

# Improvement INCOLOY Alloy 800 Weldability After 10 Years of Service Through Solution Annealing and Normalizing Method

Amir Arifin, Gunawan\* and Jaya Rizki Saputra

*Department of Mechanical Engineering, Sriwijaya University, Indralaya, Sumatera Selatan, Indonesia.*

\* Corresponding Author: gunawan@unsri.ac.id

**Submitted :** 15-12-2021

**Revised :** 11-03-2022

**Accepted :** 21-03-2022

## ABSTRACT

INCOLOY alloy 800 is widely utilized in the outlet manifold bottom header (OMBH) inside a primary reformer at a fertilizer plant. Primary reformers in fertilizer plants usually work at high temperatures and pressures for long periods. In this work, solution annealing and normalization processes were conducted to improve the weldability of INCOLOY alloy 800 after operating for ten years. Characterizations of weldability properties were carried out through dye penetrant, X-ray fluorescence, hardness, X-ray diffraction (XRD), and metallography observations. The annealing and normalization results showed that the weldability of INCOLOY alloy 800 increased, indicating a significant reduction in cracks on the weld zone surface. Microstructure observations revealed that microcracks were not found on the weld joint surface after solution annealing. The maximum hardness value on the fusion zone was obtained at 187 VHN on normalizing, and the minimum hardness was obtained on the nontreatment sample 156.33 VHN.

**Keywords:** Weldability, INCOLOY Alloy 800, Annealing, Normalizing.

## INTRODUCTION

INCOLOY alloy 800 is super austenitic stainless steel. Nickel, chromium, and iron are the base metals, with molybdenum, copper, nitrogen, and silicon serving as additions. In the petrochemical, chemical, and power generation industries, INCOLOY alloy 800 is commonly utilized in high-temperature applications (Yamawaki, Mito, et al. 1977, Dehmolaei, Shamaian, et al. 2008, Tan, Jiang, et al. 2011). These alloys are recognized for their high strengths at high temperatures and high corrosion resistance under extreme conditions (Persaud, Ramamurthy, et al., 2016). Even after prolonged exposure to high temperatures, the alloy can remain stable and maintain its austenitic structure. The alloy has strong resistance to oxidizing, reducing, and aqueous conditions.

INCOLOY alloy 800 is a solid-solution austenitic alloy with titanium nitrides, titanium carbides, and chromium carbides that are widespread in its microstructure. The nitrides are unaffected by heat treatment since they are stable at temperatures below the melting point. Austenitic alloys are sensitized to intergranular corrosion in specific aggressive environments by exposure to 540–760 °C (Kou 2003, Sahlaoui, Makhlouf, et al. 2004, Tan, Jiang et al. 2011, Gunawan and Arifin 2021). Outlet manifold bottom header (OMBH), which has been operating for ten years under extreme conditions, has certainly experienced sensitization.

The high chromium concentration in the  $M_{23}C_6$  phase decreases the chromium content in the area surrounding this chromium-rich deposit due to chromium diffusing considerably slower than carbon. The chromium concentration near the grain boundaries drops to below 13%, which is a critical value for the corrosion resistance required (Srinivasan, N. 2021, Sahlaoui, Makhlouf, et al. 2004). INCOLOY 800 may experience intergranular corrosion and intergranular stress corrosion cracking under corrosive conditions due to sensitization (Tan, Jiang, et al. 2011, Arifin, Gunawan, et al. 2020).

The primary reformer is a reactor in a fertilizer plant operated at a temperature of 600–800 °C and a pressure of 30–40 kg/cm<sup>2</sup>. The reformer breaks down hydrocarbon gas into hydrogen. This process occurs at high temperatures and pressures (Alvino, Lega et al. 2010). One of the essential parts of the primary reformer in fertilizer plants is the outlet manifold bottom header (OMBH).

OMBH is manufactured using INCOLOY alloy 800, and it has been operating for ten years. The OMBH is subjected to high-temperature heat during operation, changing the microstructure and generating residual stress (Tavassoli and Colombe 1978, Karimi, Riffard, et al. 2008).

