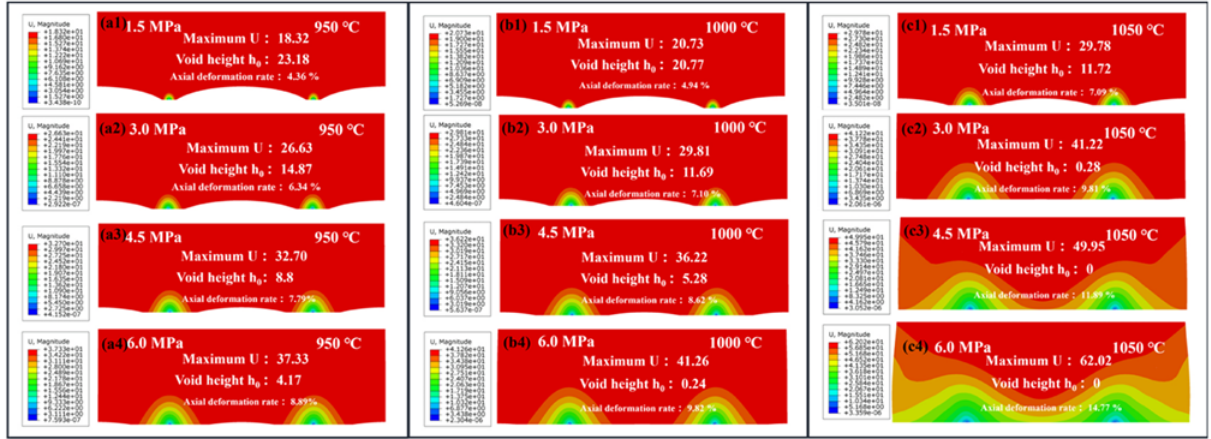


Figure 18: Simulation of displacement magnitude at the interface during void closure, under different bonding pressures (between 1.5 and 6 MP) and temperatures (between 900 and 1050°C). [14]

### Holding time

Holding time is another critical parameter in the diffusion bonding process, as it directly affects joint properties, void closure, bonding ratio evolution, diffusion mechanisms, and microstructural evolution at the interface [21].

The progressive improvement of bonding is one of the benefits of long holding time. Longer holding times allow atoms more time for diffusion, and therefore the distance between neighboring interfacial voids rises, expanding the bonded area and increasing the bonding ratio. Later, longer holding times will also result in the gradual elimination of the interfacial pores [4].

Figure 19 shows an example of the evolution of void closure at the interface over different bonding times. Initially, in (Figure 19(b)) just after 15 minutes of bonding time, a bonding ratio of 44.1% was measured, which increased close to 100% after 75 minutes, Figure 19(j) [4].

The interface in Figure 19 shows large voids after short holding times due to insufficient bonding time. These pores are evenly spread and have a semi-elliptical shape (Figure 19(a-b)). These voids shrink in size and change their shape as the holding time increases (Figure 19 (e-f) [4, 21]. At the same time, longer holding times also show an increase in distance between the pores, suggesting that the contact area and bonding ratio improved over time [4]. However, this evolution is not linear over time. During the initial stages, the contact area is small, resulting in higher stress concentrations at the tips, promoting deformation driven bonding. The contact area increases and the stress concentrations decrease over time, therefore, the effect of the deformation driven bonding is reduced. At this stage, diffusion mechanisms become dominant, and the bonding ratio speed decreases [15, 4].

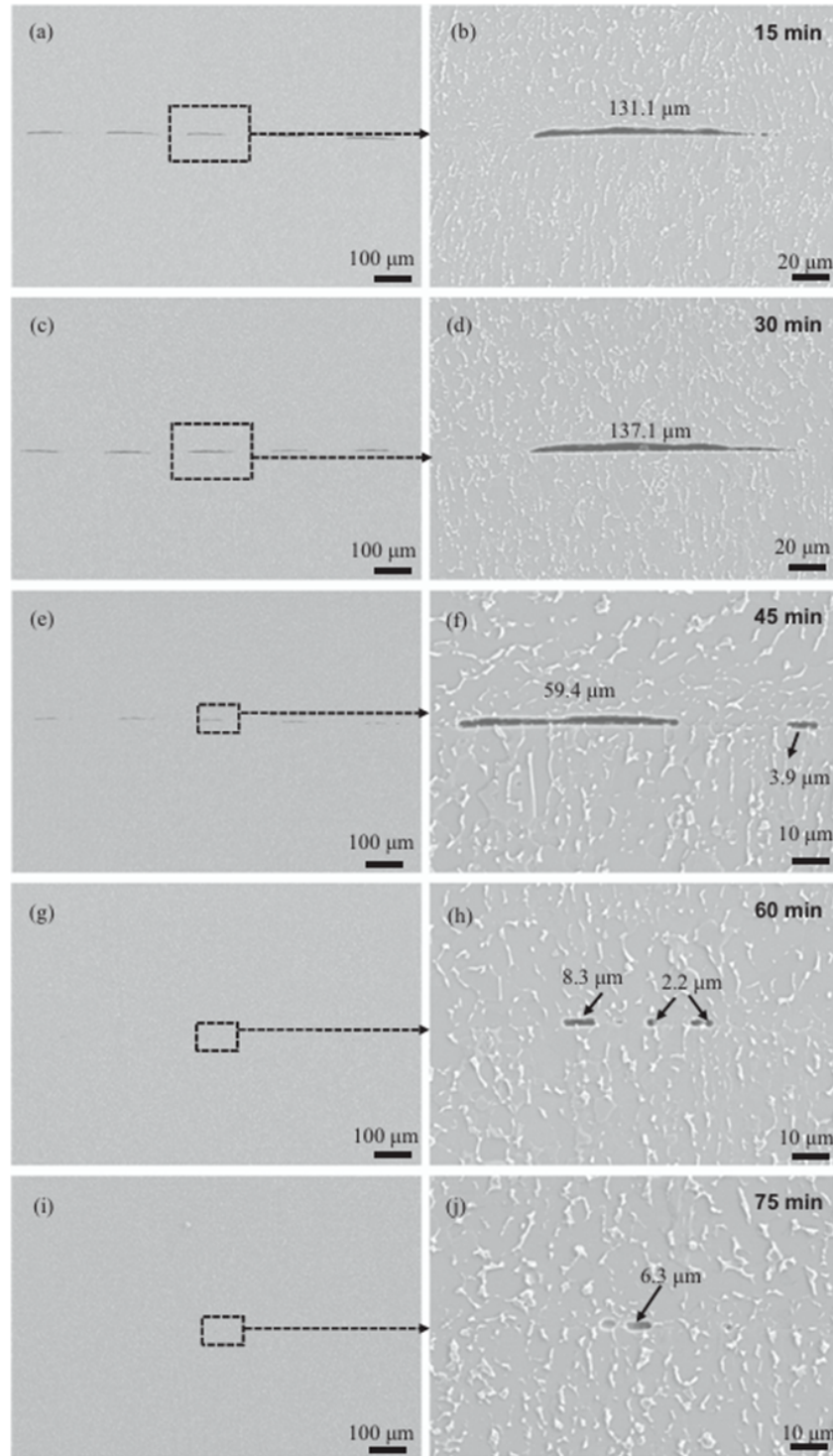


Figure 19: Void closure evolution at the interface during bonding at different bonding times (a,b)-15min, (c,d)-30min, (e,f)-45min, (g,h)-60min, (i,j)-75min. [4]

The growth of the bonding ratio is nonlinearly related to the holding time because of the power law creep exponent ( $n$ , in Equation 2), and the increase of the bonding ratio and the decrease in the void width change at a faster rate at the initial stage. [15, 4]. In addition, microstructural evolution is also influenced by the holding time. GBM is a time-dependent kinetic and tends to happen at longer holding times [4].

Both the void size reduction and GBM are promoted by increasing temperature (Figure 15) and bonding time (Figure 20). But the effect of bonding time on recrystallization (Rx) is minimal compared with that on GBM and void closure. As previously seen, recrystallization is mainly influenced by the temperature (Figure 15) rather than by the bonding time (Figure 20) [21].

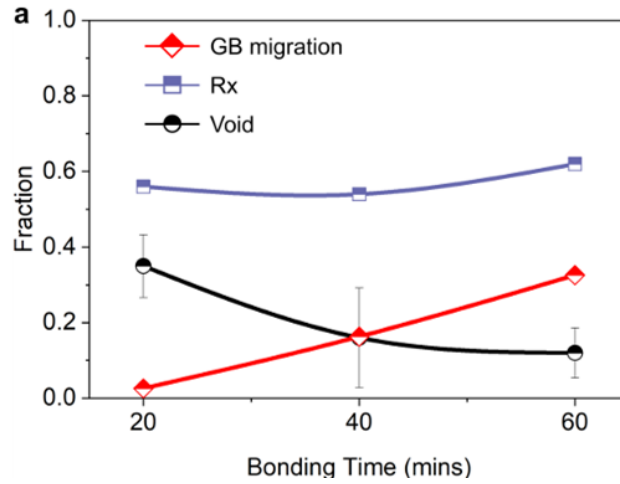


Figure 20: GBM, Recrystallization and void closure as a function of time. [21]

However, extended holding times at too high temperatures, can also have negative consequences. For example, too long holding times may lead to grain coarsening, which subsequently can lower the material's tensile strength. This highlights the importance of an optimal holding time that makes a tradeoff between the enhanced bonding ratio and the microstructural stability [20]. The effects of the holding time on the bonded materials' properties are shown in Figure 21.

From a mechanical property's perspective, increasing holding time raises joint strength and improves bonding quality. The tensile strength can approach that of the base material when the bonding ratio is high enough. Furthermore, increasing the holding time also promotes GBM, which can improve ductility (reflected by higher elongation values in Figure 21) [19].

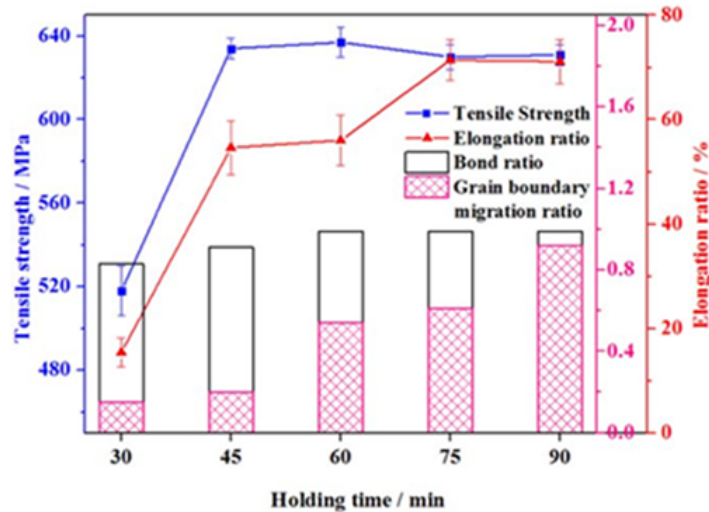


Figure 21: Tensile strength, Elongation ratio, Bonding ratio, and GBM as a function of bonding time. [19]

In conclusion, holding time drives the change from a deformation controlled to a diffusion driven bonding stage, promotes GBM and void closure, and enhances the joints' mechanical properties. In general, having longer holding times results in a better bonding quality, but if it is longer than necessary at a too high bonding temperature, it can result in grain coarsening and loss of mechanical strength, therefore, an efficient holding time is needed [4, 15, 19].

### Surface roughness

Surface roughness is a key factor influencing the diffusion bonding process, and it has a significant influence at the interface at the first stage of the bonding process. When the surface roughness is high, larger interfacial pores are formed during the initial contact area, negatively affecting the bonding quality by reducing the effective contact area at the interface. On the other hand, when the contact surfaces are smooth, larger ridge to ridge contact areas are formed, and therefore the pores at the interface are smaller, enhancing the bonding ratio. The difference between a smooth and rough surface is represented in Figure 22 [12, 20].

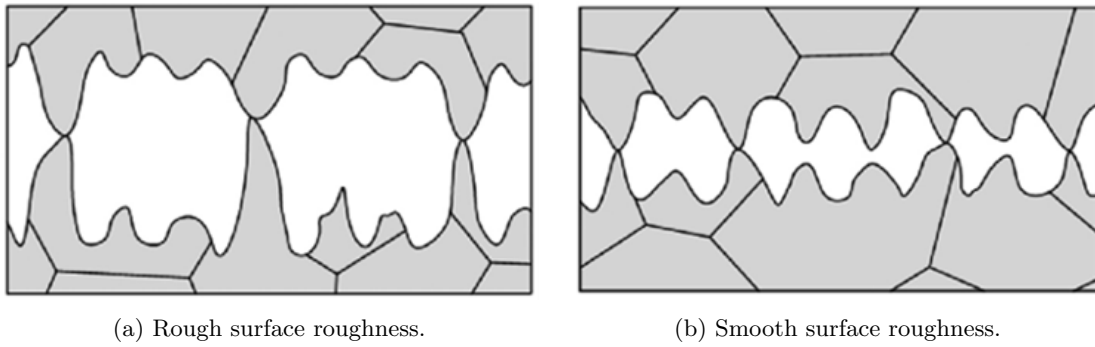


Figure 22: Surface finish comparison. [20]

There are three main phases in the bonding process where surface roughness is present. First, when the surfaces come into contact, initial pores are formed, and the surfaces deform under the applied bonding pressure and stress, increasing the contact area. In the second stage, as the temperature rises, the contact areas undergo significant deformations, and interfacial gaps disappear, resulting in an almost full surface contact. Finally, atomic diffusion takes place, further closing the residual pores and allowing a metallurgical joint [24].

Surface roughness does not have a big impact on the deformation mechanism, but it strongly affects the initial effective contact area, the initial stress values in the tips, and subsequently the diffusion stages. Smoother surfaces provide a larger effective contact area, which enhances atomic diffusion and therefore rises bonding ratio. Therefore, under the same bonding conditions, the specimens with a finer surface roughness resulted in smaller interfacial voids and larger bonded areas [12]. Figure 23 shows the difference in void closure and bonding ratio at the interface for different surface roughness conditions for a TC4 alloy, bonded at 800°C with a force of 10 MPa for 15 minutes. It can be seen how the contact area increases and the void size reduces by reducing the initial surface roughness of the components [15].

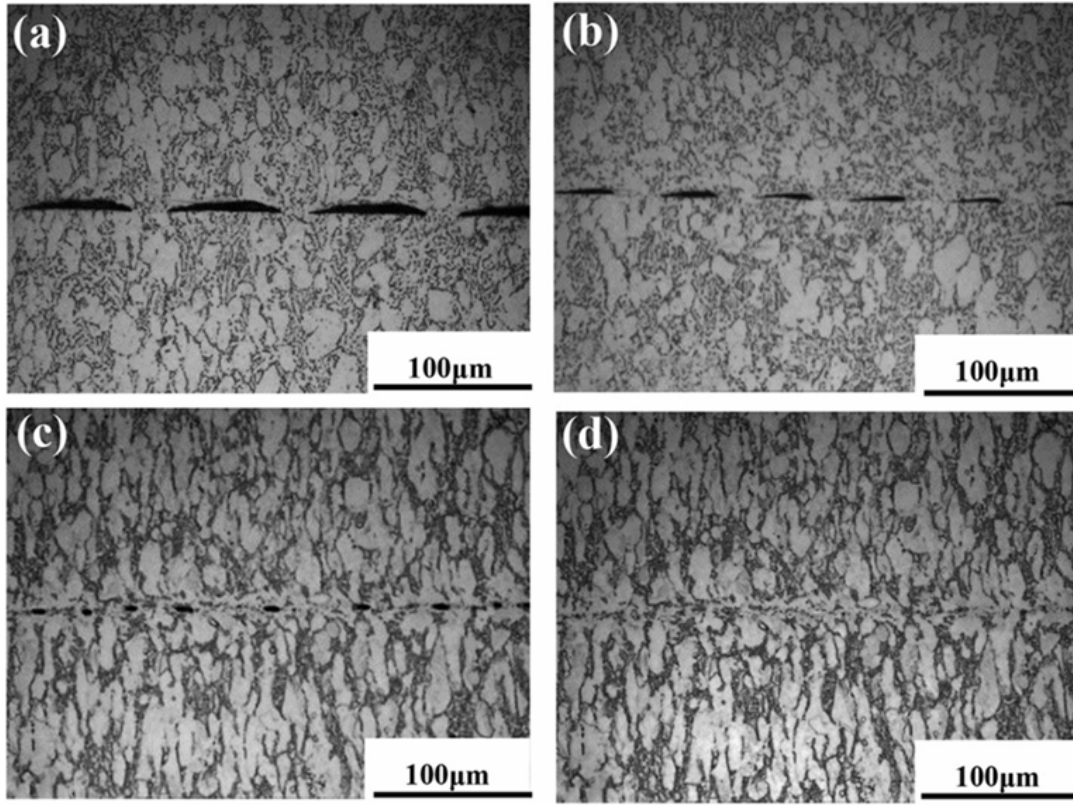


Figure 23: Diffusion bonded joints under different surface roughness, at 800 °C and 10 MPa during 15 minutes. (a)-3.2 $\mu$ m, (b)-1.6 $\mu$ m, (c)-0.8 $\mu$ m, (d)-0.4 $\mu$ m. [15]

In addition to the bonding ratio and void width, the microstructure and the GBM mechanisms are also influenced by the surface roughness. Figure 24 shows the interface evolution and GBM over time, for smoother Figure 24 (a,c,e,g) and rougher Figure 24 (b,d,f,h) surface finishes. At short bonding times (a,b), a smoother surface (a) not only shows a straighter interface (less porosity), but localized GBM can also be seen, whereas a rougher surface contact (b) still has significantly bigger interfacial voids. [20].

As the holding time increases, smoother surfaces show a faster increase in the GBM mechanism, while a rougher surface shows a slower migration ratio. At intermediate holding times, GBM becomes more noticeable for both situations (Figure 24 (e,f)), but still remains higher for smoother surfaces [20].

For smoother surfaces, GBM mostly takes place at triple junctions, where the decrease in GB energy drives the formation of these sharp structures. On the other hand, rougher surfaces show a strain induced GBM mechanism, which results in spherical cap formations caused by uneven asperities at the surfaces [20].



