DEVELOPMENT AND INTERFACIAL ANALYSIS OF DIFFUSION BONDINGS BETWEEN COPPER AND STAINLESS STEEL FOR APPLICATIONS IN ULTRA-HIGH VACUUM

Bagnato, O. R., osmar@lnls.br
Francisco, F. R., fernanda@lnls.br
Rosales, M. J. C., marcio.rosales@lnls.br
Manoel, F. E., fmanoel@lnls.br
Brazilian Synchrotron Light Laboratory, Materials Group, P.O. Box 6192, Campinas – SP, Brazil.

Abstract. *Diffusion bonding is described as a welding process of permanent joints by establishing interatomic bonds between workpieces. The process can be applied for dissimilar joint materials, even in solid state. This present work aims to acquire a technology capable to be used in ultra-high vacuum environments, such as chambers, beamline and furnaces, which allows, thus, the use in cooling systems and guarantees better contact between both OFHC copper and AISI 316L austenitic stainless steel. Workpieces are compressed by uniaxial force according to materials cross section area. ´Stainless steel and copper materials were cleaned for ultra-high vacuum environment, assuring that their surface was free of oiliness and impurities. So, samples were bonded in high vacuum environment using pressure below 10^{-4} mbar and maximum temperature around three quarters of copper's melting point. The process was held at this temperature during a time between 30 and 60 minutes. The heating curve was set to 3-6 ºC/min, depending on the pressure, and the cooling curve to 10ºC/min inertial. Polished and unpolished samples were analyzed by optical microscopy, helium leak test and mechanical properties tests, in terms of tensile and microhardness. The microstructure interdiffusion between both materials revealed a sealed structure to operate in ultra-high vacuum (tests in simulated pressure of 10^{-10} mbar did not reveal any leak). The tensile tests evidenced a maximum strength of 172 MPa. These samples broken in OFHC copper region, but not in the interface between both materials, which means that the bonded joint guarantees a mechanical resistance higher than the copper base material. The interface was also observed by scanning electron microscopy and energy dispersive X-ray spectroscopy. Scanning Electronic Microscopy (SEM) was conducted in Jeol 5900 equipment using LME facilities. These analyses show the diffusion of iron, nickel and copper, respectively, inter the bonding interface. Analyzing the interface, it is possible to note some discontinuities between both materials which do not interfere in the vacuum environment. It was possible to evidence the percentage of iron and copper during the scanning, since 7.5 microns in the iron from the interface through 15 microns.*

Keywords: *Diffusion bonding; stainless steel; copper; ultra-high vacuum.*

1. INTRODUCTION

Diffusion bonding is a solid-state welding process that allows contacting surfaces to be joined under pressure at elevated temperatures (Batra, et al, 2004 and Barrena, et al, 2009). Almost all materials with compatible chemical and metallurgical properties can be joined by the diffusion bonding process (Sabetghadam et al, 2010) The process produces joints with properties and characteristics that are singular of the interatomic diffusion. The principle welding parameters that rule the process are temperature, pressure and diffusion time. It is important to notice that this is a thermally activated process, which both diffusion coefficients materials to be bonded play an important role to produce a sound joint.

The main problem of bonding dissimilar materials such as stainless steel and copper alloys by conventional welding process lies on their different melting temperature and especially because of unexpected phase propagation at the bond interface. Furthermore, diffusion bonding is preferred for the materials in which brittle phase formation is unavoidable (Yilmaz and Çelik, 2003). Also, solid-state joining techniques such as diffusion bonding process are a natural choice since welds are produced at low temperatures thus the low heat input provides limited distortion and residual stresses; and microstructural as well as mechanical degradation of materials (Aleman et al, 1993). In industrial applications, the demand for joints of copper alloys and stainless steel has increased due to the advantages obtained from the properties of both material, such as high electrical conductibility of copper alloys and chemical resistance of stainless steels. This combination of joints would be expected to prove highly beneficial in many applications, from automobile, rail and aviation industries to smaller, more commonly used products, such as saucepans (Salehi, 1990).