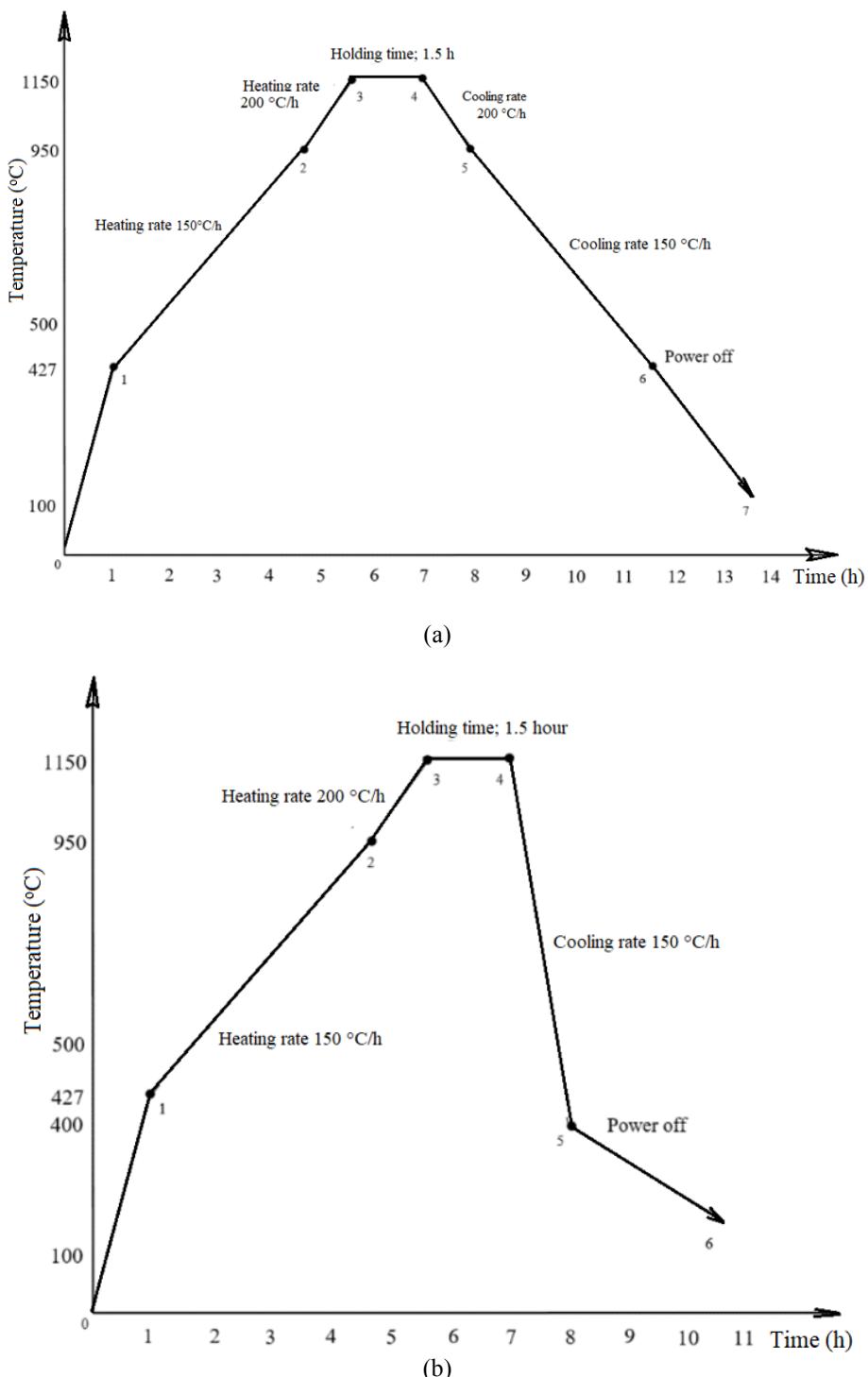
The purpose of this study is to investigate the weldability properties of INCOLOY alloy 800 materials after operating for ten years after the heat treatment process is carried out. Destructive and non-destructive tests are carried out to obtain the data needed to analyse the final result of the heat treatment. These tests include hardness testing, XRF, XRD, penetrant testing, weldability testing, and metallographic testing using an optical microscope.