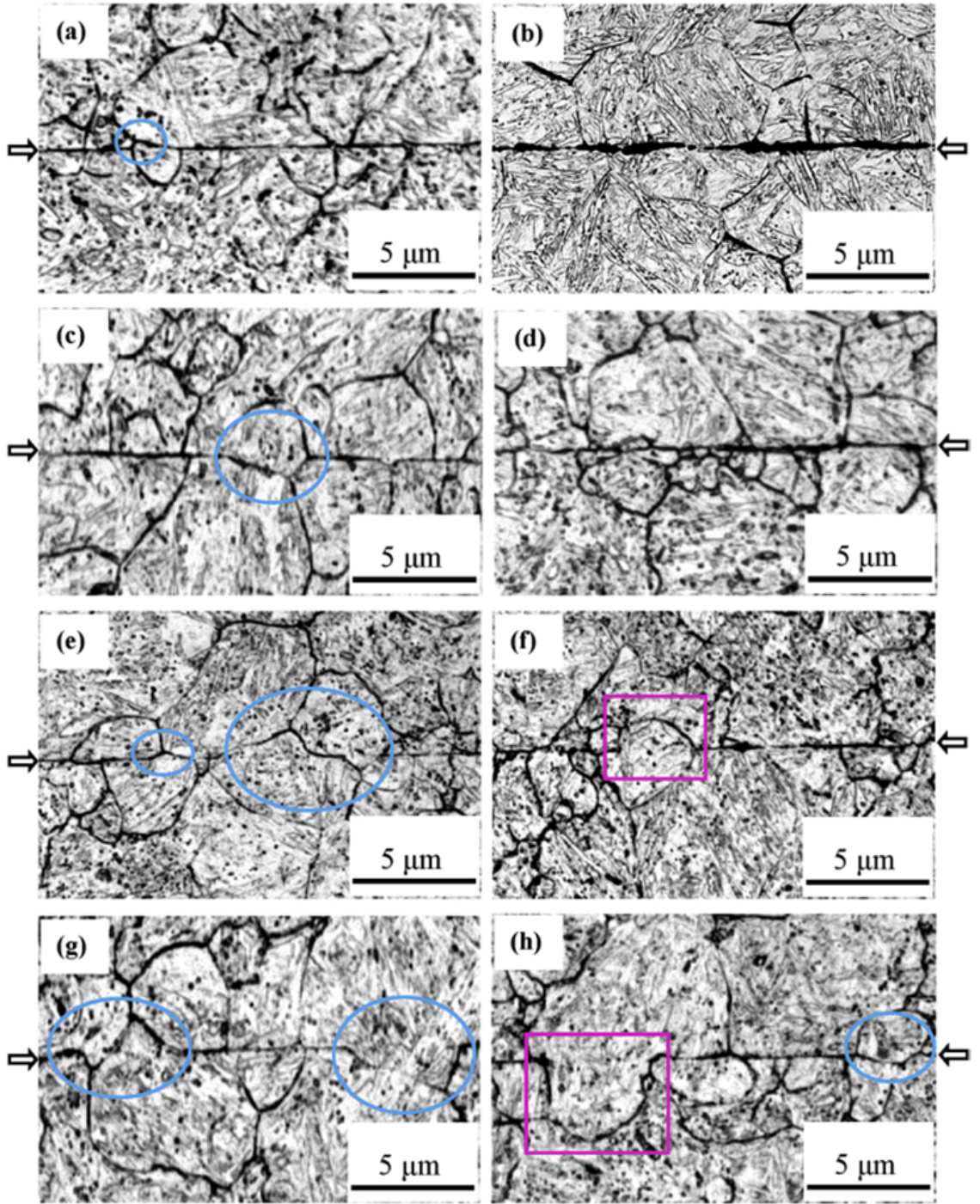


Figure 24: GBM evolution for joints bonded at 980 °C and 15MPa, with a smooth surface finish (a)-15min, (c)-25min, (e)-30min, (g)-45min and a rough surface finish (b)-15min, (d)-25min, (f)-30min, (h)-45min.[20]

Moreover, Figure 25 illustrates how the surface roughness quantitatively affects GBM across different mechanisms (triple junction migration and strain induced migration). It demonstrates that smoother surfaces promote higher GBM because of enhanced mobility at the grain boundary interface [20].

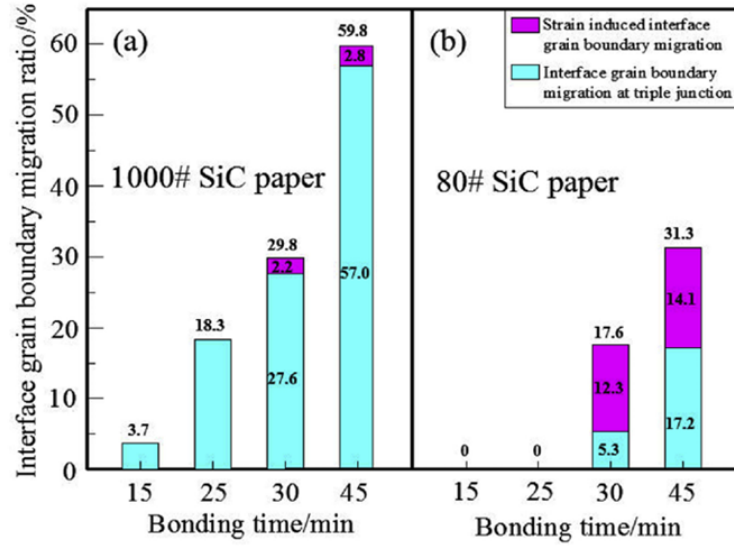


Figure 25: GBM ratio as a function of bonding time, for a rough surface finish (1000# SiC paper) and a smooth surface finish (80# SiC paper). [20]

In summary, surface roughness plays a key role in diffusion bonding. It influences the initial contact area and void formation. Smoother surfaces provide more optimal contact conditions that enhance atomic diffusion and promote greater GBM ratio, mainly driven by migration at triple junctions, and subsequently leading to a greater bonding ratio and a better overall joint quality [12, 20, 15].

### Geometrically necessary dislocation density

Geometrically necessary dislocations (GND) are defects in the material that accommodate deformation gradients [25]. These dislocations are introduced by plastically deforming the material through different processes (such as cold rolling or compression) before bonding. As deformation increases, the dislocation density also increases [5]. Figure 26 shows the difference in dislocation density between an undeformed, a 5% deformed, and a 10% deformed material [26].

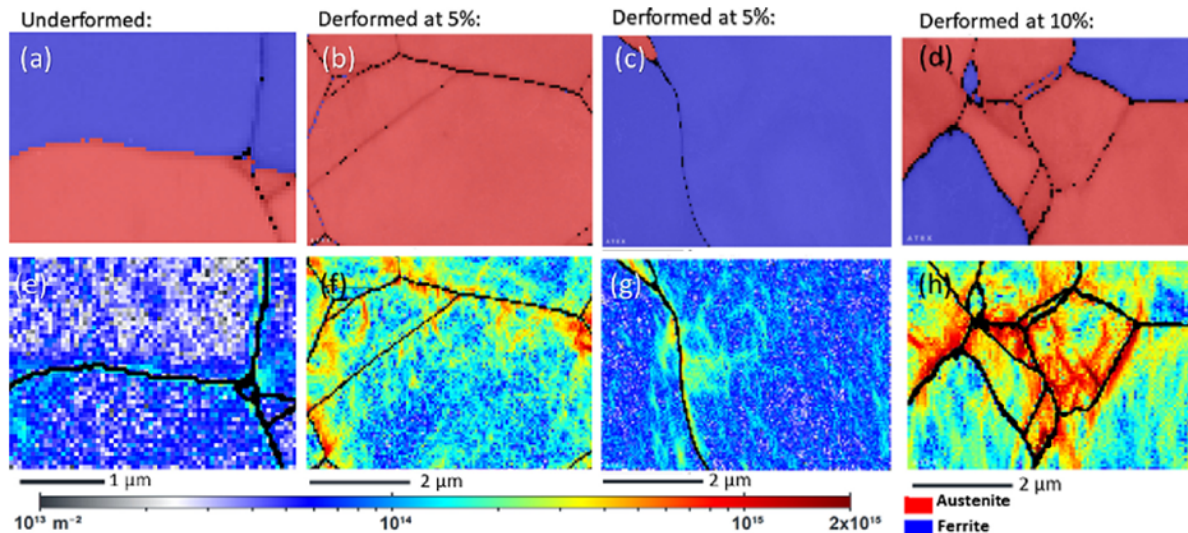


Figure 26: EBSD map for undeformed, 5% deformed, and 10% deformed ((a,b,c,d) represents austenite and ferrite (e,f,g,h) represents GND map.) [26]

When it comes to diffusion bonding, dislocations provide short circuit diffusion paths, enhancing atomic mobility at the interface and thereby accelerating the bonding ratio. Dislocations act like channels for pipe diffusion, acting as “pipes” through which atoms can move and migrate. On the other hand, lattice diffusion typically occurs through vacancies or interstitial sites. In general, pipe diffusion is faster than lattice diffusion. Therefore, dislocations not only increase the bonding ratio but these also extend the diffusion distance at the interface, strengthening the joint. As an example, it has been seen that under equal bonding conditions but by increasing the deformation rate from 0% to 20%, the bonding ratio rises from 56.1% to 81.1%, for  $Cr_{0.8}FeMn_{1.3}Ni_{1.3}$  HEA/216 SS diffusion bonded joints. [5].

When the material is undeformed, the diffusion mechanism in diffusion bonding is mainly driven by lattice diffusion. However, pipe diffusion (diffusion through dislocation channels) becomes the dominant mechanism after a certain level of deformation. As can be seen in Figure 27, the contribution of pipe diffusion grows with increasing deformation rate, whereas at the same time the lattice diffusion contribution lowers correspondingly. This highlights the significant influence that dislocations have in facilitating atomic movement and migration [5].

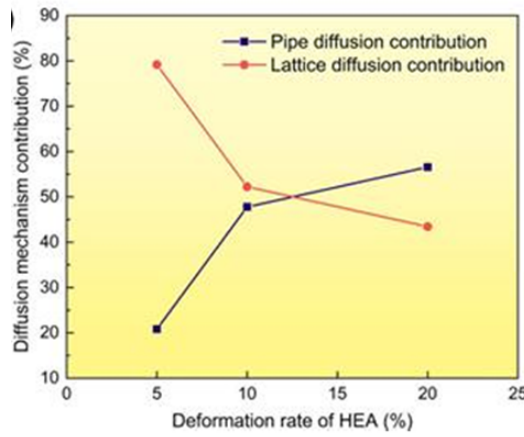


Figure 27: Diffusion mechanism contribution as a function of deformation rate of a HEA. [5]

Additionally, the curve in Figure 28 represents the relationship between the dislocation density and the diffusion distance, in this case for a high entropy alloys (HEA) material. It demonstrates that diffusion distance increases significantly with dislocation density, confirming the strong effect of dislocations in atomic migration from one component to another across the interface, while strengthening the joint [5].

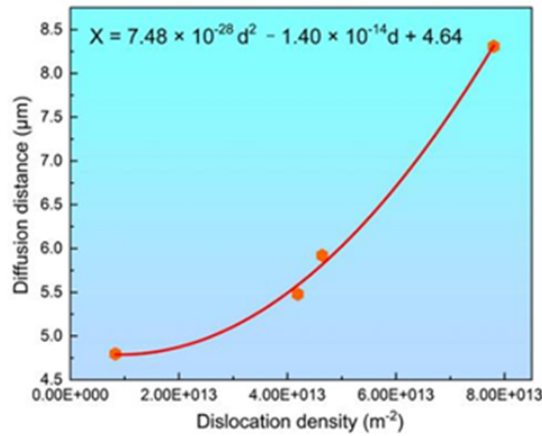


Figure 28: Diffusion distance as a function of dislocation density. [5]



As discussed previously, a minimum energy is required for atoms to overcome energy barriers. However, these dislocations reduce the needed activation energy, enabling atoms to bypass conventional lattice diffusion paths. Thereby, increasing the atomic mobility, increasing the bonding ratio and the joint quality [5, 14]. Dislocation density also has a direct relationship to the mechanical properties of the interface. Both the tensile strength and elongation were improved when introducing higher dislocation densities. [5]

The fracture behavior at the interface is also influenced by the dislocation densities. Increasing dislocation density promotes a more ductile fracture. For an undeformed material, the fracture surface exhibited shallow, small dimples, indicating a limited degree of plastic deformation at the interface and therefore a more brittle fracture. On the other hand, samples bonded with 20% of deformation showed deeper and larger dimples, indicating a more ductile fracture and suggesting a higher plastic deformation at the fracture and a stronger overall interfacial bonding. In addition, it was also found that as the dislocation density ratio increased, joint hardness approached that of the base material, further indicating an improved bonding quality. This fracture distinction between deformed and undeformed samples is shown in Figure 29 [5].

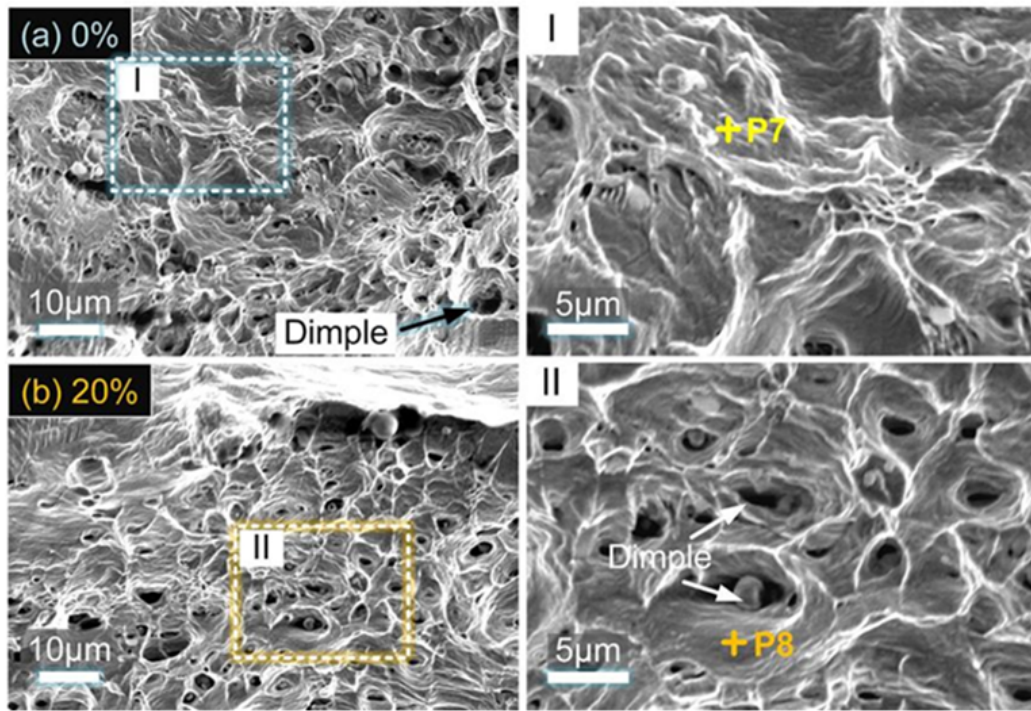


Figure 29: Fracture for an undeformed (a) and a 20% deformed material. [5]

### Grain size

Grain size is another main factor influencing the diffusion bonding process through its effect on diffusion mechanisms and mechanical properties.

An optimal grain size facilitates both void shrinkage or closure and GBM in the bonding interface. It is known that a finer grain size material enhances faster GBM and also accelerates interfacial void removal, improving bonding quality. As coarser grain sized material has less grain boundary density, and these materials show slower atomic diffusion and might not bond well under the same pressure, temperature, and holding time conditions [23].

The main reason behind this behavior is the materials' grain boundary density. These boundaries provide short circuit diffusion paths, supporting atomic transport across the interface. In consequence, atomic diffusion happens faster for the same temperatures, allowing more efficient material transport and bonding ratio. Diffusion through grain boundaries is a type of pipe diffusion, this happens along the crystalline defects same as with dislocations. Grain boundaries act like diffusion paths due to their disordered structure allowing atoms to move faster than through perfect crystal lattices [5, 15, 23, 27]. The grain size and the bonding ratio difference between a fine grain and a coarse grain size material are schematically represented in Figure 30 [23, 27].

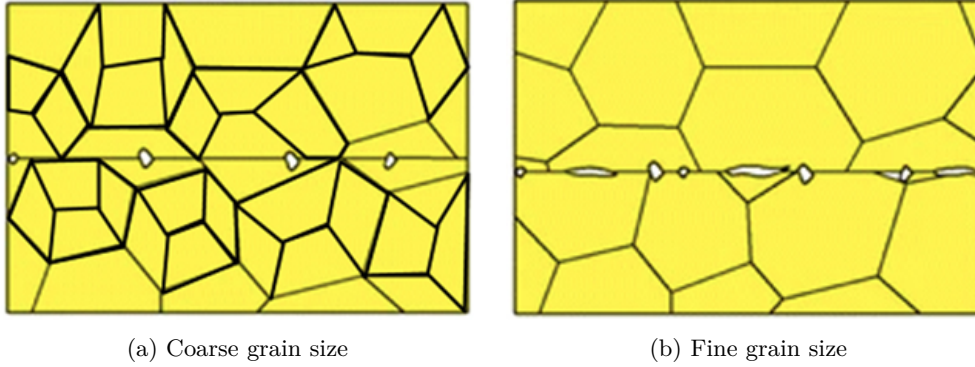


Figure 30: Illustration of different grain sized materials. [23]

In addition, the mechanical properties of the joint and the material itself are also strongly influenced by the grain size. As the grain size grows, the yield strength lowers because of fewer grain boundaries in the material or interface that can prevent dislocation movement. In general, finer grain size materials show a higher strength since these materials are more resistant to dislocation movement because of a higher grain boundary density [19].

### 3 Experimental method

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As has been seen, there are many factors that influence a diffusion bonded joint's quality. In this study, different experiments have been conducted to evaluate the effect of these key factors on the joint quality, using Alfa Laval's representative parameters. The analyzed factors include applied pressure, holding time, surface roughness, and deformation (induced dislocations). Additionally, factor interactions were also studied by combining different bonding cycle times with deformed components, as well as the effect of varying surface roughness conditions for deformed specimens.

Additionally, grain size evolution during bonding was analyzed for two materials with different starting grain sizes. Apart from this, dislocation density evolution in deformed materials under different heating rates was also investigated using Electron Backscatter Diffraction (EBSD) in a Scanning Electron Microscope (SEM). To conduct diffusion bonding experiments, four steps were followed: sample preparation, diffusion bonding, metallography sample preparation and characterization of results.

Due to confidentiality, exact values of some key parameters are not deliberately given. Nevertheless, this should not affect the outcome of the conclusions from this report.

