The Influence of Shielding Gases on Keyhole-Induced Porosity and Nitrogen Absorption in SS 304 Stainless Steel Fiber Laser Welds

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Research Article

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Abstract

As regards porosity formation and gas content, choosing the appropriate shielding gas for laser welding is essential for achieving high-quality joints. Keyhole-induced porosity formation tendency and, nitrogen content in fiber laser SS 304 stainless steel welds were investigated based on shielding gas nitrogen contents in fiber laser welding. Beads-on-plate autogenous welds were made at 5 kW continuous wave (CW) fiber laser in N\textsubscript{2} and Ar mixtures. Optical metallography, Micro-focused X-ray, X-ray radiography, and High-speed images of the molten pool were used to investigate the porosity formation. In addition, a gas analyzer was used to study the weld metal nitrogen content. The results show that nitrogen has a significant impact on the reduction of porosity in the melting zone, increases the dissolved nitrogen in the solidified weld metal, while almost no significant effect on the keyhole mode.

1. Introduction

Recently, high-performance fiber laser welding has been developed, its characteristic features are deep penetration and high-speed welding compared with conventional laser welding processes.\cite{1, 2} A thin capillary is created by high-power laser welding in the molten pool during welding, known as a keyhole. The keyhole enhances the absorption of laser energy and increases penetration. The keyhole also represents a very unstable pattern during welding. This severe instability is a key factor in the development of weld porosities. Pores in laser welding could be divided into two types, metallurgical factor-induced porosity, and keyhole-induced porosity. The porosity generated by low boiling point elements, or surface contaminations are called metallurgical factor-induced porosities, while pores that result from deep-penetration laser welding of large-thickness components in keyhole welding mode is known as keyhole-induced porosity\cite{3}.

Porosity is one of the most serious defects in high power laser welding because it degrades mechanical properties, particularly strength, fatigue, and elongation\cite{1, 4–6}.

Shielding gases are utilizing to protect the solidifying molten metal and weld keyhole from the surrounding atmosphere and hence dodge porosity and oxide inclusions, which give rise to poor, weld quality. It is common knowledge that the type of shielding gases and gas flow rate in laser welding are extremely important and influential, not only for the protection of the weld from the encirclement air but may have an impact on the characteristics and properties of the weld \cite{7–9}. Besides, reacting of shielding gases with the hot metal causes changing the weld pool surface tension, providing additional energy, and affecting plasma conditions, Which ultimately leads to modify the keyhole morphology and behavior during laser welding \cite{10}.

According to previous studies, the use of inert shielding gas during thick plate welding leads to the formation of many large porosities in deeply penetrated welds. In addition, active gases such as nitrogen shielding gas could be a source of porosities and brittle nitrides in carbon steel and ferritic stainless steel welds \cite{11}, whereas it is often a valuable element in the manufacture of austenite and duplex stainless
steels, stainless steel alloys. This is due to the willingness to use it as an alternative to nickel, herewith curtailment of the costs of the alloying element. An important finding indicated by some studies [3, 11, 12] is a porosity reduction in deep penetrating keyhole-type stainless steel welds by using nitrogen compared to inert gases such as helium or argon. On the other hand, due to its rapid thermal cycle during the high-power laser welding process, it is assumed that nitrogen loss may be eliminated through laser welding. [13].

The solubility of nitrogen in solidified weld metal of austenitic stainless steel depends on nitrogen in shielding gas, the welding temperature, as well as alloy composition [14, 15]. It is expected that during autogenic welding of austenitic stainless steel, the desorption and absorption of nitrogen will depend on the initial nitrogen content of the base metal and the strength of the surfactant of the weld. Arata, et al. [16] indicated that gathered nitrogen from the gas-metal reactions plus the residual nitrogen from the base metal represents the total nitrogen content in weld metal.

Conspicuously, the mechanism of nitrogen shielding gas in reducing the keyhole-induced porosity in deep penetration laser welds is unclear and needs further study. It is also necessary to clarify the nitrogen behavior in the weld pool. From this standpoint, this study aims to comprehend the effect of the shielding gases composition on both the porosity content and the absorbed nitrogen in the solidified weld metal of austenitic stainless steel.