## METHODOLOGY

The material used during this research process came from the outlet manifold bottom header (OMBH) component (Figure 1). The material operated at high pressures and temperatures and was categorized as a superalloy type—alloy 800—with resistance to high pressures, high temperatures and corrosion. The INCOLOY alloy 800 used by a fertilizer plant for the OMBH component produced by Manoir Industries was M900. Furthermore, the material composition was investigated through the X-ray fluorescence analyser Niton XL 2. Welding was performed using gas tungsten arc welding (GTAW) with a filler rod Er NiCrMo-3. Liquid penetrant testing was conducted before and after welding to reveal open surface welding defects. In this work, the weldability sample was measured to refer to the effect of welding on indications of defects or crack appearance after the dye penetrant test. A weldability test was performed before and after the welding process. Heat treatment for OMBH was carried out through solution annealing, and the normalizing process referred to the Association of Mechanical Engineering (ASME) standards for INCOLOY alloy 800 material. A schematic diagram for heat treatment is shown in Figure 2. The measurement of the hardness value in the weld area was carried out using the Vickers method with a diamond indenter to obtain better accuracy. The Vickers method was performed with a load of 20 kg based on the JIS Z 2244 standard with three repetitions. X-ray diffraction was conducted on the welding position to investigate phase formation. Furthermore, metallography analysis was conducted using an etching fluid (20 ml HNO<sub>3</sub> and 80 ml HCl) and optical microscope, referring to the ASTM E-407 standard.



**Figure 1.** Outlet manifold bottom header.



**Figure 2.** Schematic diagram of the heat treatment process: a) normalization and (b) solution annealing.

## RESULTS AND DISCUSSION

The XRF results show that the material used in the outlet manifold bottom header (OMBH) is INCOLOY alloy 800 type. The chemical composition is shown in Table 1.

**Table 1.** Chemical composition of INCOLOY alloy 800.

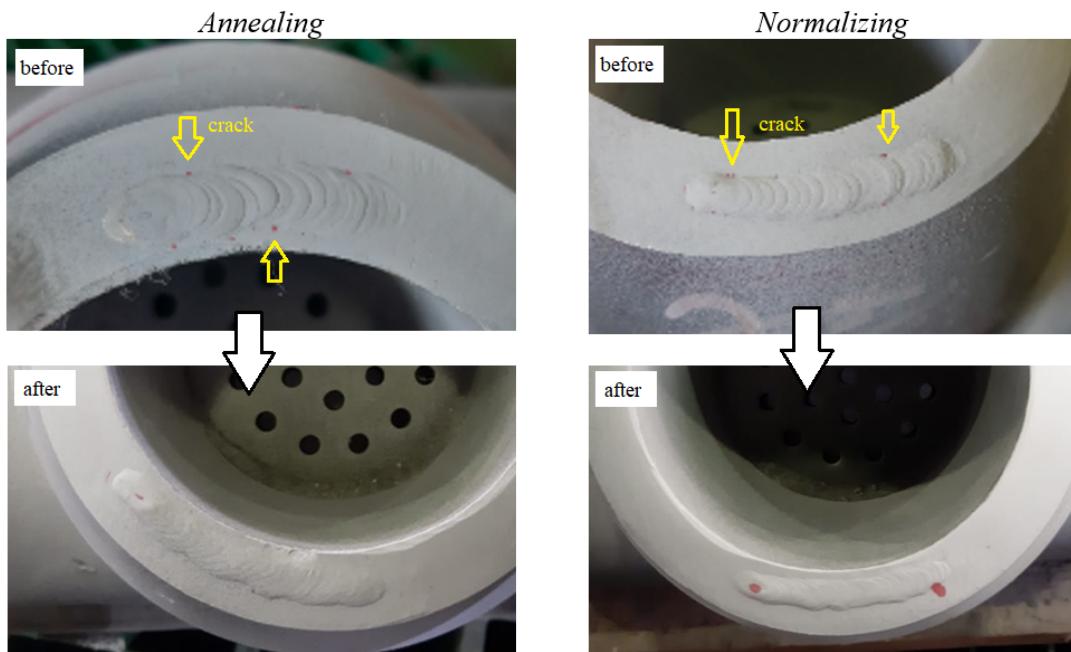
Element	Cr%	Ni%	Mn%	Fe%	Nb%
wt.%	20.97	34.30	1.14	42.09	1.21

Table 1 shows that the material included in the INCOLOY alloy 800 group with the main element is 34Ni-21Cr-42Fe with nickel that exceeds 25% and chromium that exceeds 14%. These properties can withstand oxidation reactions that cause corrosion at high temperatures. Before the weldability test, visual analysis using dye penetrant is carried out on each sample. Visual analysis reveals no defects or cracks on the surface of each sample. No defects or cracks on the surfaces are indicated by the absence of a red penetrant liquid to the surface after using a developer fluid when spraying is finished.