#### 3.1 Development of sample preparation for DB

Stainless steel 316L plates were used as the base material for this study. The material was received in laminates, with a thickness of 3.7mm, and later these were cut into the required dimensions. For each diffusion bonding analysis, two components were used: the upper and the lower component. As can be seen in Figure 31, these two components are later placed on top of each other before being introduced to the production furnace. To ensure full contact between these two components, the bottom component is cut in slightly larger dimensions (15x15mm) than the upper component (10x10mm).

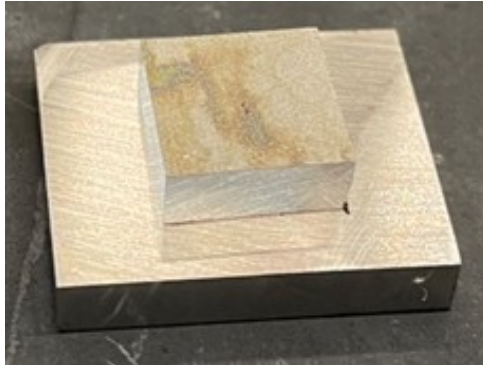


Figure 31: Sample example. [28]

Selected components were then plastically deformed using a hydraulic press (Figure 32) to introduce a controlled density of dislocations. Based on the desired dislocation density, different forces were applied, corresponding to different levels of deformation. Four deformation conditions were studied: non-deformed (ND), low deformation rate (LDR), medium deformation rate (MDR), and high deformation rate (HDR), corresponding to the amount of compressed height of the samples with the manual hydraulic press.



Figure 32: Hydraulic press. [29]

Once the components were deformed, these were flattened using a surface polishing machine to eliminate any tilt that could affect the uniformity of the samples. The components were subsequently mounted and polished to achieve the target surface roughness, three different conditions were categorized: rough surface (RS), as received surface (ARS), and smooth surface (SS). Afterwards, surface roughness was verified using a profilometer. Six different measurements were taken per component, and the average measurement was calculated to confirm that the desired surface finish was achieved. Finally, the components were cleaned ultrasonically in acetone to remove residuals from the surface.

### 3.2 Experimental set-up procedure

Once samples were prepared, pairs were put together and loaded with the desired bonding pressure. Afterwards, these were placed in a vacuum furnace. The vacuum furnace prevents the components from oxidation at elevated temperatures. Once the samples were placed in the furnace, a simple bonding cycle was used for lab-scale testing. Here, the samples were heated at a specific rate, and when the target temperature was achieved, the samples were held at this temperature for a specific holding time. Once this time was over, the samples were cooled at a specific cooling rate. Finally, once the samples were cooled and taken from the furnace, the bonding pressure was released. This process is schematically represented in Figure 33.

It is worth noting that this setup differs from a conventional diffusion bonding process (Figure 4). Here, the samples are loaded from the beginning, before heating, whereas in the conventional diffusion furnaces, bonding pressure is applied once the bonding temperature has been achieved.

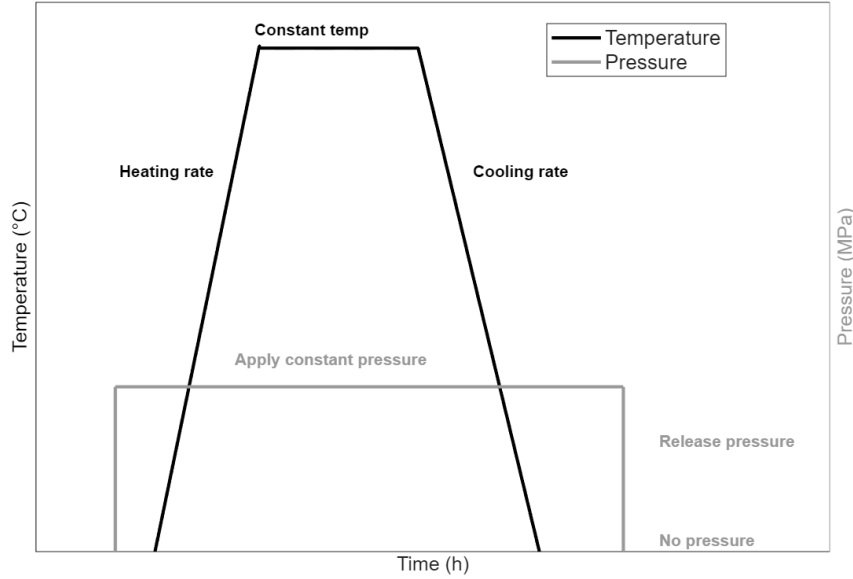


Figure 33: Furnace schematic representation, Pressure (MPa) and Temperature (°C) vs Time.

### 3.3 Metallography sample preparation and characterization of results

Once the components were bonded, these were prepared for characterization. First, the size of the upper component was measured to define the nominal bonding area. After this, the bonded assemblies were cross sectioned. The full cross section of the bonded components, together with the half cross section of the same components, were mounted together. These components were progressively polished until the oxide polishing suspension (OPS) finishing was obtained.

An optical microscope [28] was used for the characterization of the samples. With this microscope, the following features were measured: length of the pores, grain boundaries' migration length, grain size, and the length of the unbonded corner regions.

From these measurements, the effective contact area was determined by considering the length of the unbonded corners relative to the total upper component dimensions, from which the effectively applied bonding pressure was subsequently calculated. These are the quality assessment parameters used in this study:

Porosity ratio:

Porosity ratio was calculated by considering the entire cross section of the components using equation 7 explained in the previous Section 2.1.2. Porosity ratio is used to evaluate the quality of the joints, where a lower porosity ratio corresponds to a better bonded joint, as demonstrated in the previously.

$$\text{Porosity rate (\%)} = \frac{\text{Total void length } (L_1 + L_2 + L_3 + \dots + L_n)}{\text{Total interface length } (L)} \times 100\% \quad (7)$$

The following cases were analyzed:

- ND (not deformed), LDR (Low deformation rate), MDR (medium deformation rate), and HDR (high deformation rate) components under different applied pressures
- ND and MDR components under different surface roughness conditions
- ND and MDR components under different cycles

#### Average pore size:

Average pore size was calculated across the entire cross section of the components using the Equation 8, previously explained in the Section 2.1.2. The same set of cases was analyzed, including variations in deformation rate under different applied pressures, surface roughness, and bonding cycles for ND and MDR components.

$$\text{Average pore size} = \frac{\text{Total void length } (L_1 + L_2 + L_3 + \cdots + L_n)}{\text{Number of voids } (L)} \times 100\% \quad (8)$$

#### Grain Boundary migration (GBM):

For the grain boundary migration, Equation 9 explained in the previous Section 2.1.2 was used, and 4 different sections of 670 $\mu\text{m}$  within the components' cross sections were considered for each measurement. To study the effect of induced dislocations, GBM was compared between ND and MDR components under different applied pressures.

$$\text{GBM ratio (\%)} = \frac{\text{Migrated GB length } (L_1 + L_2 + L_3 + L_4 + \cdots + L_n)}{\text{Total interface length } (L)} \times 100\% \quad (9)$$

#### Grain size:

The average grain size was calculated using the linear mean intercept method by counting the number of grain boundary intersections for a given size intercept (line). For each grain size calculation, five different regions of the sample were considered, and five different lines were measured per region. With this method, ASTM number and grain size was calculated, which was later converted to grain boundary density. An example can be seen in Figure 34. In order to calculate the grain size of the materials, these were first chemically etched using Nitric acid.

Two different materials, and in each case, three different samples were analyzed: initial grain size, grain size after a fast heating rate stage, and grain size after a slow heating rate stage for two different temperatures.

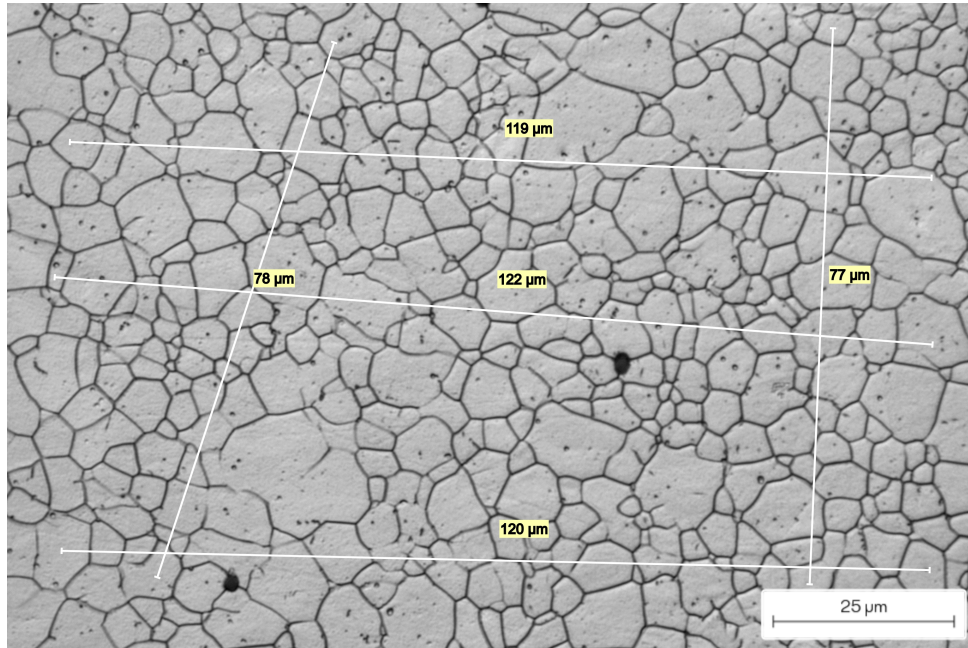


Figure 34: Example of grain size calculation. [28]



### Electron Backscatter Diffraction (EBSD):

Electron Backscatter Diffraction was used to quantify the dislocation density in the compressed (plastically deformed) samples before and after the heating stage. Measurements were taken once the target bonding temperature was reached in order to assess whether the dislocations had rearranged or remained in the material after the heating stage. Two different cases were analyzed: dislocation density for an HDR (high deformation rate) component before the heating stage and for an HDR component after the heating stage.

#### **3.3.1 Limitations**

Several limitations were identified throughout the experimental process. To start with, samples were deformed manually using a hydraulic press, and even if the same pressure was applied, variation in sample dimensions led to inconsistent deformation rates across the specimens. Furthermore, deformed samples were often uneven, producing tilted samples. These were fixed manually as precisely as possible using a surface roughness machine, but it was not always very precise. Apart from this, the target surface roughness values were also achieved by using the surface roughness machine, introducing further additional potential errors.

Moreover, when it comes to the bonding process, a standard industrial furnace was used instead of a diffusion bonding furnace, which required loads to be applied manually. This introduced the risk of uneven load distribution among the samples, meaning that some samples were potentially subjected to a different pressure than intended. In addition to this, when the pressure imbalance was high enough, tilting of samples could occur, reducing the effective contact area and therefore increasing the actual bonding pressure beyond the expected pressure. An example of a tilted sample can be seen in Figure 35.

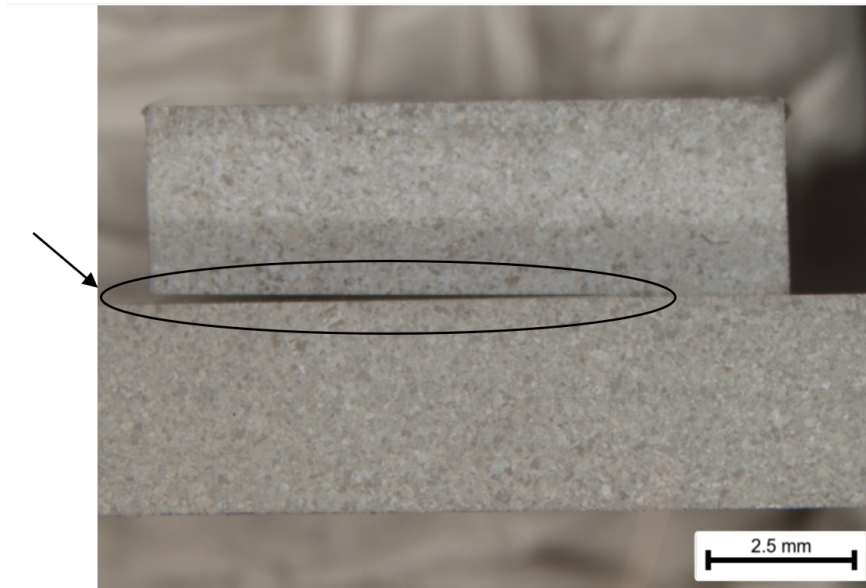


Figure 35: Example of a tilted sample. [28]

Lastly, since a production furnace was used for this study, the number of cycles was limited. Therefore, just one unit of sample was analyzed for each combination of parameters, limiting the statistical reliability of the results.

Considering all the limitations outlined above, the aim of this study was not to determine exact numerical values but rather to analyze trends associated with changes in different parameters.

## 4 Results and analysis

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This study investigates the influence of key diffusion bonding parameters (applied pressure, cycle time, heating rate, surface roughness, and dislocation density effect) on the joint quality of 316L stainless steel. Especially emphasizing on the effect of induced dislocations. The results are presented below. The joint quality was assessed by analyzing the porosity ratio, average pore size, pore size distribution, grain boundary migration, and grain size evolution.

The results are divided into three different sections. First, the methodology used was validated as reasonable. This was done by measuring the porosity at the bonding interface by varying parameters such as applied pressure, bonding time, and surface roughness. Once the results of varying these parameters were reasonable and the methodology was established, the second part of this project was conducted. Here, the effect of the induced dislocations on the bonding quality was studied. Finally, in the third stage of this project, the effect of Alfa Laval's long bonding cycles was analyzed. Here, dislocation density evolution was investigated conducting an EBSD study, to ensure the promoted bonding quality of these introduced dislocations for prolonged bonding cycles. Lastly, grain size evolution during different bonding cycles was analyzed.

### 4.1 Effect of conventional process parameters

In this first section, the influence of applied pressure, bonding cycle duration, and surface roughness on the bonding quality was analyzed. The obtained results of porosity, average pore size, and porosity distribution are shown, based on the techniques previously mentioned in the methodology.

#### Effect of differing the applied pressure

Samples bonded at higher pressure exhibited a lower porosity ratio (Figure 36), confirming that the bonding ratio increases with pressure. Higher pressure enhanced plastic deformation at the interface, and therefore, the average pore size at the interface was also reduced, from  $4.7\mu\text{m}$  up to  $2.45\mu\text{m}$ . The pore size distribution at the interface for different cases can be seen in Figure 37, representing the pore size and its relative abundance. Under lower bonding pressure, a higher amount of larger pores was observed, with a maximum pore size of  $82\mu\text{m}$ , whereas the maximum pore size decreased to  $34\mu\text{m}$  at a higher bonding pressure.



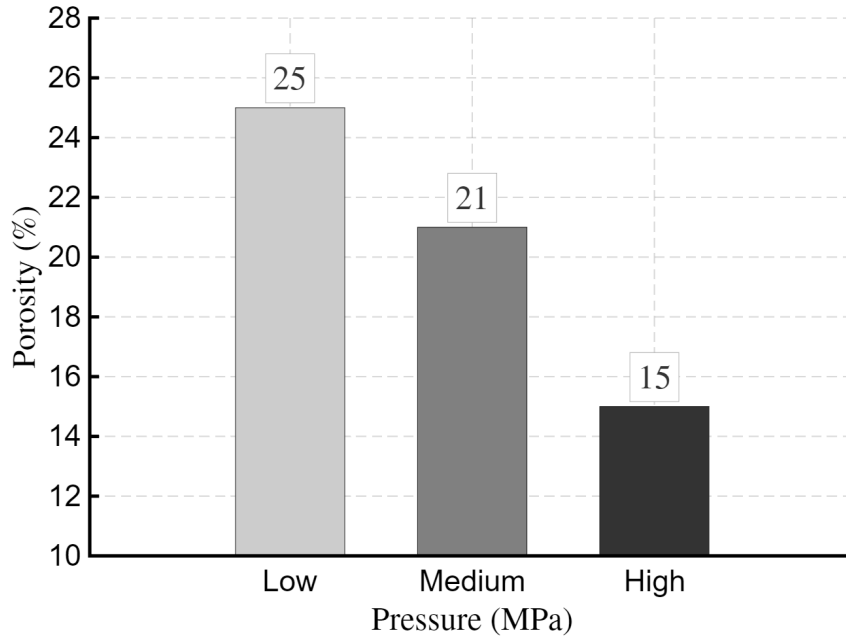


Figure 36: Porosity as a function of bonding pressure. [30]

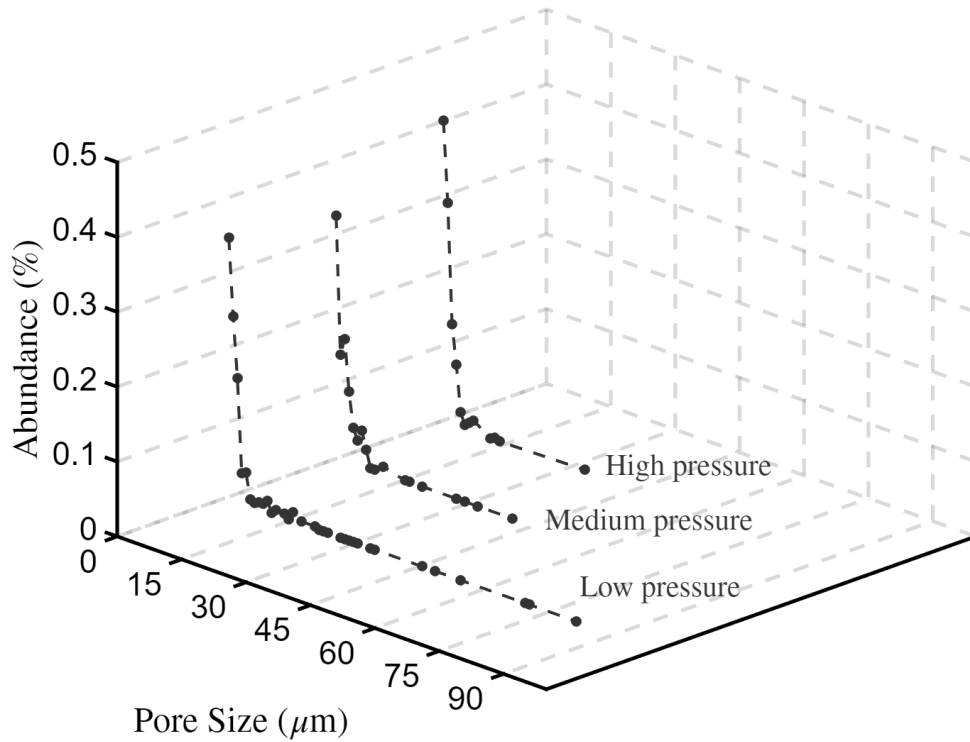


Figure 37: Pore size distribution vs. abundance, for cases with different applied pressure. [30]

#### Effect of differing surface roughness quality

Surface roughness is a critical parameter that influences the initial void formation and subsequently the bonding ratio. Smoother surfaces produced smaller initial voids compared to rougher surfaces. Figure 38 compares the porosity ratio between components with as received (AR) surface roughness and those with a rough surface roughness. This analysis confirms that under the same bonding conditions, a lower porosity ratio is obtained when using a smoother surface finish.

