

DEVELOPMENT OF DIFFUSION BONDED JOINTS OF AA6061 ALUMINUM ALLOY TO AISI 316LN STAINLESS STEEL FOR SIRIUS PLANAR UNDULATORS

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Abstract

LNLS has been commissioning Sirius, a 4th-generation synchrotron light source. The commissioning of the beam-lines has been mainly done by using planar undulator, which uses in-house built aluminum vacuum chambers with ultra-high vacuum tight bimetallic flanges. In order to manufacture these flanges, diffusion bonded joints of AA6061 aluminum alloy to AISI 316LN stainless steel were developed. Diffusion bonding was carried out at 400-500°C for 45-60 min, applying a load of 9.8MPa in a vacuum furnace. Also, the surface preparation for Al and SS was investigated. SEM observation revealed that an 1-3 μm reaction layer was formed at the AA6061/Ni-plated interface. The intermetallic compound Al_3Ni was identified in the reaction layer. The obtained Al/SS joints showed mean ultimate strength of 94 MPa, with the fracture occurring in the Al/reaction layer interface. Bake-out cycles followed by leak tests were carried out to validate the process and approve their use on the planar undulator vacuum chambers. Two undulators with Al/SS flanges have been installed and are under operation in the storage ring.

INTRODUCTION

LNLS (Brazilian Synchrotron Light Laboratory) staff is currently commissioning Sirius, the Brazilian 4th generation synchrotron light source. The commissioning of the beamlines has been mainly done by using planar undulator, which uses in-house built aluminium vacuum chambers with ultra-high vacuum tight bimetallic flanges of aluminium and stainless steel (SS). Figure 1 shows the virtual design of the planar undulator and the bimetallic flange.

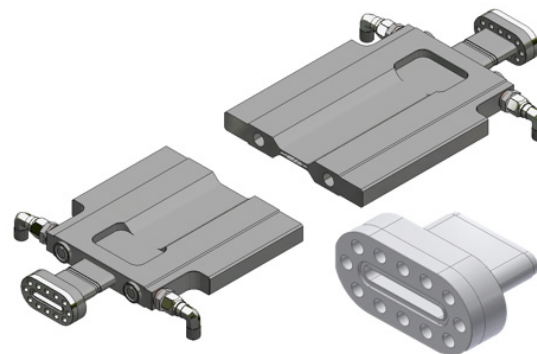


Figure 1: Virtual design of undulator and bimetallic flange.

Due to the difficulties of joining Al alloys and SS by usual methods for vacuum applications, and considering the capabilities and expertise of LNLS, it was chosen the diffusion bonding process to manufacture these bimetallic flanges. Diffusion bonding is a special type of welding that involves the interdiffusion of atoms across the interface of the weld in solid and, sometimes, the liquid state [1].

The main challenge of joining Al alloys by diffusion bonding lies on its high chemical activity [1]. To overcome this obstacle, the Al alloy surface should be properly prepared, whether by etching and passivation; by the use of an interlayer of some appropriated metal; by the deposition of some appropriated metal; or by a combination of these techniques [2]. Moreover, the SS should be electroplated with Ni or Cu in order to enhance its chemical activity [3].

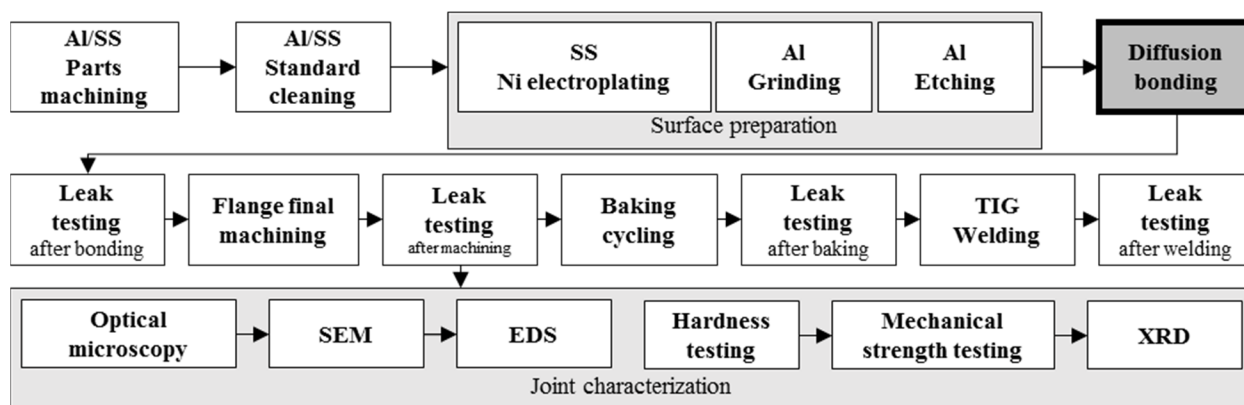


Figure 2: Fabrication and characterization flowchart.

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OBJECTIVE

This work presents the development of a manufacturing process of an ultra-high vacuum-tight diffusion bonded joint of AA6061 Al alloy to AISI 316LN SS, as well as the characterization of the process and final parts.