This present work investigates interfaces of diffusion bonded dissimilar joint of Oxygen Free High Conductibility (OFCH) copper and AISI 316L stainless steel. The welding joint microstructure was examined and the elements distribution at the interface was determined. Strengths and mechanical properties of the joints were also determined in terms of tensile test and microhardness indentation measurements. These studies resulted in an interesting set of observations relevant to the use of such bonded structures in ultra-high vacuum application. Also, the results indicated the influence process parameters on the production of a sound dissimilar joint.

2. EXPERIMENTAL PROCEDURE

AISI 316L austenitic stainless steel and ASTM B170, grade B, OFHC copper materials were used as base materials configuring the diffusion bonding dissimilar joint. Specimen mating surfaces were prepared by conventional cleaning techniques with ultrasonic equipment using alkaline detergent bath to remove adhered contaminants and oiliness, and then dried in air. AISI 316L copper assemblies were placed in a vacuum chamber of the diffusion bonding furnace (see Figure 1). The materials to be bonded were heated up to 850°C and held by 30-60 minutes at this temperature. The heating ramp turned to 3-6 °C/min, and cooling to 10 ºC/min inertial. The uniaxial compression load of 10 MPa was applied along the longitudinal direction of the workpieces to hold them in contact during the welding process. The environment pressure was set to work at high vacuum of 10^{-5} mbar aiming to guarantee the absence of oxygen that would create an oxide barrier at the joint interface and also to avoid oxidation of the pieces and furnace components.

Figure 1. Experimental set up and diffusion bonding furnace.

For metallurgical characterization, metallographic investigations were conducted on bonded materials from transversal specimens of the dissimilar joint and prepared by conventional metallographic, grinding and polishing techniques. For examining the microstructural features in the bonded and surrounding zones, optical microscopy was carried out. The distribution of various elements across the bonded zones was performed with scanning electron microscopy (SEM). SEM micrographs were taken with a JEOL JMS 5900 operating at 20 keV and fitted for energy dispersive X-ray spectroscopy (EDS). Also, mechanical properties of the bonded materials were evaluated in terms of tensile strength and microhardness indentation measurements across the interface. Tensile tests were investigated at room temperature using a universal tensile strength machine on flat dissimilar specimens. The microhardness measurements were realized at the bonded interface using Shimadzu equipment under HV_{10g}. Finally, the bimetallic component of AISI 316L stainless steel and copper diffusion bonded (see Figure 2) was tested to operate in ultra-high vacuum environment using a Pfeiffer Quality Test Dry leak detector machine with helium gas at the 10^{-10} mbar.l/s.

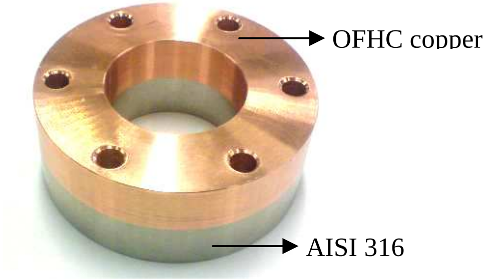


Figure 2. Diffusion bonded AISI 316L to OFHC copper specimen machined for leak test.

3. RESULTS AND DISCUSSIONS

3.1. Metallographic investigations

Microscopically analyses through Energy Dispersive Spectroscopy (EDS) were performed as shown in Figure 3. Diffusional processes, which are activated thermo-mechanically, caused a formation of a new interphase or reaction zone due to diffusion of alloy elements of both materials, especially of Ni element. Thus, the qualitative analysis of the diffusion area was carried out on the interface between copper and stainless steel dissimilar joint. It is possible to notice that the thickness of the reaction zone is less than 1 µm, once the intersection of 50% weight of Cu (green) and Fe (red) curves, showed in Fig. 3, represents a minimal micron distance.