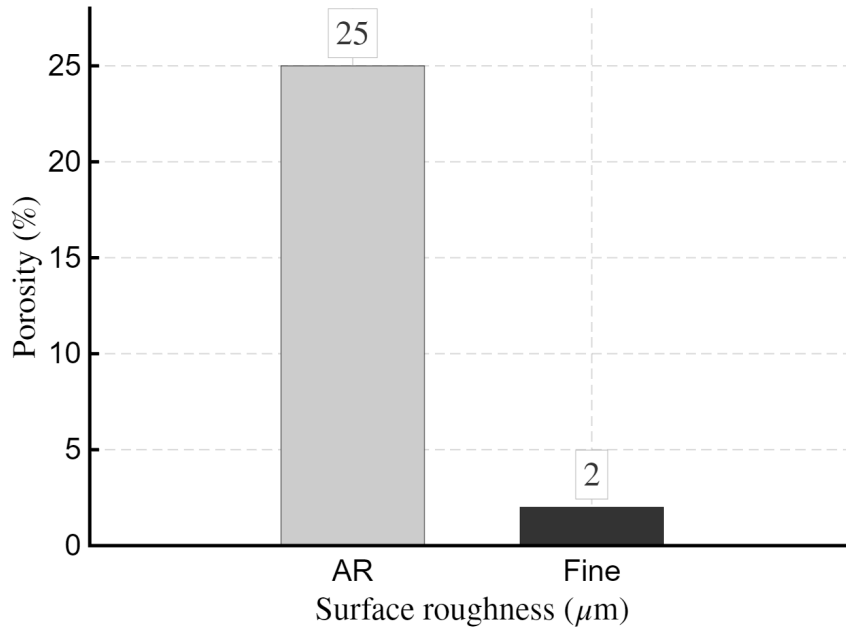


Figure 38: Porosity as a function of surface roughness. [30]

Apart from the porosity ratio, smooth surfaces also promote average pore size reduction. Rougher surfaces resulted in larger voids due to incomplete closure of these voids. The average pore size decreased from  $4.7\mu\text{m}$  up to  $1.1\mu\text{m}$  as the surface roughness was reduced. The comparison of the pore size distribution can be seen in Figure 39, representing the pore size and their relative abundance at the interface. Samples with rougher surfaces showed a wider distribution of the pore sizes, with a higher number of larger pores. The maximum pore size increased from  $7\mu\text{m}$  up to  $82\mu\text{m}$  with rougher surfaces. The interfacial voids' comparison of these two cases can be observed in Figure 40.

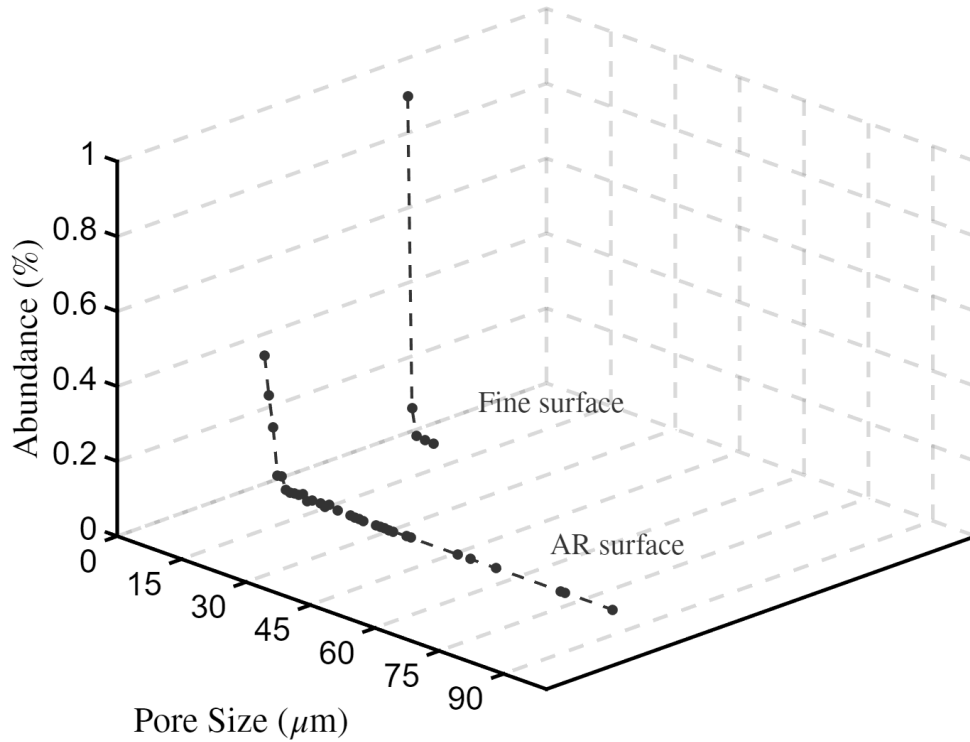


Figure 39: Pore size distribution vs. abundance, for cases with different surface roughness. [30]

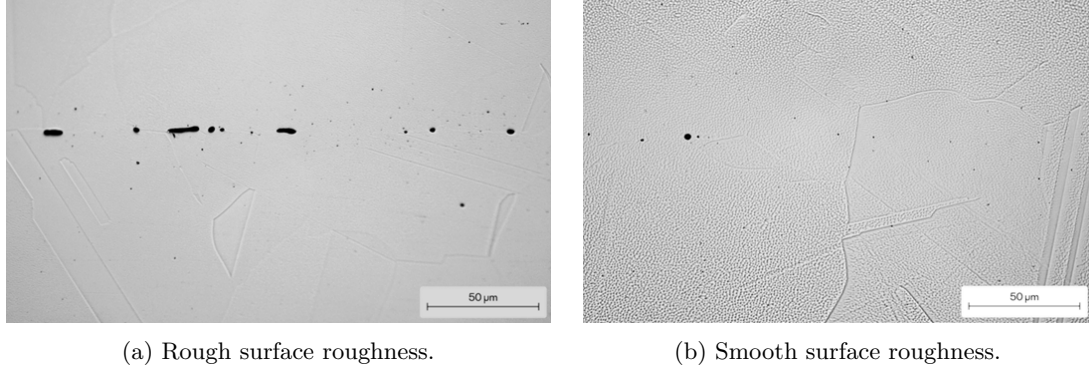


Figure 40: fig: Porosity at the interface for cases with different surface roughness. [28]

#### Effect of differing cycle time

Bonding time is also another key parameter that influences the void evolution and the bonding ratio at the interface. Longer bonding cycles provide more time for atomic diffusion and pore closure. As shown in Figure 41, under identical bonding conditions, a lower porosity ratio was achieved when a longer cycle time was used.

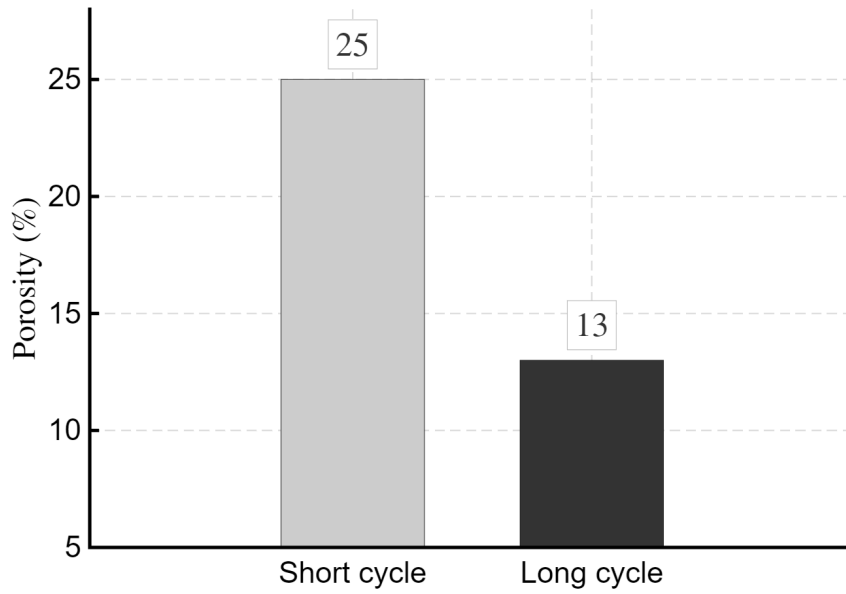


Figure 41: Porosity as a function of bonding cycle. [30]

Longer bonding cycles also promote the reduction in the average pore size of the voids, in this case a reduction from  $4.7\mu\text{m}$  up to  $2.6\mu\text{m}$  was observed. The microstructural differences at the interface can be observed in Figure 43. Samples bonded for longer bonding times exhibited smaller interfacial pores, indicating that the longer cycle time enhanced pore closure. A comparison of pore size distribution can be seen in Figure 42, showing the relationship between the observed pore sizes and their relative abundance at the interface. The maximum pore size of  $82\mu\text{m}$  was reduced to  $24\mu\text{m}$  when increasing the cycle time.

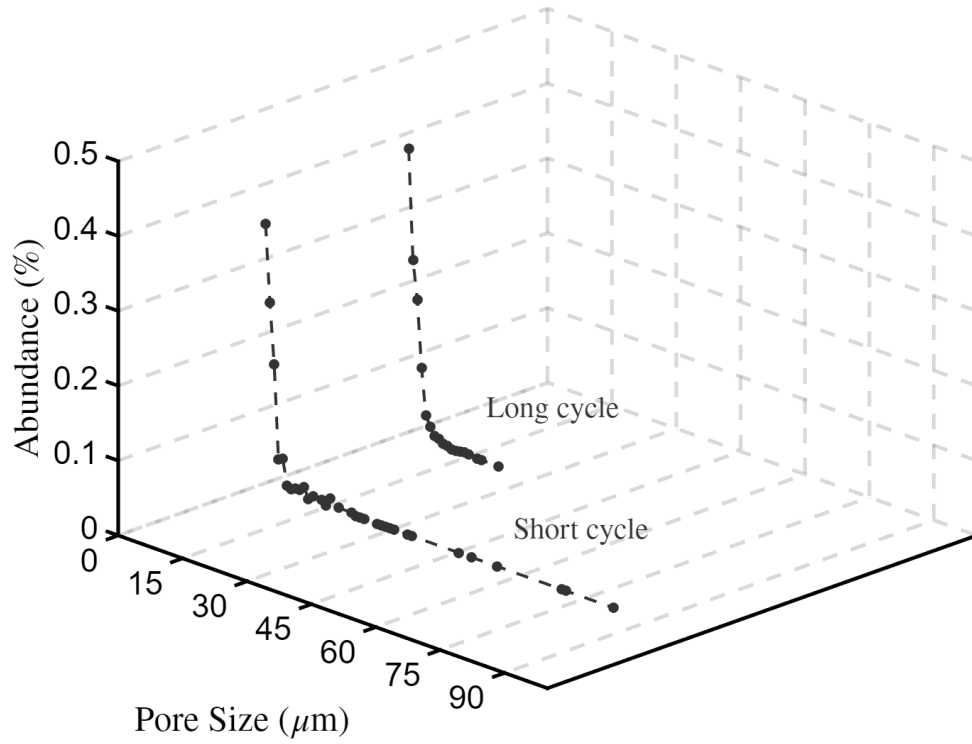


Figure 42: Pore size distribution vs. abundance, for cases with different bonding cycles. [30]

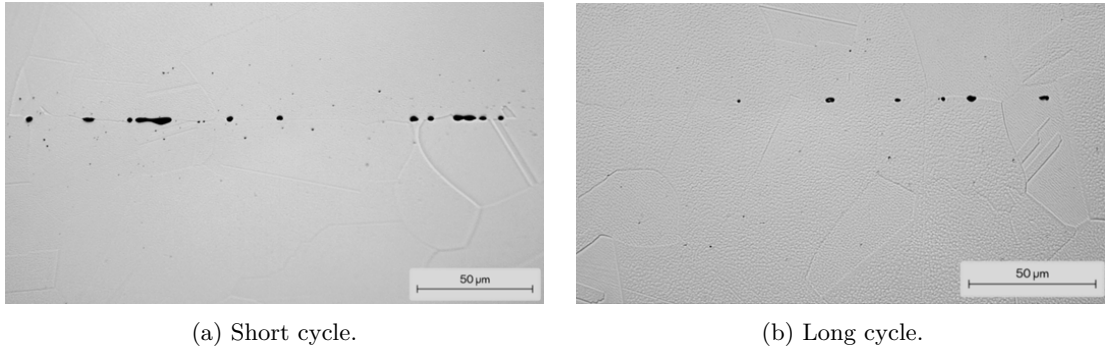


Figure 43: Porosity at the interface for cases with different bonding cycles. [28]

In this first section, the positive effect of changes in pressure, surface roughness, and bonding time on void closure and bonding quality was proved. Furthermore, it was observed that porosity reduction is strongly correlated with both the average pore size and the maximum pore size at the interface. These results correspond to the DB theory previously explained in the literature review. Therefore, the following methodology was considered to be effective for predicting trends when changing parameters' values.

## 4.2 Effects of dislocations in diffusion bonding quality

Dislocations in diffusion bonding provide many advantages that effectively enhance the bonding process. As previously discussed in section 2.1.3, these dislocations act as a rapid diffusion channel facilitating atomic migration across the interface, accelerating void closure and improving joint quality. In this study, samples were deformed using a hydraulic press, Figure 32. Four different deformation conditions were analyzed: non-deformed (ND), low deformation rate (LDR), medium deformation rate (MDR), and high deformation rate (HDR). Under equal bonding conditions, Figure 44 shows the obtained results of porosity ratio as a function of bonding pressure for each deformation condition. It was observed that increased dislocation density improved bonding by reducing the porosity ratio.

ND, MDR, and HDR all show a similar decreasing trend of porosity when increasing bonding pressure. However, a big difference on porosity ratio was observed between ND and MDR samples, indicating the key role that dislocations play in enhancing the bonding process. On the other hand, the difference between a medium deformed material and a highly deformed material is minimal, suggesting a stabilizing effect of dislocations at higher densities. However, it was assumed that in general, dislocations enhanced the diffusion bonding process quality.

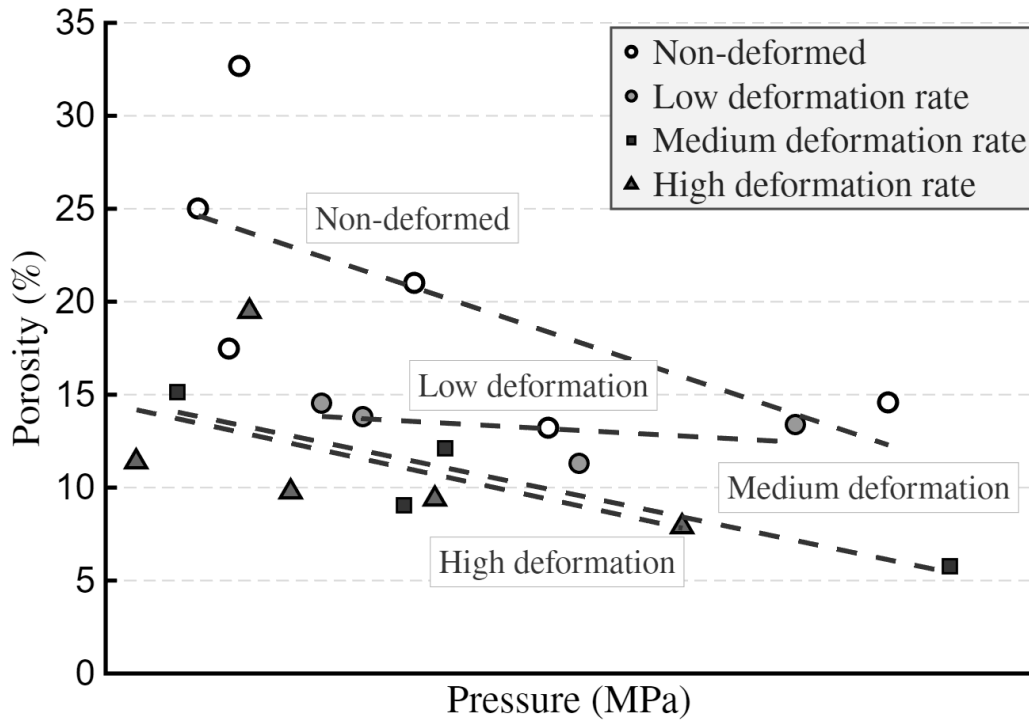


Figure 44: Porosity as a function of boning pressure for bonded components with different deformation rates. [30]

Under the same applied pressure, the influence of deformation on porosity ratio can be seen in Figure 45. Even if the results showed that there was a significant influence of dislocation on decreasing the porosity ratio, it was also observed, that beyond a certain deformation rate, the effect of dislocation stabilized and that there was no further notable improvement in the bonding ratio beyond this deformation rate.