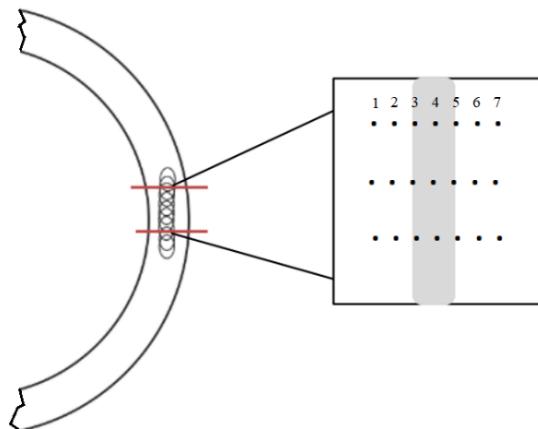
Heat input accompanied by a relatively fast cooling rate in the welding process causes a temperature difference and a cooling difference that results in residual stress (Kou 2003, Lippold and Kotecki 2005). INCOLOY alloy 800 is believed to have relatively high residual stresses due to exposure to heat during operation. The presence of residual stress causes cracks, as proven through dye penetrant testing. Visually indicated defects or cracks are shown in the boundary between the fusion zone and the base metal.

Sample heat treatment is carried out by solution annealing and normalizing methods that have the main objective of reducing residual stresses to improve the ductility of alloy 800. Parameters in heat treatment refer to ASME Section VIII Div 1 Part UCS-56 and UNF-56.

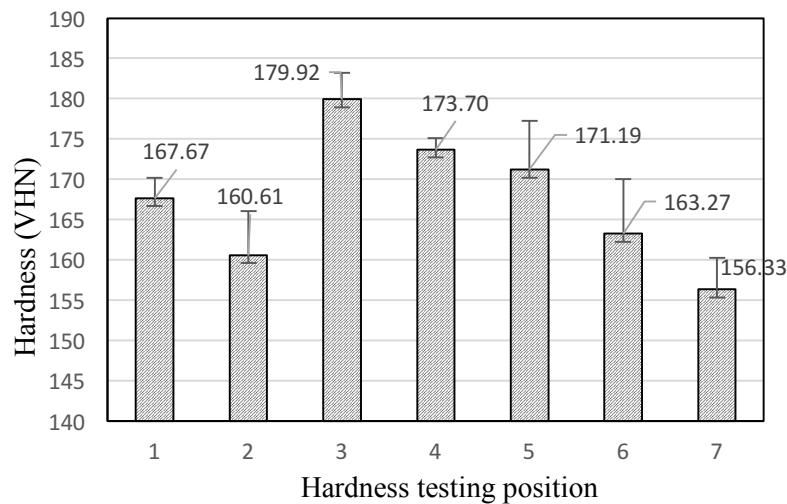
The results of the annealing solution sample and the normalizing sample after the heat treatment process indicate that there are still indications of defects or cracks on the surface of the normalizing sample that the welding process carries out. The indication of defects or cracks in normalizing samples has been significantly reduced, as shown in Figure 3. Defects or cracks can be seen from the results of the penetrant test, resulting in a red penetrant liquid after developer fluid spraying. The weldability test reveals that the sample annealing solution shows no defects or cracks after welding is carried out on the sample surface.

**Figure 3.** Weldability test before and after heat treatment.

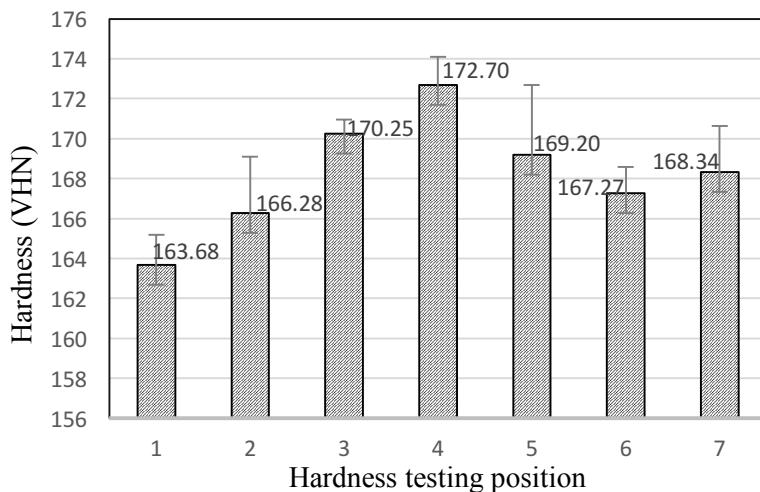
Hardness testing is used to measure the resistance of the material to deformation caused by penetration of the surface of a material. The position hardness test of the sample refers to the red line, which is shown in Figure 4. INCOLOY alloy 800 samples that have been subjected to a weldability test are prepared to perform a hardness test by cutting the sample into 2x2 cm sizes and by carrying out a framing process derived from a mixture of resin and catalyst.