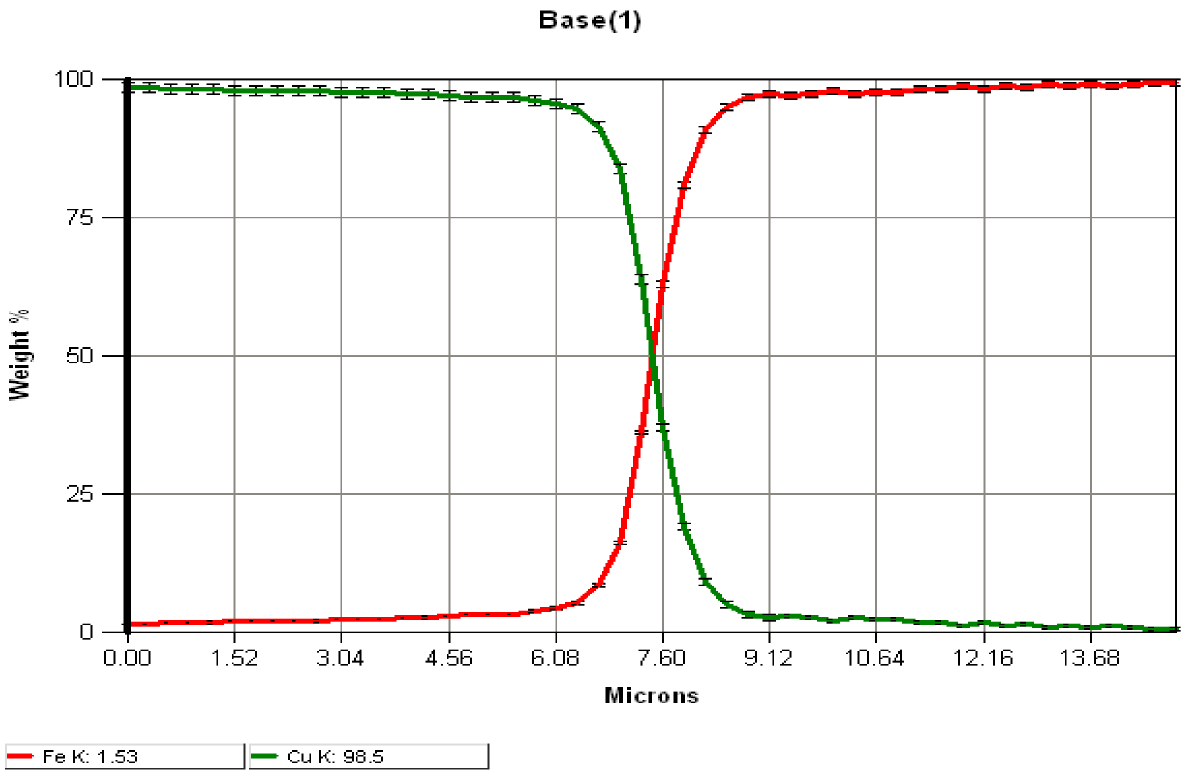
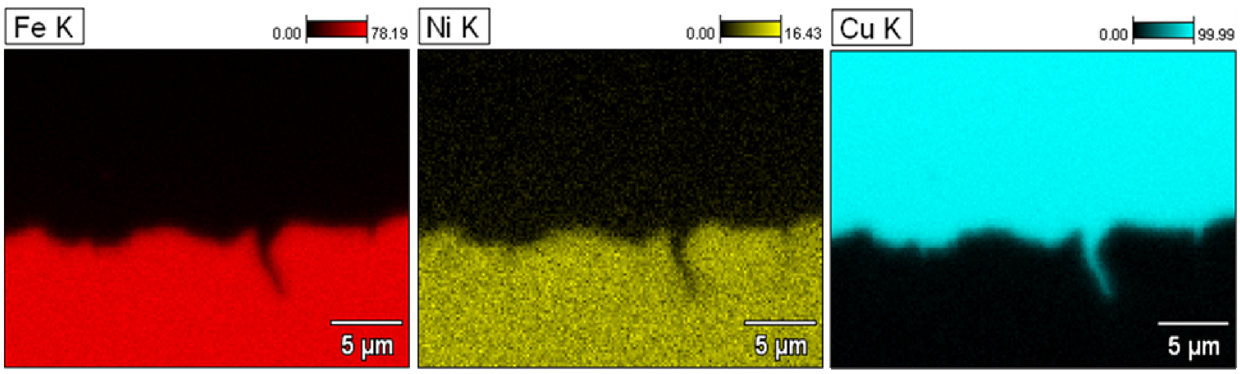