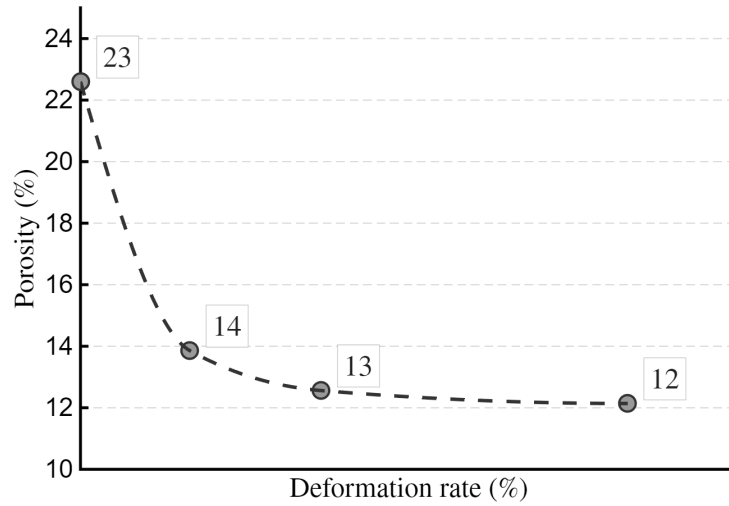


Figure 45: Porosity as a function of deformation rate. [30]

The average pore size also decreased when materials contained higher dislocation densities under the same bonding conditions. At low bonding pressures, the average pore size decreased from  $4.98\mu\text{m}$  to  $3.35\mu\text{m}$  when dislocations were introduced. At intermediate bonding pressures, the difference in average pore size between MDR and ND materials also decreased, but slightly less, from  $3.92\mu\text{m}$  to  $2.98\mu\text{m}$ . However, at higher pressures, as the porosity ratio continued decreasing for both the deformed and non-deformed materials, and since the porosity ratio is directly related to the average pore size at the interface, at lower porosity ratios (when higher bonding pressure was applied), the average pore size showed little difference between a deformed and a non-deformed material. This effect on the average pore size can be observed in Figure 46.

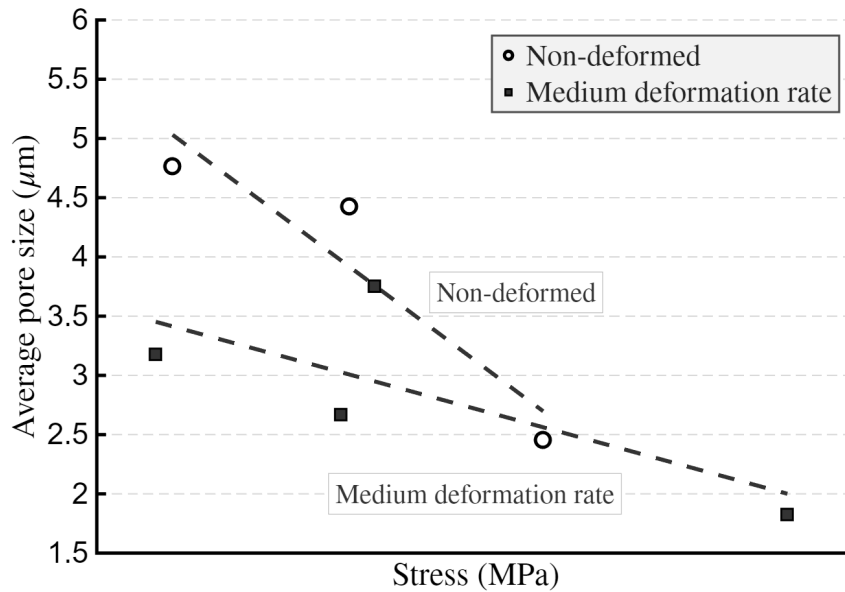
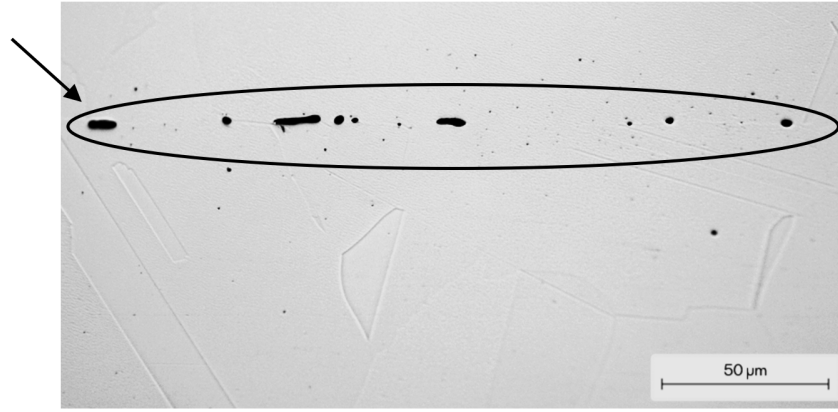


Figure 46: Average pore size as a function of deformation rate for bonded components with different deformation rates. [30]

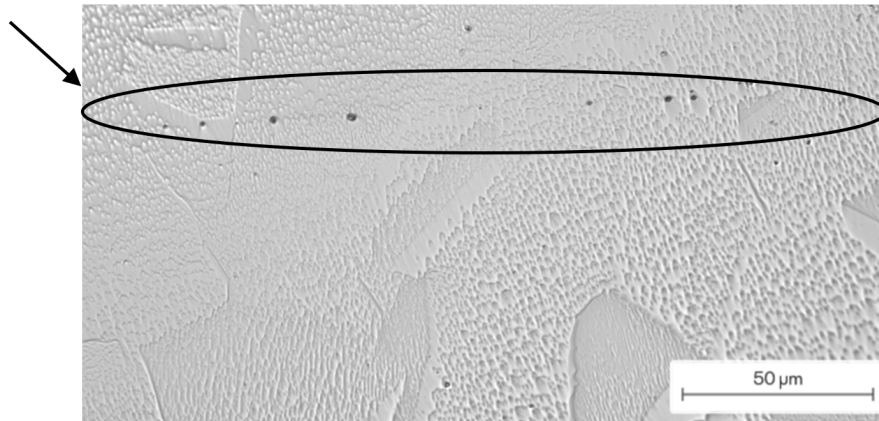
The difference in porosity at the interface between a non-deformed material (a), a material with medium deformation rate (b), and a material with high deformation rate (c) can be observed in Figure 47. As can be seen, porosity abundance, together with the pore size, become smaller for those with a higher density of dislocations.



(a) Non-deformed material.



(b) Medium deformation rate material.



(c) High deformation rate material.

Figure 47: Porosity at the interface for cases with different deformation rates. [28]

#### Effect of dislocations on Grain Boundary Migration

Apart from the porosity ratio and the average pore size at the interface, the effect of dislocations on grain boundary migration (GBM) was also analyzed in this study. GBM ratio is also used to quantify bonding quality, and the results are shown in Figure 48. Here, GBM ratio values as a function of porosity can be seen.

These results showed that GBM ratio increased for the cases where dislocations were introduced, whereas GBM was lower for those where dislocations were not present. However, both cases showed a similar trend: as porosity decreased, GBM ratio increased. Therefore, it seems that GBM ratio is porosity de-



pendent and that an increase on GBM ratio is not directly caused by dislocations. Rather, dislocations significantly enhance pore closure and reduce porosity, and it is this porosity reduction that subsequently drives the increase on GBM ratio. It can be observed that when porosity was reduced from 35% to 25%, there was no significant improvement on GBM ratio. However, when porosity was further reduced from 25%, a significant increase on GBM ratio started to happen.

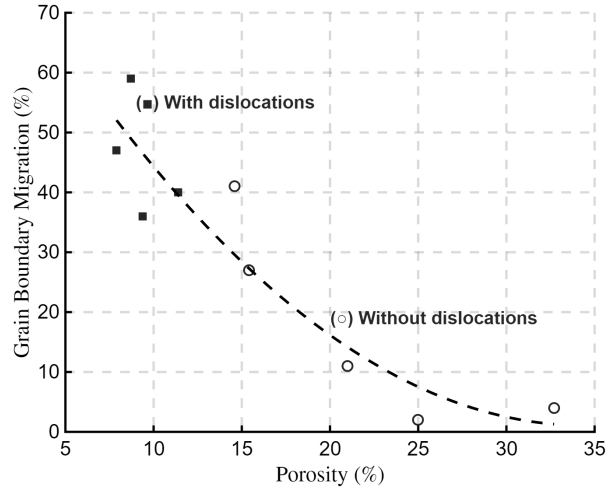


Figure 48: GBM as a function of Porosity ratio for bonded components with and without dislocations. [30]

The same concept can be analyzed in Figure 49. The results showed that GBM ratio increased as the average pore size decreased. Since the average pore size is directly related to the porosity ratio, GBM ratio followed a similar trend as in the previous analysis. A significant improvement on GBM ratio began to happen when the average pore size started to decrease below 4  $\mu\text{m}$ .

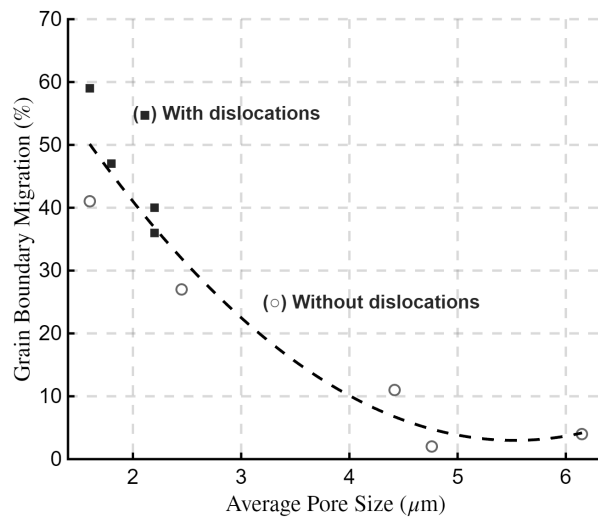


Figure 49: GBM as a function of average pore size for bonded components with and without dislocations. [30]

Furthermore, when considering the results from the two previous Figures (48 and 49), when two different samples with the same porosity (approximately 10%) were considered, a difference of more than 20% on GBM ratio was observed. These two cases are shown in Figure 50, and it can be observed that even if these interfaces experienced the same porosity, the average pore size differed, and that the one with the lowest porosity experienced a higher GBM ratio.

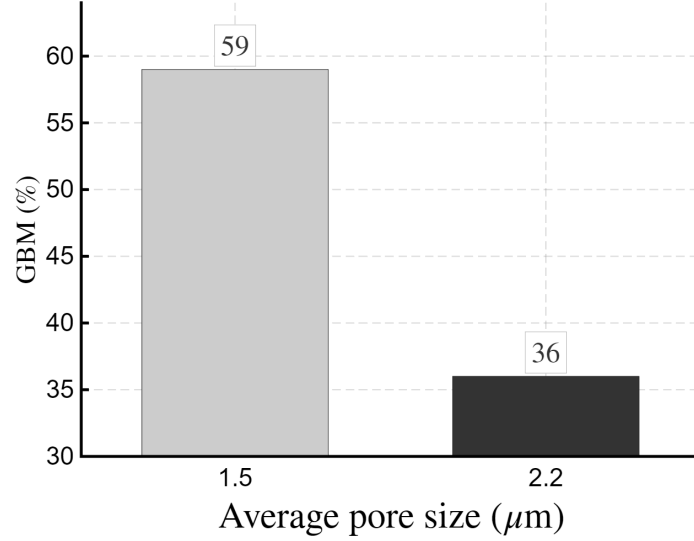


Figure 50: GBM as a function of average pore size for components with the same porosity. [30]

On the other hand, when two different cases with the same average pore size values (approximately  $1.6\mu\text{m}$ ) but different porosity were compared (an example is shown in Figure 51), it was observed that the one with lower porosity exhibited a larger GBM ratio. As shown in Figure 51, a difference of almost 20% on GBM ratio was observed between these two cases with the same average pore size but different porosity ratio.

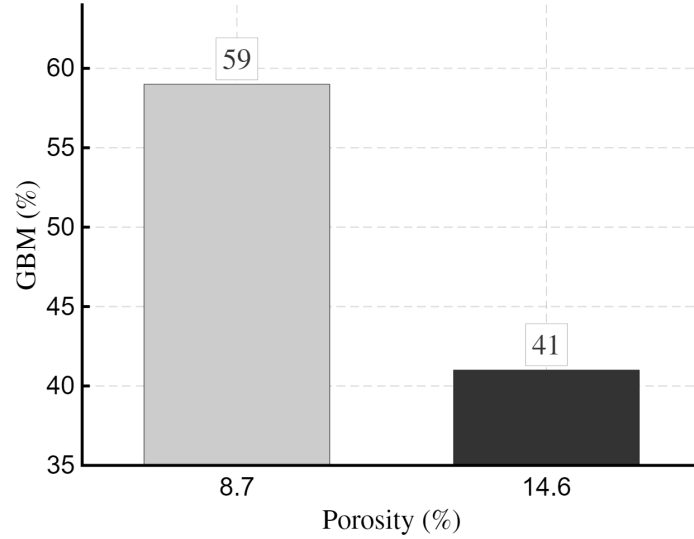


Figure 51: GBM as a function of porosity for components with the same average pore size. [30]

This leads to the conclusion that GBM ratio is dependent on both the average pore size and the porosity ratio, independently. This can be explained by the fact that the average pore size only considers the length of the individual voids, whereas the porosity ratio accounts for both the number and the size of these voids. Therefore, even when the average pore size is small, the porosity ratio can vary depending on the number of these pores. Conversely, for the same porosity ratio, the average pore size can also vary, for instance, a material can have few but very large pores (increasing the average pore size), or more but small pores (decreasing the average pore size). Both parameters consider different aspects of the voids at the interface, therefore, even if in general their values are connected, they enhance GBM independently.

Figure 52(a) shows an example of the microstructure at the interface where large pores, high porosity (21%), and a low GBM ratio of 11% were observed. In this case, the pores are placed at the grain boundaries. In contrast, Figure 52(b) shows the example where a large GBM ratio of 59% was obtained. Here, the pores and the porosity (9%) are lower, therefore, the grains migrated through the interface, and subsequently, the pores were left inside the grains. In this case, the bonding interface is no longer that clear due to the extended GBM ratio. In this comparison, the importance of a low porosity ratio and a low average pore size for GBM ratio is remarked.

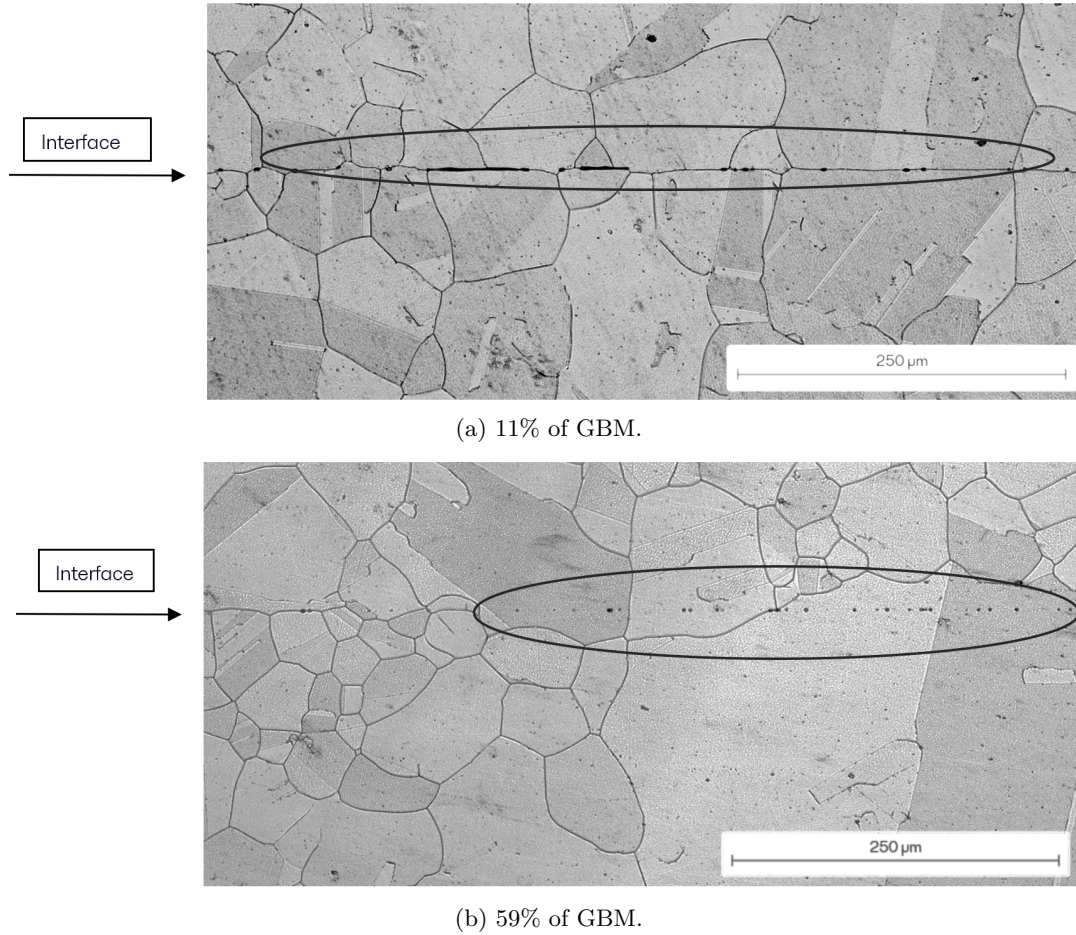


Figure 52: Microstructure comparison for different degrees of GBM. [28]

As has been seen, when GBM occurs, the pores were no longer located at the grain boundaries. Therefore, at this conditions, when the bonded component undergoes stresses, these stresses are not further concentrated at the pores, which directly diminishes their negative effect, subsequently increasing the joint strength. Thus, for components with the same porosity, those with higher GBM ratio will exhibit better bonding quality, indicating that porosity and the pore size are mainly relevant in cases where GBM is low. Once the porosity and the average pore size at the interface are small enough for GBM to occur, the negative effect of these pores is significantly mitigated.

The overall influence of dislocations on bonding quality can be seen in Figure 53. For different bonding pressures, the introduced dislocations showed a significant positive effect on void closure, reducing porosity and the average pore size, which subsequently led to an increase on GBM ratio. Both materials, with and without dislocations, showed a similar increasing trend on GBM ratio, as porosity and the average pore size decrease when increasing pressure. However, porosity and average pore size values for the material with dislocations were consistently lower, due to the enhanced atomic migration at the interface facilitated by dislocations, promoting more effective void closure and subsequently increasing GBM ratio.