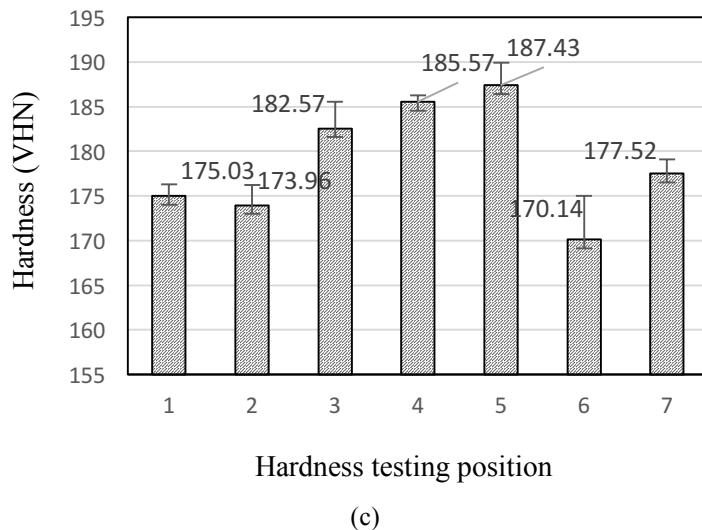
**Figure 4.** Hardness test position.



(a)



(b)



(c)

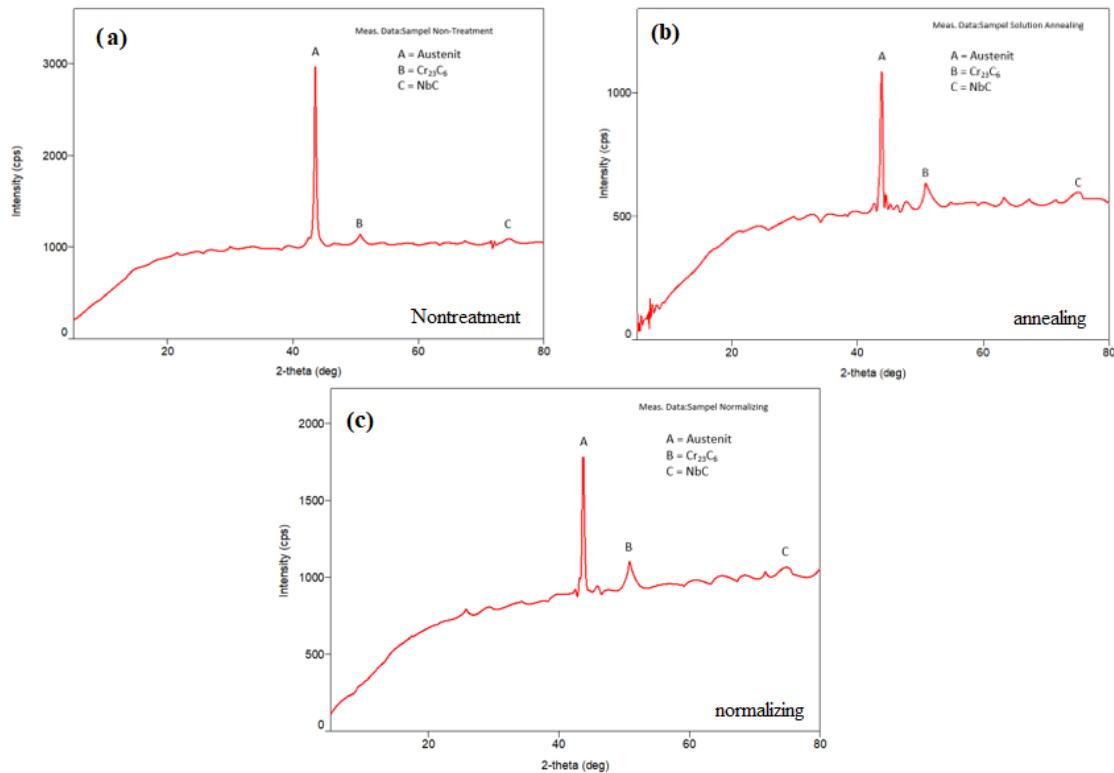
**Figure 5.** Hardness profile on the weld zone: (a) nontreatment, (b) solution annealing, and (c) normalizing.