Figure 3. EDS diffusion analysis on interface of copper and stainless steel joint.

Figure 4 shows the concentrations according to the penetration profiles of Fe, Cu and Ni across the bonded interface. As the diffusion process is associated with mass transportation and because Ni has a high diffusivity coefficient on Cu, Ni element was highly diffused into the Cu base material (Nishi, et al, 1997 and 1998). Concentrations of both metals vary with position. This result indicates that Ni atoms have migrated into the Cu. This process, whereby atoms of Ni diffuse into Cu, occurs over time. There is a net drift of atoms from high (stainless steel) to low-concentration (copper) regions.



(A) (B) (C)

Figure 4. EDS of (A) Fe (red) and Cu (black); (B) diffusion between Ni and Cu and (C) detail of Cu in the interface.

Figure 5 shows the typical microstructure and microhardness profile measurements of dissimilar stainless steel and copper joint transition area. It can be observed that the interdiffusion zone has occurred smoothly between both base materials. Also, the bonding interface is planar and thin. The microhardness of the diffusion layer in stainless steel side is higher than that of Cu, which is due to the softer nature of copper alloys. It is possible to notice that the microhardness indentation is bigger than the thickness of the bonded interface, which means that the characterization of the interface mechanical properties has to be measured using nanohardness indentation. The joints appear to be sound, with no porosities or any presence of oxide and impurities elements.