METHODOLOGY

Manufacturing and welding process, as well as characterization methods followed the flowchart shown in Fig. 2.

Pre-diffusion Bonding Routine

Parts Machining The machining process was done by LNLS' mechanical workshop. Figure 3 shows the virtual design of Al and SS parts.

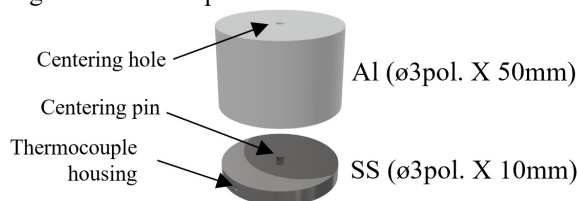


Figure 3: Virtual design of Al and SS parts.

Standard Cleaning The machined parts were cleaned using an alkaline detergent with a soft sponge. After that, parts were submitted to an ultrasonic bath for 15 minutes, with the same detergent. In the end, parts were rinsed with water and dried with nitrogen (N₂) gas flow.

Surface Preparation For the Al alloy, each part was grinded with 600-grit size grinding paper. After that, parts were submitted to standard cleaning again. Subsequently, the parts were etched with a 5% hydrofluoric acid (HF), 10% nitric acid (HNO₃) aqueous, and then rinsed with water and dried with a N₂ gas flow. An alternative solution of Keller reagent (1% HF; 2,5% HNO₃; 1,5% hydrochloric acid (HCl)) was also tested. For the SS, each part was electroplated with a 5 µm layer of nickel in a two-step procedure. After that, parts were rinsed with deionized water and dried with a N₂ gas flow.

Diffusion Bonding Procedure

The welding process took place in a vacuum furnace and with a built-in hydraulic press, which allows to apply heat and load, simultaneously. The inner atmosphere was 5.0E-5 mbar or lower.

The three main parameters of the diffusion bonding process are: working temperature, soaking time and applied load. For all performed tests, the applied load was 9.8 MPa and it was applied only during the soaking time. Five different working temperatures were used: 400 °C, 425 °C, 450 °C, 475 °C and 500 °C. The soaking time is the time in which both heat and load were applied to the parts. Tests were performed with 45 min and 60 min. All parameters used are summarized in Table 1.

Table 1: Parameters of Diffusion Bonding Process

Condition	Working Temperature [°C]	Soaking Time [min]	Al Alloy Surface Preparation
1	400	60	Al etchant
2	400	60	Keller reagent
3	425	45	Al etchant
4	425	45	Keller reagent
5	450	45	Al etchant
6	450	45	Keller reagent
7	450	60	Al etchant
8	475	45	Al etchant
9	500	45	Al etchant

Leak Testing and Final Flange Fabrication

Leak Testing Leak testing was performed after the welding and after each step of the flange fabrication, which are: flange machining; baking cycling and TIG welding.

Baking Cycling The vacuum baking cycling was used to validate the usage of these flanges by simulating the real conditions that it will be submitted during Sirius operation and NEG activations. It consisted in the parts warm up at 1 °C/min until 180 °C, followed by a 24-hours soaking time and then cool down to room temperature.

TIG Welding It was the final step of flange fabrication and consisted in the TIG welding of the flange to the Al vacuum chamber.

Diffusion Bonded Joints Characterization

Metallography To make the microstructural analysis of the diffusion bonded joint feasible, some samples were submitted to metallographic preparation.

Optical Microscopy (OM) This technique was used to inspect the joint and the presence of porosity and gaps.

Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) Using these techniques, it was possible to evaluate the joint microstructure and reaction layer thickness and to qualitative inspect the existence of atomic diffusion in the interface.

Hardness Testing In order to evaluate the effect of the working temperature in the aluminium mechanical properties, hardness tests were conducted.

Mechanical Testing The objective of this mechanical test was the evaluation of the ultimate tensile strength of the diffusion bonded joint.

X-Ray Diffraction (XRD) After the mechanical test, XRD was performed in both sides of the samples fracture surface to identify the reaction layer structure.

RESULTS AND DISCUSSIONS

Leak Tests

Leak tests showed that the joints welded in working temperatures below 450 °C presented leak at same point of the fabrication process. Also, joints that used Keller reagent were not approved. Finally, 45 minutes proved to be enough soaking time to assure tight joints.

The following analysis were performed only in the three best conditions: 5, 8 and 9 presented in Table 1.

OM, SEM and EDS Analysis

The OM images showed that all conditions analysed resulted in sound joints with no porosity or voids. The SEM analysis showed the Ni electroplated layer and the reaction layer that is formed during the diffusion bonding process. It can be seen in Fig. 4 that the higher the working temperature, the thicker the reaction layer. EDS analysis of the joints made explicit the atomic diffusion phenom and revealed that the reaction layer is made up mainly of Al and Ni, in nearly 50:50 proportion. Figure 4 shows the OM images and scanline of EDS for condition 8 sample.