The hardness test results shown in Figure 5 indicate that the base metal represented by zones 1, 2, 6 and 7 has decreased hardness from the technical data standard of alloy 800. The material standard technical data show that the hardness value of the superalloy material with the main element made from nickel and chromium is 170 VHN, while the highest hardness in the base metal is 167 VHN. The low hardness value of the nontreated samples is due to the prolonged exposure to heat during the operation process, as shown in Figure 5 (a). A decrease in the hardness value results in failure, such as creep. The hardness value increases in the fusion zone (zones 3, 4, and 5) due to new alloys from the filler rod, as shown in Figure 5 (b).

The normalizing sample is machined and polished for preparing the hardness sample test. Furthermore, hardness testing is carried out seven times at different zones with three repetitions, as shown in Figure 4. Figure 5 (c) shows the hardness testing results for the normalized samples. The test results show that the hardness value of the base metal is close to the INCOLOY alloy 800 standard hardness value. The base metal hardness value increases after normalizing treatment relative to solution annealing and nontreated samples. Because of the slow cooling rate, the greatest hardness value on the base metal is reached at 177 VHN during the normalization process. The preservation of carbide throughout the heat treatment procedure with a slow cooling rate is significant in the resultant growth. An increase in the amount of carbide plays a role in increasing the hardness value.

The maximum hardness value is 187 VHN in zone 5 on the welding zone. Several variables, such as carbide precipitation and additional alloying elements, cause the hardness value to be high in the welding area. Figure 6 (a) shows the XRD test results from nontreatment samples. The phase characteristics of alloy 800, which has been operating for a relatively long time, consist of phases of austenite,  $\text{Cr}_{23}\text{C}_6$ , and NbC. The main peaks show the austenite phase with a  $2\theta$  value of 43.582 and a peak intensity of 768. A  $2\theta$  value of 50.1 indicates the second-highest peak phase with a peak intensity of 368 diffusions (Arifin, Gunawan, et al., 2020). The lowest peak is the diffusion of niobium and carbon, forming NbC carbides with an angle of  $2\theta$  of 74.2 and a peak intensity of 90.

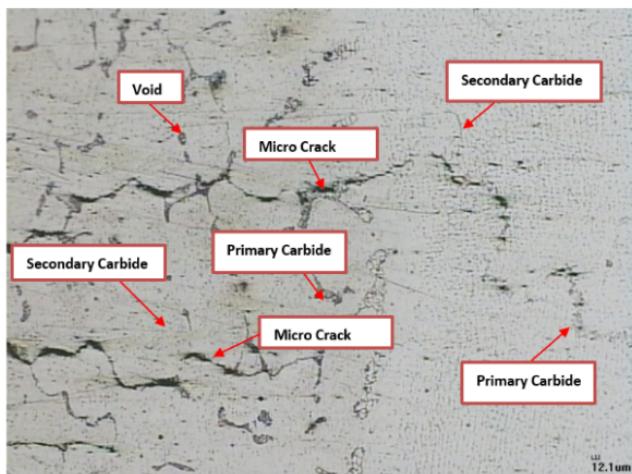
Figure 6 (b) shows the XRD test results of the INCOLOY alloy 800 heat treatment process with the solution annealing method. The results show that the phase characteristics consist of austenite,  $\text{Cr}_{23}\text{C}_6$ , and NbC phases. The peak list shown by the graph states that the austenite phase is the peak phase formed with a  $2\theta$  value of 43.82 and a peak intensity of 279. The second highest peak of the phase is the diffusion between carbon and chromium, which forms the  $\text{Cr}_{23}\text{C}_6$  carbide. The diffusion of niobium and carbon has the lowest peak to form NbC carbides.