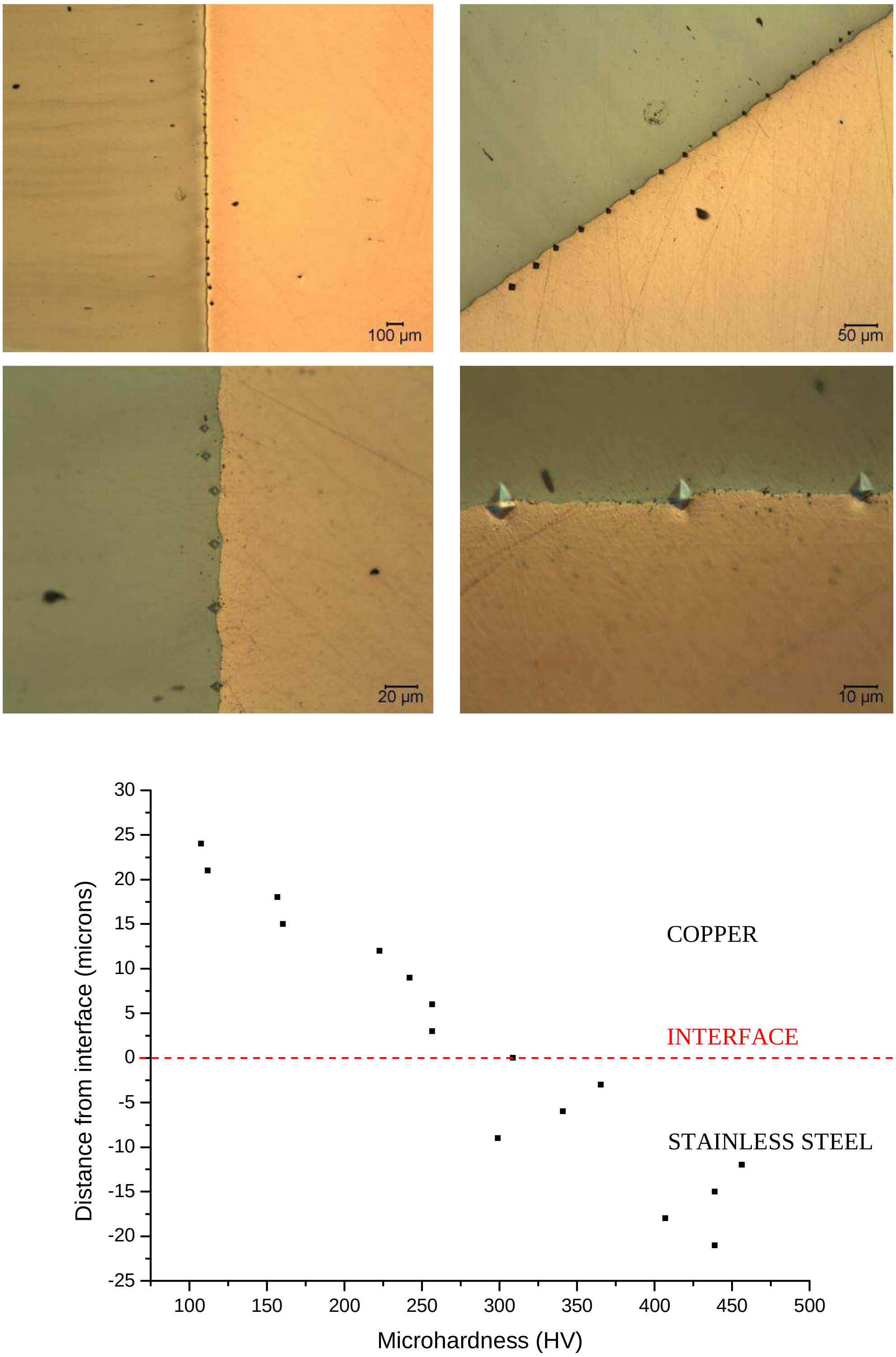


Figure 5. Microstructural characterization and microhardness profile across the diffusion interface.

3.2. Tensile tests and leak analysis

Figure 6 shows the engineering tensile tests stress-strain curves for five bonded specimens, which revealed a strong resistant bond between copper and stainless steel. All curves are displaced from origin in 10 units to facilitate the graphic understanding. The ultimate tensile occurred outside the bonding interface, which leads to the conclusion that the welding processes were succeeded (see Figure 7). The dependence of diffusion parameters, such as temperature, pressure and time, on bond specific strength, is clearly evident to result a sound mechanical property joint. In other words, the mechanical testing results showed that the optimum conditions for bonding copper and austenitic stainless steel by diffusion process are temperature of 850°C for a working pressure of 10 MPa and soaking times of 30-60 min. Also, the tensile tests detected a maximum strength of 172,5MPa (see Figure 6), that is coherent to the mechanical properties of copper base material from literature review.