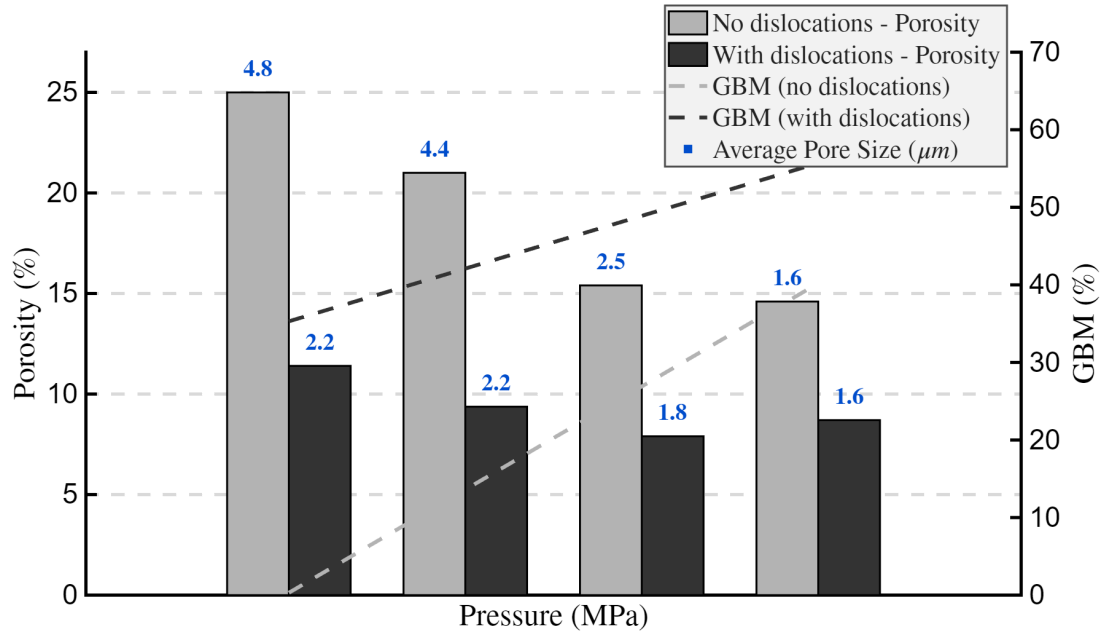


Figure 53: Comparison of bonded components with and without dislocations at different bonding pressures, in terms of porosity, average pore size, and GBM. [30]

#### 4.2.1 Influence of dislocation in rougher surface conditions

In this case, the influence of dislocations on a rough surface was measured (Figure 54). It was seen that for a non-deformed material, the obtained porosity ratio was slightly larger than when dislocations were introduced. There was not much difference between these two cases, and it could be assumed that the obtained joint quality was almost the same for both cases. Concluding that in this case, introducing dislocations did not enhance bonding. As is known, having an initial rough surface leads to larger initial pores at the interface, thereby reducing the contact area between the components. If the contact area is low, the dislocations in the component do not act like pathways for atomic migration, mitigating their positive effect and therefore not further increasing bonding quality.

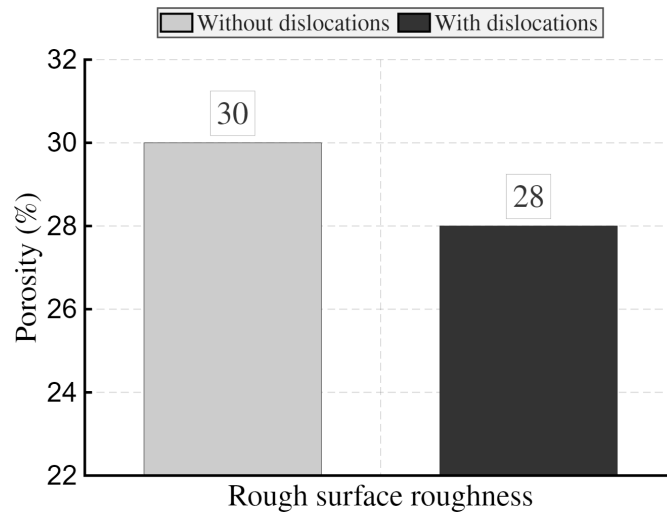


Figure 54: Porosity ratio between bonded components with or without dislocations when using a rough surface finish. [30]

Figure 55 shows porosity as a function of applied bonding pressure for three different sample conditions. From all these three conditions, it was seen that components with medium deformation rate and rough surfaces resulted in the highest porosity at the interface (lowest joint quality). Demonstrating that introducing dislocations does not improve joint quality if the surface roughness is not good enough.

Comparing the two cases where a medium deformation rate was applied, the one with a rougher surface showed significantly higher porosity. As expected, reducing surface roughness quality increased porosity. In this case, this was caused by both the initially created larger and more numerous voids but also because the influence of dislocations was mitigated when having lower component's contact area at the interface.

When comparing the case of a rough surface with dislocations, with the case of a smoother surface without dislocations, it was seen that a finer surface without dislocations achieved lower porosity. Concluding that the surface quality had a more dominant effect on bonding quality than just the presence of dislocations. In summary, it was observed that if the interfacial contact length between the components is not large enough, introducing dislocations does not further improve bonding quality.

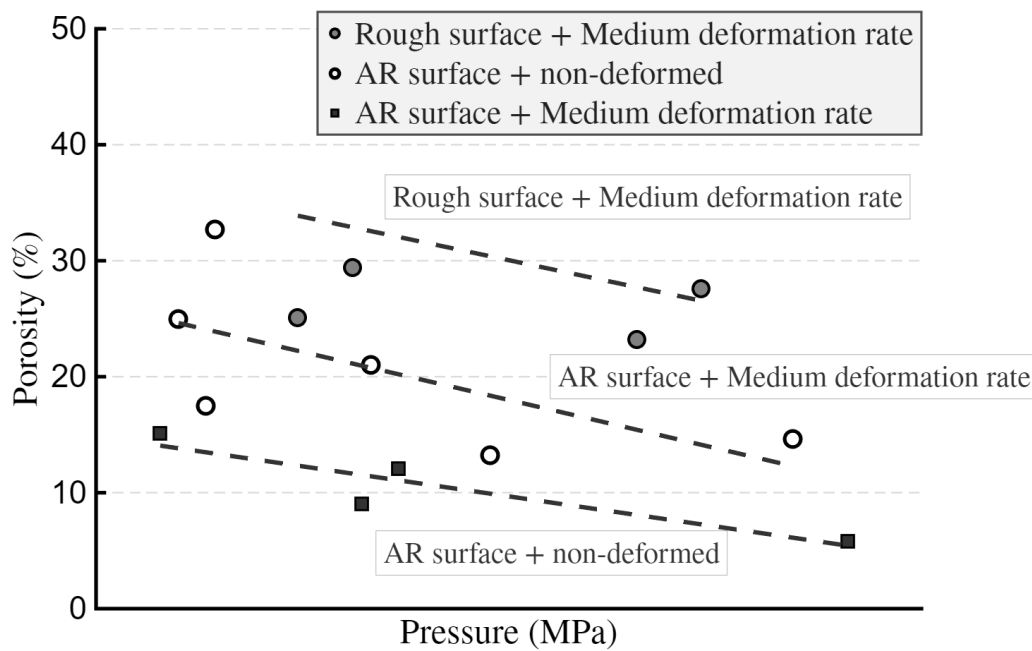


Figure 55: Porosity as a function of boning pressure for components with or without dislocations at different surface finishes. [30]

In this second section, it was concluded that dislocations reduced both porosity and the average pore size by enhancing atomic migration and therefore accelerating void closure at the interface. Subsequently, the introduction of dislocations also promoted higher GBM ratio. However, it was observed that a sufficiently fine surface roughness was required to ensure a large enough contact area at the interface, through which atoms could effectively migrate and subsequently enhance bonding quality.

### 4.3 Alfa Laval's long bonding cycle analysis

Due to the size of diffusion bonded units produced at Alfa Laval, bonding cycles can be significantly long with slow heating rates. As is known, dislocations tend to rearrange at elevated temperatures during long exposure times, which can diminish their beneficial effect in the process [14]. Therefore, the effect of dislocations on long cycles was analyzed in this third section. A schematic comparison of a lab-scale representative cycle (the one used in the previous sections) and a real cycle (Long cycle) can be observed in Figure 56.

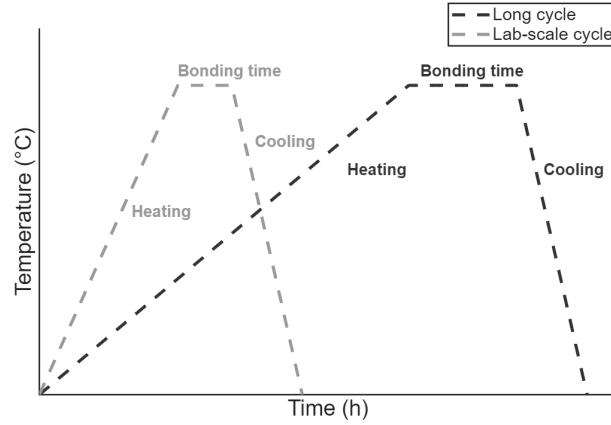


Figure 56: Schematic comparison between a lab-scale bonding cycle and a long bonding cycle. [30]

Apart from this, elevated temperatures combined with prolonged exposure times can coarsen the grains in the material and consequently reduce the grain boundary density [31]. As previously seen, materials' grain boundary density is directly related to diffusion bonding, since a larger density provides more boundaries that act as short circuit diffusion paths for atomic migration, thereby increasing the bonding ratio [23].

Grain size evolution for different exposure times and temperatures for an original austenite grain size can be seen in Figure 57. As can be seen here, once beyond 950 °C, austenitic grains start to coarsen significantly with increasing holding time. However, the material does not experience significant grain coarsening when exposed at 900 °C or 950 °C, not even for longer holding times. Therefore, in this section, grain size evolution at different conditions was investigated for 316L material [32].

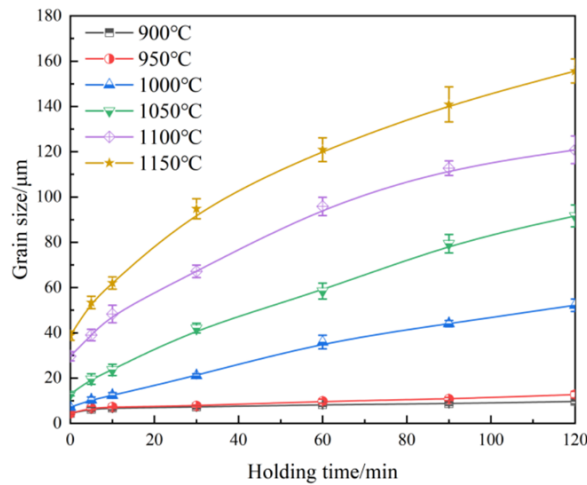


Figure 57: Grain size as a function of holding time for different temperatures. [32]

#### 4.3.1 Dislocation effect in longer bonding cycles

First, the dislocation density for different components was measured using an EBSD. The dataset was then processed using MTEX ([33], [34], [30]) by measuring the local grain misorientation, known as Kerbel Average misorientation (KAM [35], [30]), and later maps representing dislocation density energy were simulated in  $\mu\text{m}^{-1}$  using the MTEX Toolbox ([36], [30]). Using these measurements, the dislocation density of a deformed material was compared before and after the heating stage (of a long bonding cycle). The idea was to analyze whether dislocations would remain in the material after the heating stage or whether they would rearrange (mitigating their positive impact). The obtained results for the deformed material before the heating stage can be seen in Figure 58(a), and after the heating stage in Figure 58(b).

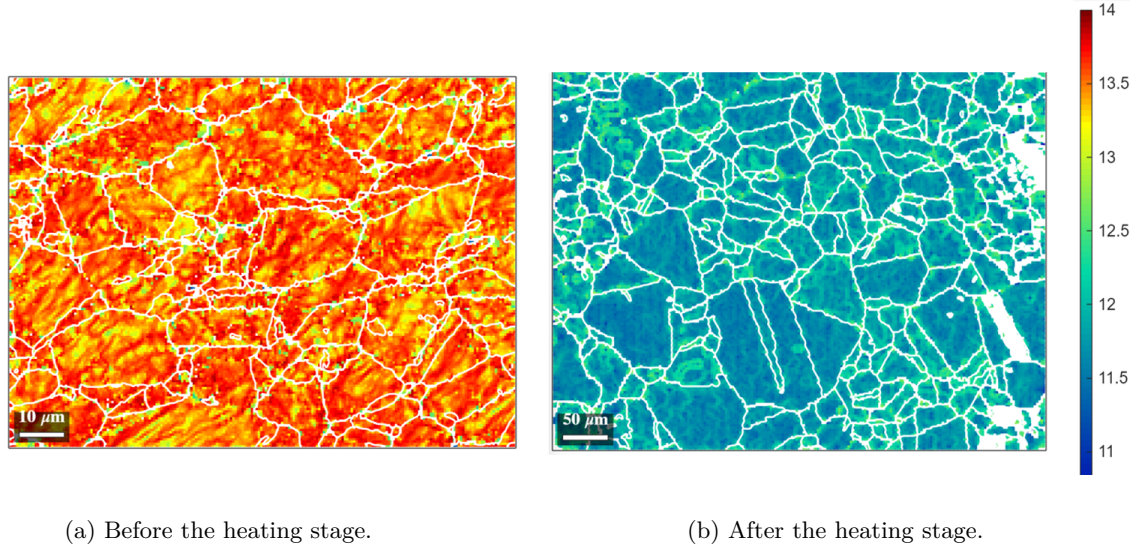


Figure 58: GND density comparison between two deformed materials. [30]

As expected, the dislocation density energy before the heating stage, Figure 58(a) was higher than after the heating stage, Figure 58(b). However, Figure 58(b) showed that there was some remaining dislocation energy in the material, which should act as diffusion pathways and enhance bonding.

Even if the KAM results confirmed the presence of partially remaining dislocations, it was not known whether these would be sufficient to enhance bonding. Therefore, the porosity of a deformed material was compared with that of a non-deformed material, both under the same long bonding cycle. The results can be seen in Figure 59.

These results showed that the bonding quality improved when dislocations were introduced during long bonding cycles. Therefore, it was assumed that the remaining dislocations shown in Figure 58(b) were sufficient to contribute to bonding. Furthermore, Figure 59 compares the porosity of a short cycle with that of a long cycle (both with dislocations). It can also be seen that the long cycle resulted in a lower porosity ratio. It was concluded that the remaining dislocation density after the long heating stage was enough to contribute to diffusion bonding, and that the longer bonding cycle allowed more time for atomic diffusion and pore closure, further increasing bonding quality. The combination of a long bonding cycle with dislocations effectively improved bonding quality.

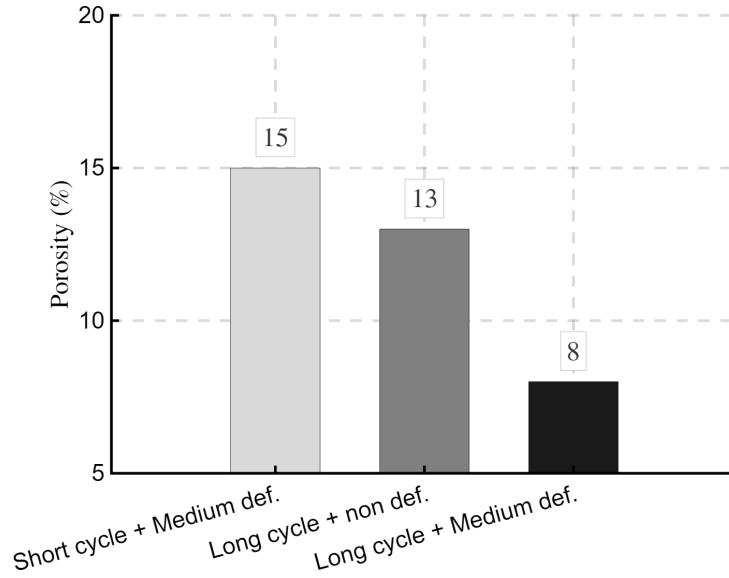


Figure 59: Porosity as a function of bonding cycles and deformation rates. [30]

In addition, the pore size distribution along the interface for these three cases is shown in Figure 60, where the pore size and the abundance of these ones can be observed. As expected, for the case with the lowest porosity value (long cycle + dislocations), the voids at the interface were smaller, whereas for the case with the highest porosity value (short cycle + dislocations, and long cycle + without dislocations), the voids were larger, increasing the average pore size. The combination of dislocations with long bonding cycles improved bonding quality not only by reducing porosity but also by reducing the average pore size at the interface.

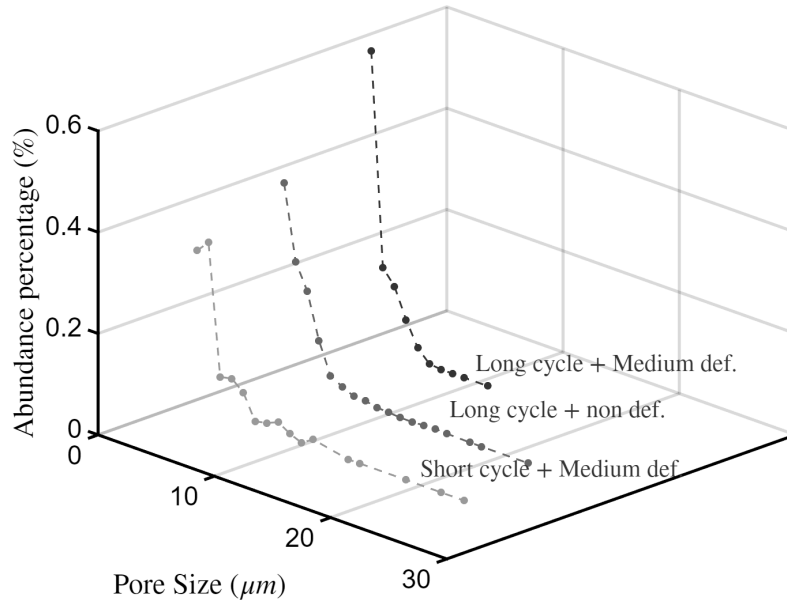


Figure 60: Pore size distribution vs. abundance, for different bonding cycles with and without dislocations. [30]

All in all, the presented results show that dislocation density was partially reduced after the long cycle. However, the remaining dislocation density was sufficient to enhance diffusion through these pathways, reducing porosity and average pore size at the interface.



#### 4.3.2 Grain size evolution after longer cycles and different temperatures

As previously mentioned, a finer grain sized material is known to enhance diffusion bonding performance. Therefore, the effect of different heating rates (corresponding to short and long bonding cycles) and different bonding temperatures on grain size evolution of two 316L materials with different initial grain sizes (fine and coarse grain sized material) was analyzed. The objective was to determine and quantify the grain size evolution at different points during the cycle. Furthermore, compare whether there is any benefit in using a material with smaller grain size.

First, the initial grain size of each material was measured together with their respective grain sizes after exposure at two different heating rate stages, corresponding to a short cycle (fast heating rate) and a long cycle (slow heating rate), as schematically represented in Figure 61.