**Figure 6.** XRD testing results: (a) nontreatment, (b) annealing, and (c) normalizing sample.

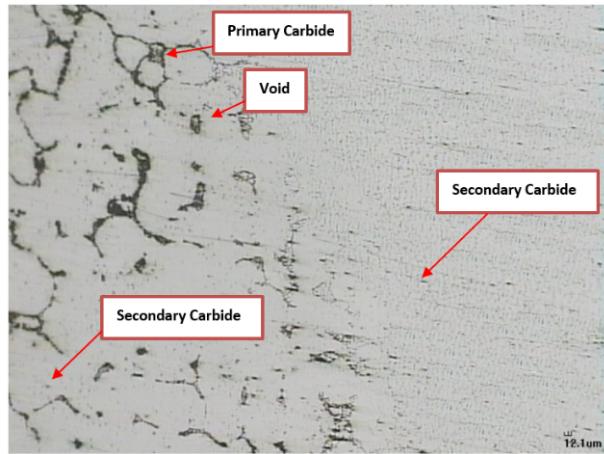
XRD test results for INCOLOY alloy 800 through the normalizing method can be seen in Figure 6. The results show that the phase characteristics consist of austenite, Cr<sub>23</sub>C<sub>6</sub>, and NbC phases. The peak list shown by the graph states that the austenite phase is the peak phase formed with a 2θ value of 43.82 and a peak intensity of 279. The second highest peak of the phase is the diffusion between carbon and chromium, which forms the Cr<sub>23</sub>C<sub>6</sub> carbide. The diffusion of niobium and carbon has the lowest peak to form NbC carbides.

The XRD test results show almost the same pattern in each trial sample, but the results of the diffraction peak pattern show a widening curve. In the heat-treated sample, the peak shift to 2θ is more significant. The solution-annealed samples have experienced a more significant peak shift than the normalized samples. If the material is affected by an inhomogeneous strain field, the peak position shifts, and the diffraction peaks are expelled. The peak shift in the XRD result indicates a change in residual stress in each sample due to the effect of strain correlating with the residual stress.



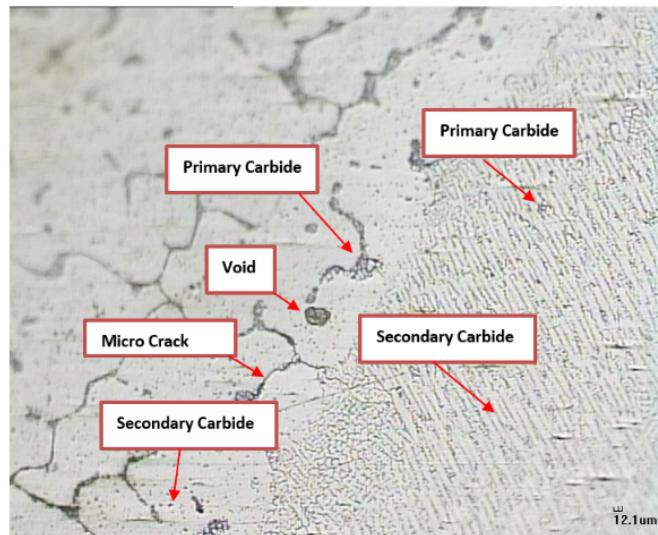
**Figure 7.** Metallographic testing results of the nontreatment sample.

The observation result of the nontreatment sample is shown in Figure 7. The microcrack attacks the grain boundaries because there are voids that have fused and are connected. The voids have been spread out from grain boundaries to around grains. Voids can coalesce and be connected due to the influence of residual stress from thermal activity. The microcrack causes creep cracking on the surface, leading to cracking in the ageing INCOLOY alloy 800. According to the standard of Manoir Industries, the appearance of microcracks following the weldability test method indicates that the performance of the material has deteriorated (Anderoglu 2004).



**Figure 8.** Metallographic testing results of the solution-annealed sample.

The annealing solution sample shown in Figure 8 reveals no microcracks similar to the nontreated sample. The sample annealing solution indicates the presence of primary carbide, secondary carbide, and voids, but microcracks do not occur in the annealing solution sample. The absence of microcracks is caused by a reduction in residual stress, which minimizes the formation of connected and unified voids.