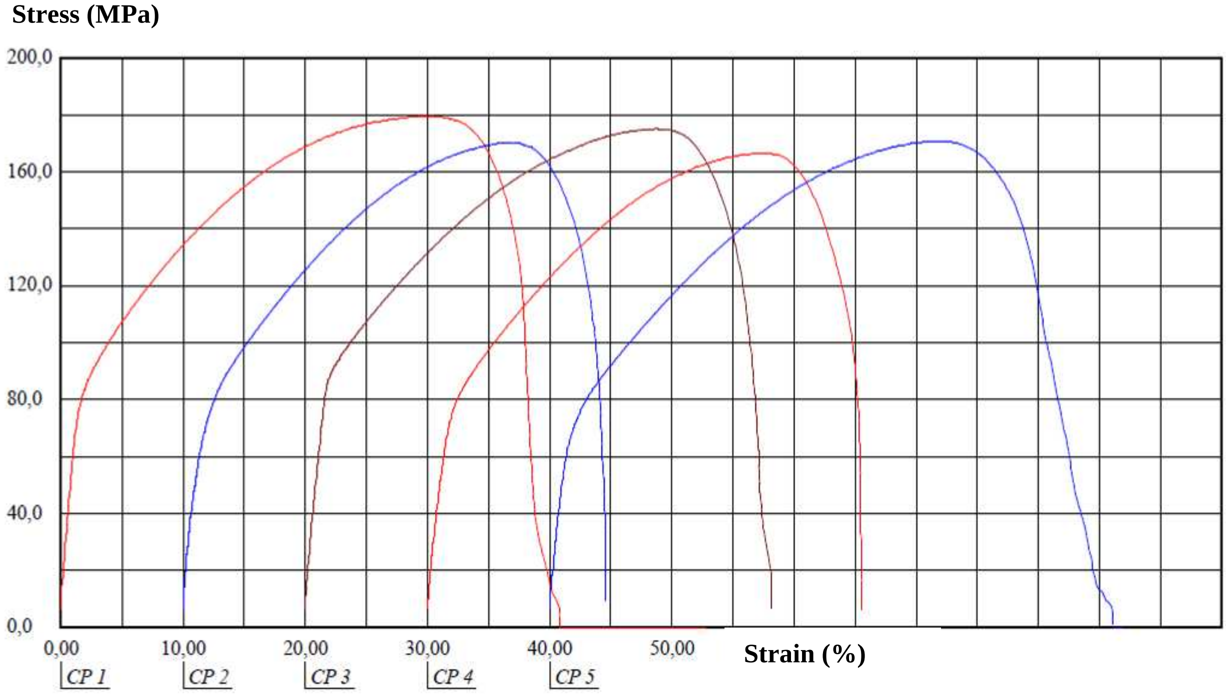


Figure 6. Stress-strain curves of five diffusion bonded specimens submitted to tensile tests.

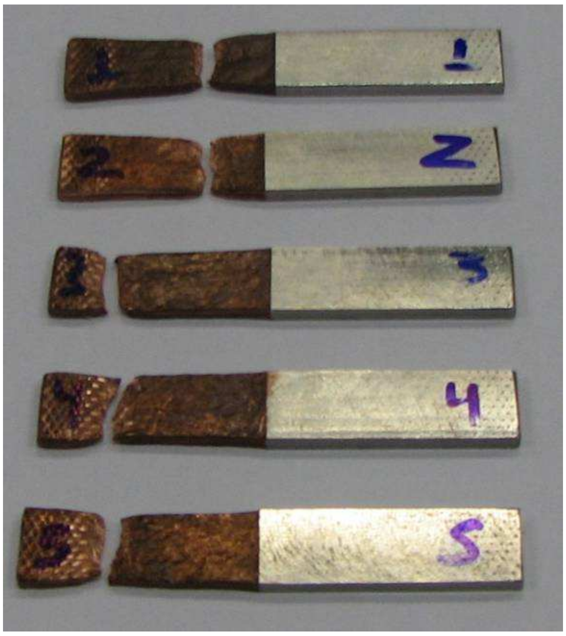


Figure 7. Diffusion bonded specimens after tensile test, resulting in a rupture in copper base material.

It is possible to observe in Figure 7 that all tests specimens resulted in a weld joint stronger than the copper base material. This is despite the presence of the reaction zone at the joint interface, which can be observed in Fig. 4. Even though copper is soft and ductile, the presence of the interface as a continuous layer in the reaction zone is not effective in lowering the strength of the joints due primarily to their extremely small thicknesses. The fracture at these thin layers is prevented from taking place due the development of stresses because of the plastic constraint during tension. The stresses reduce the available stress for plastic deformation so much that the fracture takes place in the base material, as it occurs in copper, which is softer than stainless steel.