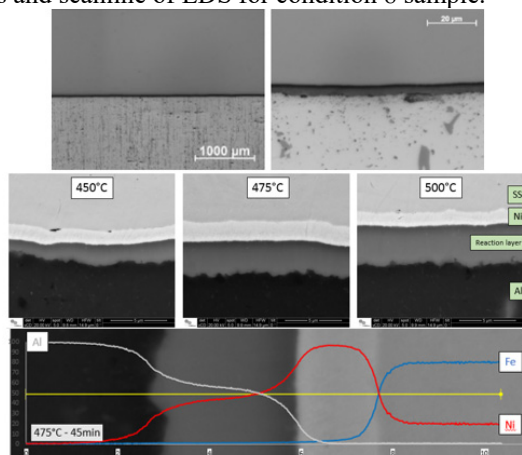


Figure 4: OM of cond. 8 sample, i.e., 475 °C and 45 min; SEM of cond. 5, 8 and 9, i.e., 450, 475 and 500 °C; and EDS of cond. 8 sample.

Hardness and Mechanical Strength Tests

The result of the Al hardness test showed that above 400 °C, the Al is fully annealed. It means that the base materials will present the same mechanical properties for working temperatures between 400 and 500 °C.

The mechanical tests showed that the joint bonded at 450 °C presented the best result, showing a mean ultimate strength of 94 MPa. It corroborates a previous study from Kuroda [3] that showed that a reaction layer of 1-2 µm is the best for mechanical strength. Figure 5 shows the mechanical test results.

X-Ray Diffraction

X-Ray diffraction results indicates that the reaction layer is formed by the intermetallic Al_3Ni and that the fracture happened in the interface between the aluminum and the reaction layer. Figure 6 shows the diffractogram.

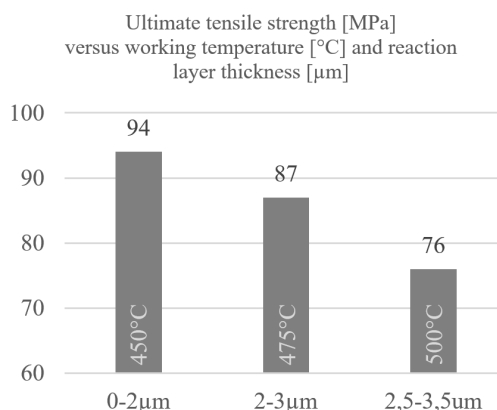


Figure 5: Joints tensile strength vs working temperatures.

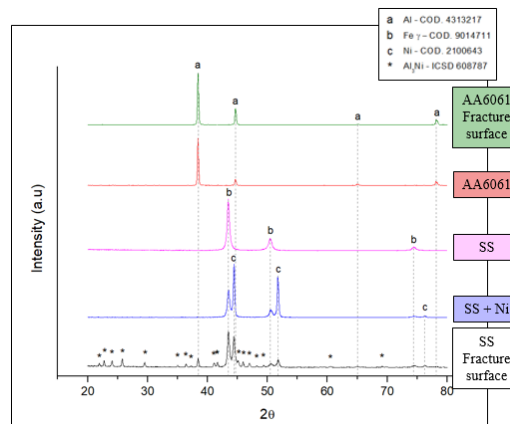


Figure 6: Diffractogram of Al and SS fracture surfaces.

CONCLUSIONS

A manufacturing process of an ultra-high vacuum-tight diffusion bonded joint of AA6061 to AISI 316LN was successfully developed.

The best parameters found for the welding were: working temperature of 450 °C; soaking time of 45 minutes; and applied load of 9,8 MPa. Additionally, the best surface preparation for the Al alloy was achieved when using an 5% HF, 10% HNO_3 aqueous solution etchant.

SEM observation revealed that an 1-2 µm reaction layer was formed at the AA6061/Ni-plated interface; and the intermetallic compound Al_3Ni was identified in the reaction layer. The obtained Al/SS joints showed an average ultimate strength of 94 MPa, with the fracture occurring in the Al/reaction layer interface.

The flanges were approved in bake-out cycles followed by leak tests and two undulators with Al/SS flanges have been installed and are under operation in storage ring.

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REFERENCES

- [1] N. F. Kazakov, *Diffusion Bonding of Materials*. Moscow, Russia: Pergamon Press, 1985.
- [2] K. Nishimoto *et al.*, “Removing of surface oxide film of SUS316 and improvement of diffusion-bondability of A6061 Aluminum Alloy to SUS316 Stainless Steel using surface-activated pre-coating technique,” *Quartely Journal of the Japan Welding Society*, vol. 18, no. 4, pp. 563-571, 2000.
doi:10.2207/qjjws.18.563
- [3] S. Kuroda *et al.*, “Microstructure and properties of directly bonded joint of A6061 Aluminum Alloy to SUS316 Stainless Steel,” *Quartely Journal of the Japan Welding Society*, vol. 17, no. 3, pp. 484-489, August 1999.
doi:10.2207/qjjws.17.484