**Figure 9.** Metallographic testing results of the normalized sample.

The normalizing sample is shown in Figure 9, which shows that there are microcracks that have spread to the boundaries of the weld area. Microcrack propagation occurs due to the influence of heat on the weldability test process. Reductions in residual stress occur in the normalized samples, which is shown in the microcrack formation results being smaller than the nontreatment samples. The heat treatment minimizes residual stress that can affect voids so that voids do not coalesce and instead coalesce to form a microcrack.

## CONCLUSIONS

Heat treatments for improving the weldability of INCOLOY alloy 800 have been performed successfully. Experimental results show that heat treatments with solution annealing and normalization affect the microstructural and mechanical properties. Solution annealing and normalization processes are effective in improving the weldability of INCOLOY alloy 800. The solution-annealed sample reveals no cracks after the weldability test. On the normalized samples, some cracks after normalization are observed. However, the reduction crack on normalizing samples is more significant than before heat treatment. On the nontreated samples, microstructure observation shows that microcracks spread from the base metal to the fusion zone. Microcrack propagation is found only in the base metal in the normalized sample. The microcrack occurs due to the residual stress effect on the voids formed due to the influence of carbide precipitation. The hardness test results show that each sample experiences an increase in its highest hardness value in the fusion zone caused by new alloy elements from the filler rod and residual stress.

## ACKNOWLEDGEMENTS

The authors would like to convey their great appreciation to Universitas Sriwijaya for supporting this research.

## REFERENCES

- Alvino, A., et al. 2010.** "Damage characterization in two reformer heater tubes after nearly 10 years of service at different operative and maintenance conditions." *Engineering Failure Analysis* **17**(7-8): 1526-1541.
- Anderoglu, O. 2004.** Residual Stress Measurement Using X-Ray Diffraction. *Mechanical Engineering* Texas A&M University. **Master of Science**.
- Arifin, A., et al. 2020.** "Failure analysis of AISI 304 stainless steel pipeline transmission a petrochemical plant." *IOP Conference Series: Materials Science and Engineering* **857**(1).
- Dehmolaei, R., et al. 2008.** "Microstructural characterization of dissimilar welds between alloy 800 and HP heat-resistant steel." *Materials Characterization* **59**(10): 1447-1454.
- Gunawan, G. and A. Arifin 2021.** "Intergranular Corrosion and Ductile-Brittle Transition Behaviour in Martensitic Stainless Steel." *Indonesian Journal of Engineering and Science* **2**(3): 031-041.
- Karimi, N., et al. 2008.** "Characterization of the oxides formed at 1000°C on the AISI 304 stainless steel by X-ray diffraction and infrared spectroscopy." *Applied Surface Science* **254**(8): 2292-2299.
- Kou, S. 2003.** *Welding Metallurgy*, John Wiley & Sons, Inc.
- Lippold, J. C. and D. J. Kotecki 2005.** *Welding Metallurgy and Weldability of Stainless Steels*, John Wiley & Son.
- Persaud, S. Y., et al. 2016.** "The influence of the high Fe and Cr contents of Alloy 800 on its inter- and intragranular oxidation tendency in 480 °C hydrogenated steam." *Corrosion Science* **106**: 117-126.
- Srinivasan, N. (2021).** "Sensitization of Austenitic Stainless Steels: Current Developments, Trends, and Future Directions." *Metallography, Microstructure, and Analysis*, **10**(2), 133-147.
- Sahlaoui, H., et al. 2004.** "Effects of ageing conditions on the precipitates evolution, chromium depletion and intergranular corrosion susceptibility of AISI 316L: experimental and modeling results." *Materials Science and Engineering: A* **372**(1-2): 98-108.

**Shah Hosseini, H., et al. 2011.** "Characterization of microstructures and mechanical properties of Inconel 617/310 stainless steel dissimilar welds." *Materials Characterization* **62**(4): 425-431.

**Tan, H., et al. 2011.** "Evaluation of aged Incoloy 800 alloy sensitization to intergranular corrosion by means of double loop electrochemical methods and image analysis." *Nuclear Engineering and Design* **241**(5): 1421-1429.

**Tavassoli, A. A. and G. Colombe 1978.** "Mechanical and microstructural properties of alloy 800." *Metallurgical Transactions A* **9**(9): 1203-1211.

**Yamawaki, M., et al. 1977.** "Oxidation of heat-resistant Fe-base incoloy 800 alloy." *Transactions of the Japan Institute of Metals*.