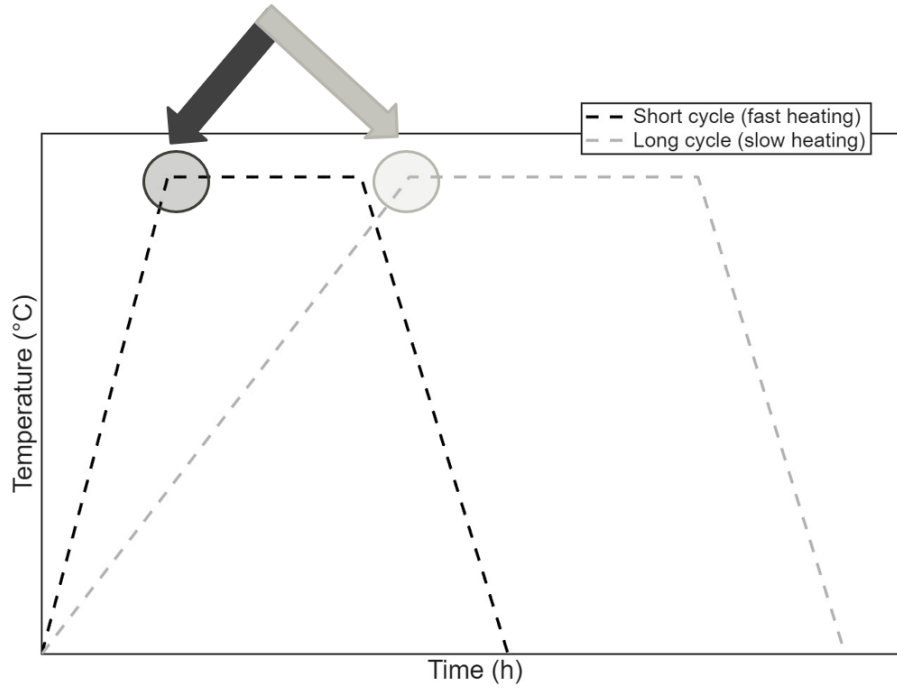


Figure 61: Schematic representation of tested short and long cycles. [30]

The obtained grain size and grain boundary density values for each case can be seen in Table 2. As can be observed, during the heating stages both materials experienced grain coarsening together with grain boundary density reduction. Even if at the end of the heating stage, the material with an initial coarser grain size got a higher grain size value, the material with a finer initial grain size experienced a significantly higher grain coarsening, and thereby a higher GB density reduction. The comparison can be seen in Table 3. However, both materials' grain boundary densities were reduced almost to  $0 \mu\text{m}^{-1}$ .

Table 2: Grain size and GB density for coarse and fine initial grain sizes.

| Condition         | Coarse initial grain size    |                                   | Fine initial grain size      |                                   |
|-------------------|------------------------------|-----------------------------------|------------------------------|-----------------------------------|
|                   | Grain size ( $\mu\text{m}$ ) | GB density ( $\mu\text{m}^{-1}$ ) | Grain size ( $\mu\text{m}$ ) | GB density ( $\mu\text{m}^{-1}$ ) |
| Initial condition | 22                           | 0.09                              | 6                            | 0.340                             |
| After fast cycle  | 67                           | 0.03                              | 66                           | 0.030                             |
| After slow cycle  | 102                          | 0.02                              | 82                           | 0.024                             |

Table 3: Grain size increase and GB density drop for coarse and fine initial grain sizes.

| Condition         | Coarse initial grain size |                 | Fine initial grain size |                      |
|-------------------|---------------------------|-----------------|-------------------------|----------------------|
|                   | Grain size increase       | GB density drop | Grain size increase     | GB density reduction |
| Initial condition | –                         | –               | –                       | –                    |
| After fast cycle  | 2×                        | 66%             | 10×                     | 91%                  |
| After slow cycle  | 3.6×                      | 77%             | 12.76×                  | 93%                  |

If Figure 62 is analyzed, it can be seen that both materials followed a similar coarsening trend, and when the corresponding standard deviation values were considered, the obtained grain sizes were effectively equivalent after the long heating stage.

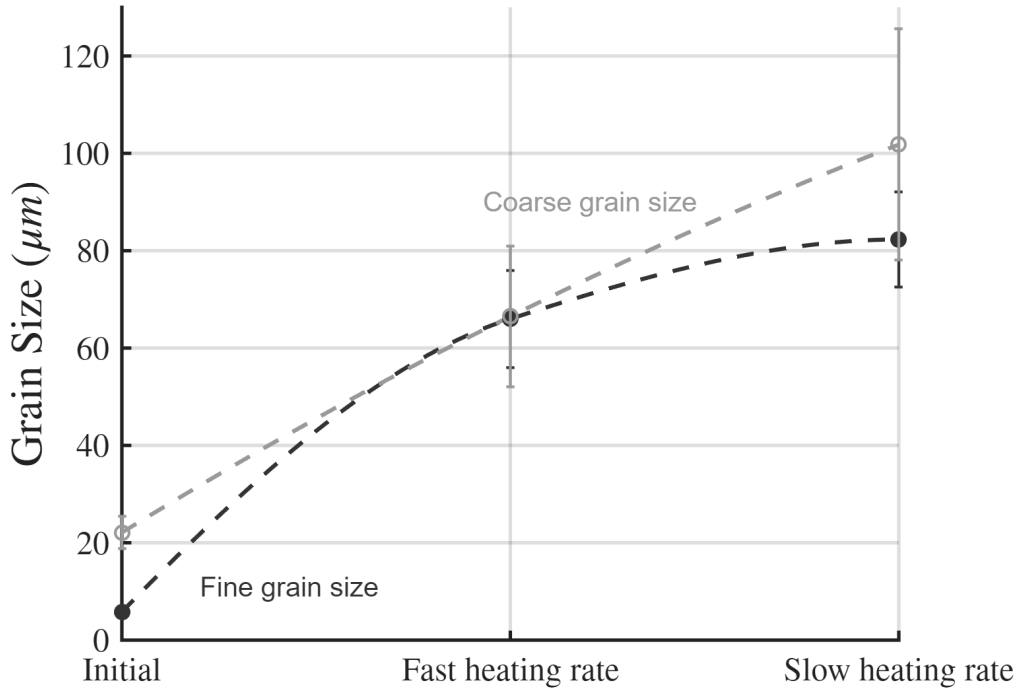


Figure 62: Grain size evolution after different heating rate stages, for a coarse grain size material and a fine grain size material. [30]

The initial grain boundary density of the coarse-grained material was  $0.091 \mu\text{m}^{-1}$ , whereas the initial grain boundary density of the fine-grained material was  $0.331 \mu\text{m}^{-1}$ . After the long heating stage, these values were reduced to  $0.021 \mu\text{m}^{-1}$  and  $0.0241 \mu\text{m}^{-1}$ , respectively. Demonstrating that even if the initial grain boundary density of that of the fine-grained material was more than three times higher than that of the coarse-grained material, after the heating stages, the final densities were very similar without a significant difference. Suggesting that grain boundary density tends to decrease to a comparable level when achieving the aimed bonding temperature in the cycle, even if the initial grain boundary density significantly differs. The studied grain boundary density reduction trend for both materials can be seen in Figure 63.

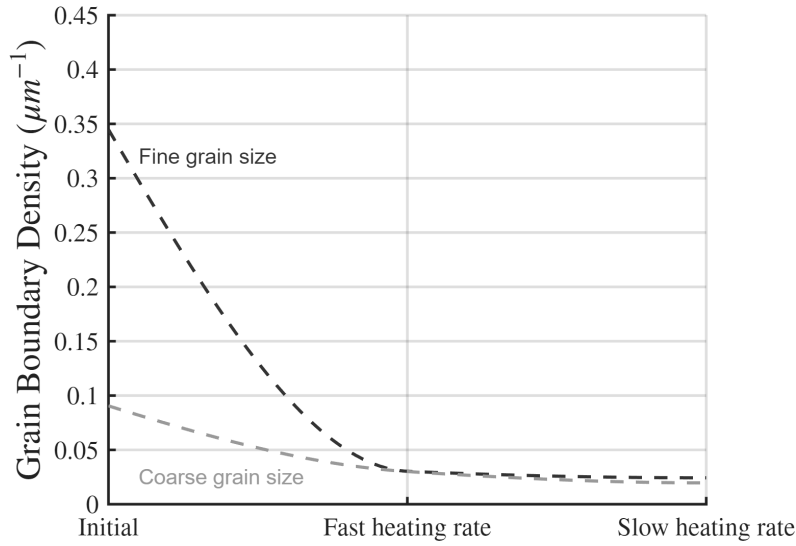


Figure 63: Grain boundary density evolution after different heating rate stages, for a coarse grain size material and a fine grain size material. [30]

Figure 64 shows how the grain boundary density (GBD) decreases as the average pore size increases. Here, a big difference in the initial grain boundary densities between the coarse and fine grain sized materials was observed. However, after heating both components at a high temperature, either at a fast or at a slow heating rate, the grain boundary density was significantly reduced to almost  $0.01 \mu\text{m}^{-1}$  for both materials, mitigating the positive effect that grain boundaries have on atomic diffusion across the interface. Therefore, there was no significant difference in having a coarse or a fine grain sized material after heating it at this analyzed high temperature, regardless of whether a slow or a fast heating rate was applied.

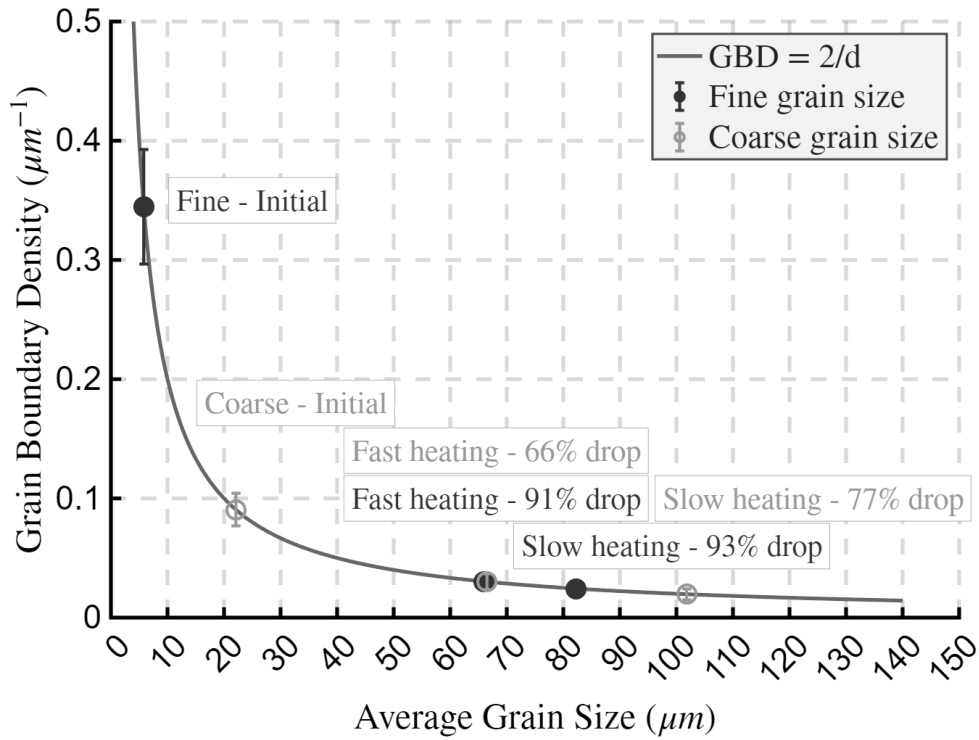
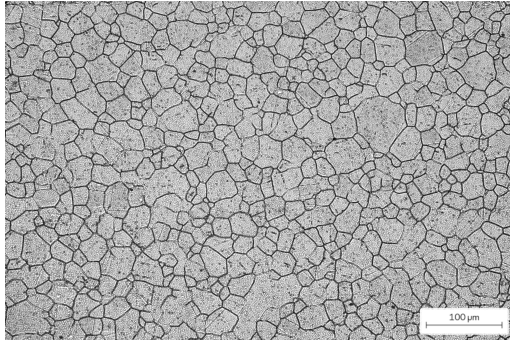
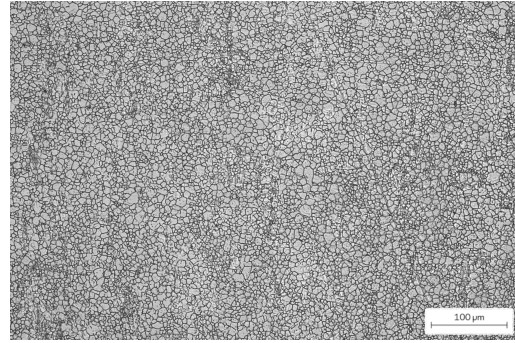


Figure 64: Grain boundary density as a function of average grain size after different heating rate stages, for a coarse grain size material and a fine grain size material. [30]

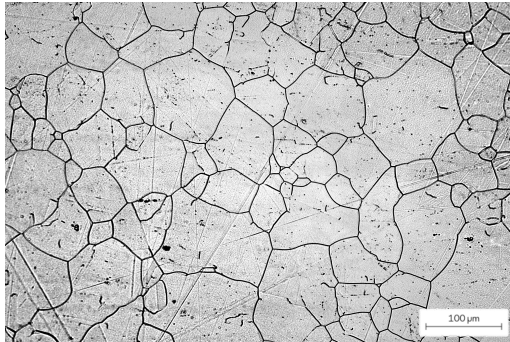
The microstructural grain size evolution of these two materials before the heating stages and after the heating stages is shown in Figure 65. It can be observed that there was a significant difference in grain sizes between the two initial materials 65(a-b), but this difference decreased considerably as the exposure time increased, as can be seen in 65(e-f).



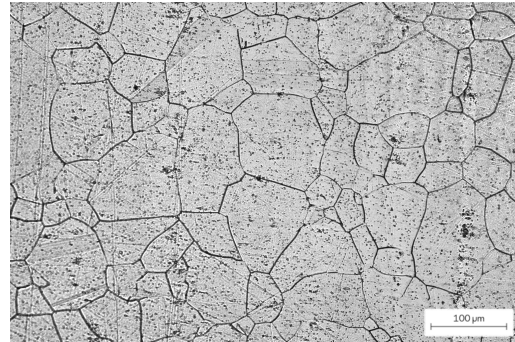
(a) Coarse grain size - Initial stage.



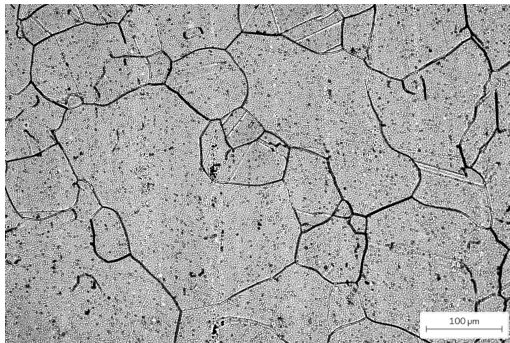
(b) Fine grain size - Initial stage.



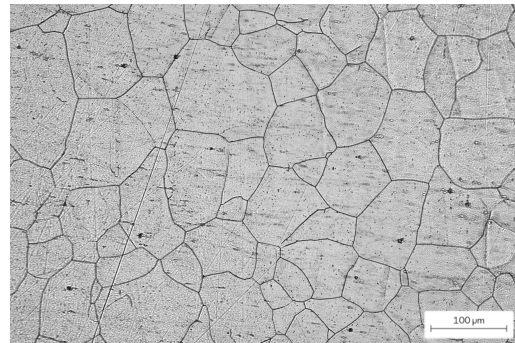
(c) Coarse grain size - After fast heating rate stage.



(d) Fine grain size - After fast heating rate stage.



(e) Coarse grain size - After slow heating rate stage.



(f) Fine grain size - After slow heating rate stage.

Figure 65: Microstructural grain size evolution of a coarse grain size material (a,c,d) and a fine grain size material (b,d,e). [28]

As has been observed, from the grain boundary density perspective, there was no significant difference in having a faster or slower heating rate stage. A slower heating stage is only used to ensure homogeneous heating and temperature across all components. Therefore, heating rate based on the size of the components should be optimized to prevent the use of a slower heating rate than needed, but at the same time ensuring that this stage is not faster than what is needed. This would further optimize the process, making it more effective, reducing production time and costs. Furthermore, as at the end of the heating stages (at this specific aimed bonding temperature), the grain boundary densities were the same for both materials, it was assumed that there is no further benefit in having a finer initial grain size.

### Grain size after longer cycles at a lower temperature

As there was almost no difference in the grain boundary densities between fine and coarse grain sized materials at the previously aimed high bonding temperature, the effect of a lower temperature was studied in this section. As shown in Figure 57, the grains do not coarse after long exposure times below 950 °C. Consequently, in this study, the components were exposed to a similar temperature to ensure that grain coarsening would not occur at lower temperatures for the used materials.

The obtained average grain size and grain boundary density values for both coarse and fine grain sized materials after the heating (below 950 °C) can be seen in Table 4 and Figure 66. As can be observed, when considering standard deviation values, neither of the materials experienced grain coarsening. Therefore, a significant difference in grain boundary densities was still observed between the coarse and the fine grain sized materials. The finer material keep providing around 4 times larger GB density after heating.