The leak tests were completed in several points of the diffusion bonded interface, revealing an excellent tightness for ultra-high vacuum environments application across all specimens analyzed.

4. CONCLUSION

OFCH copper base material was bonded to AISI 316L austenitic stainless steel during solid-state diffusion process using optimal operating conditions. This was carried out at 850°C for 30-60 minutes under a 10 MPa uniaxial compression load in high vacuum environment. Microscopy analyses revealed the interdiffusion of Ni and Cu elements. It is evident Ni element diffusivity on copper base material due to low concentration of this element in copper matrix and high Ni diffusivity coefficient. Further tests using transmission electron microscope will be completed to better characterize the diffusion interface, inclusive in terms of Ni distribution along the interface distance.

Tensile tests showed strong and high-quality results. The interface strength is higher than copper base material, which means that the mechanical properties of dissimilar joints are suitable for mechanical applications. Microhardness measurements were inconclusive due to indentation magnitude that is bigger than the interface reaction zone. Further tests will be investigated using a nanohardness scale to guarantee the bonding mechanical interface characterization.

Finally, the dissimilar diffusion bonded specimens revealed a complete leaked component to operate in ultra-high vacuum environments, such as in synchrotron particles accelerator.

5. ACKNOWLEDGEMENTS

The authors would like to gratitude the financial support from FINEP (Financiadora de Estudos e Projetos) and for the technical and infrastructural assist from LNLS (Laboratório Nacional de Luz Síncrotron), specially LME (Microscopy Laboratory from CNPEM).

6. REFERENCES

Aleman, B. Gutierrez, I. Urcola, J. Interface microstructures in diffusion bonding of titanium alloys to stainless and low alloy steels, Mater. Sci. Technol. 9 (1993) 633–641.
Barrena, M. I. Salazar, J. M. G. Matesanz, L. Ni–Cu alloy for diffusion bonding cermet steel in air, Materials Letters 68 (2009) 2142-2145.
Batra, I. S. et al. Diffusion bonding of a Cu-Cr-Zr alloy to stainless steel and tungsten using a nickel as an interlayer, Materials Science and Engineering A269 (2004) 119-123.
Dieter, G. Mechanical Metallurgy, McGraw-Hill, New York, 1988.
Nishi, H. Araki, T. Eto, M. Diffusion bonding of alumina dispersion-strengthened copper to 316 SS with interlayer metals, Fusion Engineering and Design 39-40 (1998) 505-511.
Nishi, H. Kikuchi, K. Influence of brazing conditions on the strength of brazed joints of alumina dispersion strengthened copper to 316 stainless steel, ICFRM-8, Sendai, Japan, 1997.
Sabetghadam, H. Zarei Hanzaki, A. Araee, A. Hadian, A. Microstructural evaluation of 410 SS/Cu diffusion bonded joint, J. Mater. Sci. Technol. (2010) 163-169.
Sabetghadam, H. Zarei Hanzaki, A. Araee, A. Difffusion bonding of 410 stainless steel to copper using a nickel interlayer, Materials Characterization 61 (2010) 626-634.
Salehi, M. T. Isostatic diffusion of some super plastic alloys, Ph.D. Thesis, UMIST, Manchester, 1990.
Yilmaz, O. Aksoy, M. Investigation of Micro-crack occurrence conditions in diffusion bonded Cu-304 stainless steel couple, Journal of Materials Processing Technol. 121 (2002) 136-142.
Yilmaz, O. Çelik, H. Electrical and thermal properties of interface at diffusion-bonded and soldered 304 stainless steel and copper bimetal, Journal of Materials Processing Tech. 141 (2003) 67-76.