Table 4: Grain size and GB density for coarse and fine initial grain sizes.

| Condition                 | Coarse Initial Grain Size       |                                      | Fine Initial Grain Size         |                                      |
|---------------------------|---------------------------------|--------------------------------------|---------------------------------|--------------------------------------|
|                           | Grain Size<br>( $\mu\text{m}$ ) | GB Density<br>( $\mu\text{m}^{-1}$ ) | Grain Size<br>( $\mu\text{m}$ ) | GB Density<br>( $\mu\text{m}^{-1}$ ) |
| Initial condition         | 22                              | 0.09                                 | 6                               | 0.34                                 |
| After low temp heating -2 | 19                              | 0.10                                 | 5                               | 0.40                                 |

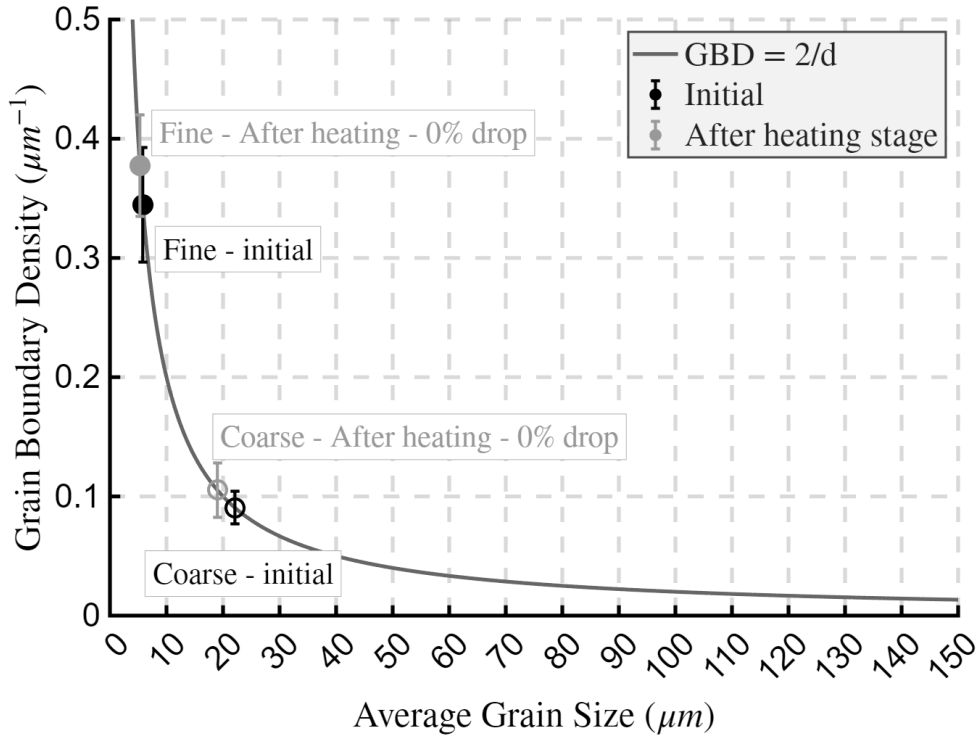


Figure 66: Grain boundary density as a function of average grain size after slow heating rate after (below 950 °C), for a coarse grain size material and a fine grain size material. [30]

The grain size evolution for both materials can be seen in Figure 67. The initial microstructure of the coarse material is shown in Figure 67(a), while Figure 67(c) shows the microstructure of the same material

after heating. On the other hand, the microstructure of the fine grain sized material before heating is shown in Figure 67(b) and the microstructure after the heating in Figure 67(d). As previously discussed, no difference in grain size was observed after the heating stage for either of the materials.

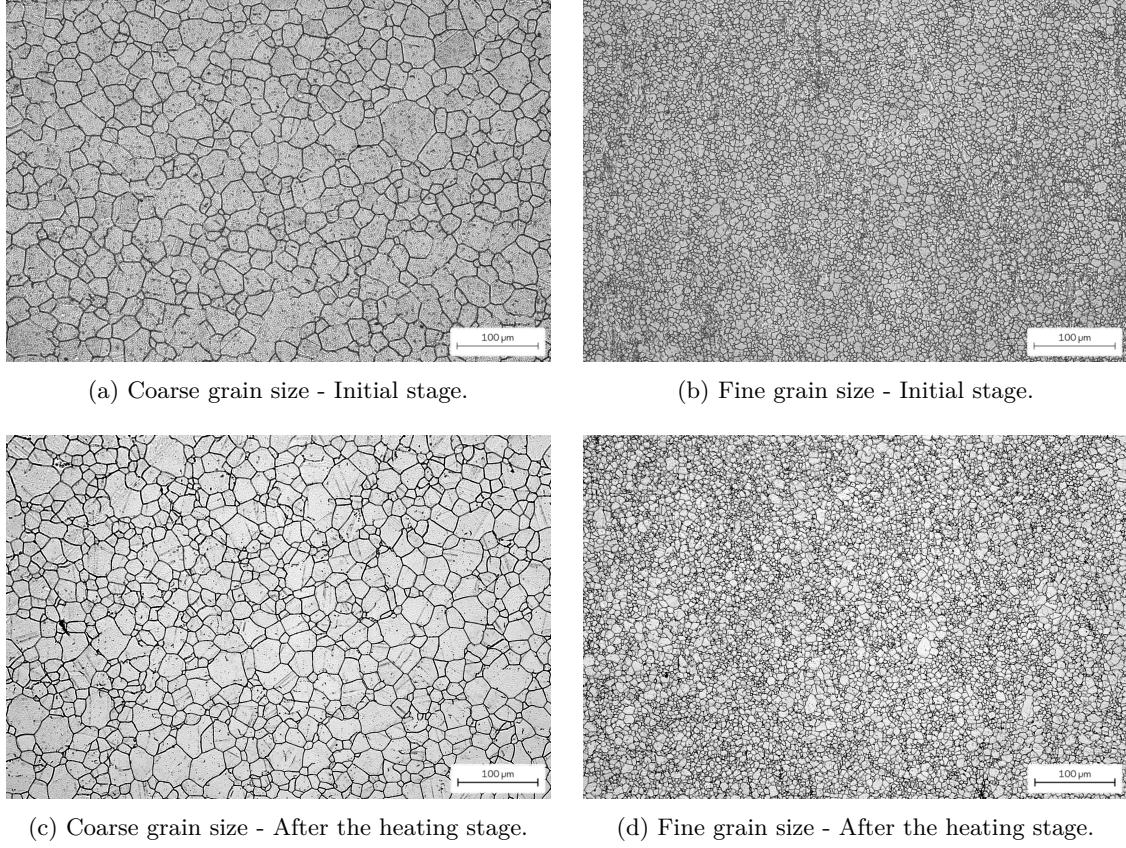


Figure 67: Microstructural grain size evolution of a coarse grain size material and a fine grain size material, after the heating stage below 950 °C. [28]

These results indicate that reducing the bonding temperature would enhance atomic migration by ensuring a higher grain boundary density in the components. Even if the grain size evolution was only tested for the heating rate stage, this result also suggests that grain size would not be further coarsened through the entire diffusion bonding process. Therefore, the grain size of the final bonded components would remain the same as at the beginning of the process. Grain boundary density is also related to the material's strength. An increase in grain boundary density prevents dislocation movement, increasing the strength of the final component [37]. Therefore, a reduction in the bonding temperature would not only promote void closure at the interface but would also potentially increase the mechanical properties of the final component.

## 5 Conclusion

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This study was divided into three different sections, and in summary, this first section shows that the applied pressure, surface roughness, and cycle time are key parameters that significantly affect the bonding quality. As expected, optimizing these factors notably reduced both the porosity ratio and the average pore size, thereby enhancing overall bonding quality. However, these were not the parameters of interest in this study, as optimizing them would result in high production costs, resource consumption, and time investment. Therefore, in order to improve bonding quality in a simpler, more cost-effective, and less time consuming way, an alternative approach was investigated. Nevertheless, this first study was useful to validate the methodology followed, demonstrating that it correctly predicted the expected bonding quality when different parameters were changed.

In the second section of this study, it was concluded that dislocations enhance bonding quality by reducing porosity ratio and the average pore size, when using lab-scale bonding cycles (Figure 44 and Figure 46). It was also observed that there is no further improvement in the joint quality over a certain amount of induced dislocation density (Figure 45). Since dislocations improve bonding quality, other key parameters, such as bonding time, temperature, and pressure, could be lowered while still getting the same porosity level. This would be beneficial for a more sustainable, cost-effective, and time-efficient production.

Apart from this, it was also observed that even though dislocations do not directly enhance GBM, they contribute to reducing the average pore size and the porosity ratio at the interface, which subsequently increases GBM ratio and therefore improves join quality (Figure 48 and Figure 49 ). The overall influence of dislocations was also summarized in Figure 53.

Finally, the effectiveness of dislocations for rougher surfaces was discussed. It was concluded that a fine enough surface finish is needed to ensure a large enough contact area between the components (Figure 54 and Figure 55), for atoms to migrate through dislocation diffusion pathways and enhance bonding quality.

In the third part of this study, longer bonding cycles were analyzed in order to assess whether dislocations would still remain in the material after a long heating stage, and if so, whether these would be sufficient to enhance diffusion bonding. First, by using the EBSD analysis, it was confirmed that dislocations remained in the components after the heating (Figure 58). Then, it was concluded that the remaining dislocation density was sufficient to reduce both the porosity and the average pore size at the interface. Apart from this, it was also observed that the combination of induced dislocations together with longer bonding cycles further improved bonding quality, by reducing both the porosity ratio and the average pore size at the interface (Figure 59), due to the extended available time for atomic migration from one component into another.

On the other hand, grain size evolution was assessed for different initial grain sizes, different bonding cycles, and temperatures. It was observed that at a high bonding temperature, regardless of having fast or slow heating rates, both materials would experience significant grain coarsening, leading to a similar average grain size and grain boundary density values (Figure 64). Furthermore, the grain size evolution of the same materials was investigated for lower bonding temperatures. In this case, it was seen that the grain size did not coarsen and that the grain boundary density remained the same after the heating stage below 950 °C. Highlighting the remaining difference in grain boundary densities between the two materials, after the heating (Figure 66). Therefore, if lower bonding temperatures were used for bonding, the material with lower initial grain size would further enhance atomic diffusion across the interface, improving bonding quality.

In summary, it was concluded that the followed methodology in the study was effective in predicting bonding quality trends. Furthermore, the presence of dislocations proved to be beneficial, increasing the

bonding quality, even for longer bonding cycles. Finally, the positive effect of lowering the bonding temperature was assessed, this would increase grain boundary density and potentially improve the bonding quality.



## 6 Future work

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This research opens many options for further investigation. In this study, only small samples were analyzed. However, in reality, larger components are bonded using longer bonding cycles and different parameters, therefore, the influence of dislocations on larger components could be further investigated. Moreover, larger components require longer heating times, resulting in the need for slower heating rates to ensure a uniform temperature distribution across the components. If the heating rate is faster than required, the bonding quality could be negatively affected, whereas if the heating rate is slower than necessary, the process would be inefficient, increasing production cost and time. Therefore, understanding time dependent heat transfer within the components during the heating stage could ensure a more optimal choice of heating rate based on the size of the components, increasing the efficiency of the process by reducing cost and production time.

Furthermore, the influence of higher grain boundary density on atomic migration and subsequently on pore closure and bonding quality could be further analyzed and quantified. This would provide justification for the use of a lower bonding temperature. Apart from this, a lower bonding temperature would not only prevent grain coarsening during the heating stage but also throughout the entire bonding process, resulting in a higher final grain boundary density. This could also be further investigated to determine how a smaller final grain size could enhance the mechanical properties in the bonded component.

Since dislocations tend to rearrange at higher temperatures and longer exposure times, lowering the bonding temperature could also partially mitigate this rearrangement, leading to a higher remaining dislocation density in the material after the heating stage. Moreover, dislocation density after different heating stages and deformation rates could be quantified. Apart from this, lowering the bonding temperature would effectively reduce the overall cycle time, thereby increasing the efficiency of the process.

On the other hand, in order to correlate bonding quality improvements with the actual joint strength, tensile tests could be conducted. So, the assessed quality methods based on porosity reduction, average pore size reduction, and grain boundary migration could be validated.

Lastly, since a production furnace was used for this study, the number of cycles was limited, and one unit of sample was measured for each combination of parameters. Therefore, increasing the number of measured units per combination would also consider standard deviation values for every single case, resulting in a more robust result and conclusion.

# Appendix

## A) Failure Mode and Effects Analysis (FMEA)

| FMEA |                                                                  |                                                          |                                                                                   |                 |                                                                                   |                   |                                                                      |                  |     |                                                                                                                                             |                                                               |                                                                                             |                 |                   |                  |     |
|------|------------------------------------------------------------------|----------------------------------------------------------|-----------------------------------------------------------------------------------|-----------------|-----------------------------------------------------------------------------------|-------------------|----------------------------------------------------------------------|------------------|-----|---------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------|---------------------------------------------------------------------------------------------|-----------------|-------------------|------------------|-----|
|      | Process Step/Input                                               | Potential Failure Mode                                   | Potential Failure Effects                                                         | SEVERITY (1-10) | Potential Causes                                                                  | OCCURRENCE (1-10) | Current Controls                                                     | DETECTION (1-10) | RPN | Action Recommended                                                                                                                          | Resp.                                                         | Actions Taken                                                                               | SEVERITY (1-10) | OCCURRENCE (1-10) | DETECTION (1-10) | RPN |
|      | What is the process step, change or feature under investigation? | In what ways could the step, change or feature go wrong? | What is the impact on the customer if this failure is not prevented or corrected? |                 | What causes the step, change or feature to go wrong? (how could it occur?)        |                   | What controls exist that either prevent or detect the failure?       |                  |     | What are the recommended actions for reducing the occurrence of the cause or improving detection?                                           | Who is responsible for making sure the actions are completed? | What actions were completed (and when) with respect to the RPN?                             |                 |                   |                  |     |
| R1   | Surface preparation                                              | Insufficient pre-treatment                               | Low dislocation density → weak/poor bonding                                       | 7               | Incorrect treatment parameters or insufficient treatment                          | 4                 | Standard operating procedures for treatments; technician supervision | 6                | 168 | Perform initial parameter screening tests; measure hardness/roughness after treatment; use replicates; consult technician                   | Uxue (Student)                                                | Measure hardness/roughness after treatment, consult technician                              | 7               | 2                 | 3                | 42  |
| R2   | Diffusion bonding cycle                                          | Incorrect temperature or pressure                        | Incomplete bonding / porosity → failed joints                                     | 9               | Equipment miscalibration or wrong setpoints / operator error                      | 4                 | Procedure checklists; logged setpoints; equipment manuals            | 3                | 108 | Calibrate furnace/press before runs; verify setpoints each run; run control samples; log all parameters and alarms                          | Uxue + Lab Technician                                         | Calibrate furnace/press before runs                                                         | 9               | 2                 | 2                | 36  |
| R3   | Diffusion bonding cycle                                          | Safety risks with press or furnace                       | Injury in the worker or furnace breakage                                          | 10              | Lack of safety training, Equipment malfunction or poor maintenance                | 1                 | Safety instructions provided in the lab, Emergency stop systems      | 5                | 50  | Mandatory safety training; follow SOPs                                                                                                      | Uxue + Lab Technician                                         | Follow SOPs                                                                                 | 10              | 1                 | 2                | 20  |
| R4   | Sample cleaning                                                  | Contamination on surface                                 | Voided and weak interface → reduced joint strength                                | 8               | Improper cleaning procedure or handling (oils, dust)                              | 2                 | Cleaning SOPs (solvents), use of gloves, clean bench                 | 3                | 48  | Standardize and document cleaning steps (acetone/ethanol); train personnel; use clean storage; perform surface energy/contamination checks  | Uxue                                                          | train personnel; perform surface energy/contamination checks                                | 8               | 1                 | 1                | 8   |
| R5   | Surface treatment                                                | Over-treatment (excess deformation)                      | Cracks / local damage → immediate failure at bond                                 | 6               | Excessive deformation intensity or indentation depth                              | 3                 | Operator experience; visual inspection                               | 4                | 72  | Optimize parameters on sacrificial samples; set treatment limits; inspect by optical microscope/SEM before bonding                          | Uxue + Technician                                             | Optimize parameters on sacrificial samples; set treatment limits;                           | 6               | 1                 | 2                | 12  |
| R6   | Post-bonding handling                                            | Sample mix-up or mislabelling                            | Incorrect data correlation → invalid conclusions                                  | 5               | Poor labeling/documentation on practice                                           | 3                 | Labeling protocol exists but informal                                | 4                | 60  | Implement strict labeling (barcode/unique ID), digital logbook, two-person check for transfers                                              | Uxue                                                          | Implement strict labeling, two-person check for transfers                                   | 5               | 1                 | 1                | 5   |
| R7   | SEM characterization                                             | Poor imaging / wrong settings                            | Misinterpretation of microstructure → wrong conclusions                           | 7               | Inexperience, wrong detector/settings, calibration issues                         | 3                 | SEM training available; technician support                           | 3                | 63  | Book SEM with experienced technician; prepare imaging checklist; run reference samples; verify calibration before imaging                   | Uxue + SEM Technician                                         | Book SEM with experienced technician                                                        | 7               | 1                 | 2                | 14  |
| R8   | Hardness testing                                                 | Indentation errors / wrong load                          | Inaccurate mechanical property results                                            | 6               | Misalignment, wrong load, or machine not calibrated                               | 2                 | Hardness SOP; occasional calibration                                 | 4                | 48  | Calibrate hardness tester; perform multiple indentations; training on loading/alignment; log test conditions                                | Uxue                                                          | training on loading/alignment                                                               | 6               | 1                 | 2                | 12  |
| R9   | Experimental design                                              | Insufficient number of parameter variations              | Inconclusive optimization; weak thesis conclusions                                | 8               | Time constraints or poor prioritization of variables                              | 5                 | Project plan / Gantt but limited buffer                              | 3                | 120 | Prioritize key parameters using DoE (fractional factorial); run pilot experiments early; reserve buffer weeks; agree scope with supervisors | Uxue + Supervisors                                            | Prioritize key parameters using DoE (fractional factorial); reserve buffer weeks            | 8               | 2                 | 3                | 48  |
| R10  | Experimental design                                              | Limited time for extensive parameter testing             | Reduced reliability of results, Incomplete understanding of parameters            | 6               | Tight project deadlines, Limited lab access, Poor initial planning of experiments | 5                 | Initial experimental plan, Supervisor guidance                       | 5                | 150 | Apply Design of Experiments (DoE) to reduce number of tests, Prioritize critical parameters                                                 | Uxue                                                          | Apply Design of Experiments (DoE) to reduce number of tests, Prioritize critical parameters | 6               | 3                 | 3                | 54  |
| R11  | Equipment availability                                           | scheduling conflicts                                     | Delays in experiments and loss of time (timeline slip)                            | 7               | High demand for shared equipment; maintenance                                     | 4                 | Equipment booking system, some advance booking                       | 3                | 84  | Book equipment well in advance; arrange alternate lab/facility access; cross-train staff; schedule contingency slots                        | Uxue + Company / Lab Manager                                  | Book equipment well in advance                                                              | 7               | 2                 | 2                | 28  |
| R12  | Equipment availability                                           | Equipment downtime                                       | Delays in experiments and loss of time (timeline slip)                            | 7               | High demand for maintenance, Equipment overuse                                    | 3                 | Maintenance schedule, Booking system for equipment use               | 4                | 84  | Plan experiments with buffer time, Improve preventive maintenance scheduling                                                                | Uxue + Lab Technician                                         | Plan experiments with buffer time                                                           | 7               | 3                 | 2                | 42  |

Figure 68: Failure Mode and Effects Analysis (FMEA) [6]

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