2. Experimental Procedures

Austenitic stainless-steel SS 304 plates thickness of 5mm and 8mm were used in this study with a chemical composition that is shown in Table 1

<table>
<thead>
<tr>
<th>Element</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>S</th>
<th>P</th>
<th>Cr</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td>%</td>
<td>0.05</td>
<td>0.98</td>
<td>0.62</td>
<td>0.004</td>
<td>0.028</td>
<td>19</td>
<td>8.9</td>
</tr>
</tbody>
</table>

A continuous-wave (CW) fiber laser welding machine (IPG YLS-10000) with a maximum power of 10 kW and the beam parameter product (BPP) 4.5 mm·mrad was used to implement weld lines. Five types of shielding gas compositions were used for beads on plates laser welding at flow rate of 40 L/min as an axial shielding gas, in which the argon/nitrogen ratios (per unit volume of the gas mixture) were 0, 0.25, 0.5, 0.75, and 1.0 respectively. Argon-nitrogen mixtures were premixed prior to welding.

A 10000 f/s framing rate high-speed camera was used to observe the molten pool behavior during welding. The porosity formation behavior during laser welding was observed by the in situ micro-spot X-ray transmission imaging system developed by Katayama, et al. [17–19] The system uses a micro-focused X-ray source near a point source so that the minute area can be magnified and the image can be
photographed. A typical example of in situ X-ray surveillance with Pt particles under 5 kW laser power-welding conditions and welding speeds of 2 m/min are shown in Fig. 1.

Subsequently, the weld lines were visually inspected and radiographically examined from the upper surface for observing the weld porosity. Hence, samples were cut out from the weld for optical observation and gas analysis. For microstructural investigation, the specimens were cut, ground, polished, and electro-etched in an oxalic acid etchant (10 g oxalic acid and 100 mL distilled water). Weld metal nitrogen content was analyzed using the Horiba EMGA-520 analyzer, Fig. 2 shows the location of the solidified weld sample used for the nitrogen analysis.

3. Results And Discussion

3.1 Monitoring of laser keyhole and keyhole-induced porosity formation

The fluctuation of the molten pool at different shielding gas could be observed by high-speed camera as shown in Fig. 3. The high-speed camera images show that in the case of using pure argon as a shielding gas, the keyhole front is irregular in shape with the appearance of some spatter, which indicates the keyhole instability, while the situation changes when adding nitrogen, where the keyhole front became more stable, and if only nitrogen is used, the front of the keyhole is close to a regular oval.

The low stability of the molten pool with argon shielding gas gives chance for the trapped gas bubbles to create pores in the solidified weld metal. Whereas when nitrogen gas is used, the keyhole becomes so stable that it suppresses or prevents the formation of bubbles, thereby reducing porosity or lack of porosity.

X-ray transmission imaging snapshots through CW fiber laser welding of 304 stainless steel are shown in Fig. 4, welding was performed with 5 kW power and 2 m/min speed in shielding gases of 100%Ar, 50%Ar + 50%N2, and 100%N2, respectively. The results of monitoring X-ray transmission confirm that bubbles are generated from the tip of the keyhole to form porosity. In the case of using pure argon as a shielding gas, bubbles that lead into pores were started to form from the keyhole tip, then moved with the anticlockwise vortex to the rear part along with the molten pool bottom. Afterwards, bubbles are captured by the solidification front due to the high solidification rate of laser welding. By adding nitrogen in the shielding gases, the keyhole becomes more stable, both the number and the volume of bubbles were reduced with nitrogen increasing in the shielding gas. In the utilization case of pure nitrogen, the keyhole was stable and no significant bubbles were formed.

The previous investigation was assured by a radiographic test. The results of the X-Ray radiographic examination shown in Fig. 5 reveal the presence of a few pores at laser welding with argon shielding gas as well as argon with a low percentage of nitrogen, but at a higher percentage of nitrogen, pores were disappeared. It is confirmed that the cause for lesser or no porosity in welds shielded with nitrogen gas is
imputed to the difficulty of bubbles forming in liquid metal nature. [15]. In addition, the ability to absorption of nitrogen in the molten pool of austenitic stainless steel may be another reason for the porosity reduction when using nitrogen gas [11].

3.2. Weld metal nitrogen content

The nitrogen content of weld metal was measured in a variety of samples and the average results are represented graphically based on the nitrogen percentage in shielding gas in Fig. 6. The results show that the measured base metal nitrogen content was 360 ppm, while it was slightly reduced to be 340 ppm when applying pure argon as a shielding gas.

Also, the results showed that an increase of nitrogen percentage in argon - nitrogen mixture from 25% and more increases the N content of solidified weld metals. In the case of 75% N₂ or more as a shielding gas, only a slight increase in nitrogen content. It seems that it reaches the solubility limit of 490 ppm in the solidified weld metal.

As the high temperature of the laser in the welding area leads to the dissociation of nitrogen from polyatomic molecular nitrogen gas N₂ to the monatomic nitrogen gas N [20], as shown in Eq. 1. Thus, facilitating the absorption of nitrogen in molten iron.

\[ \text{N}_2 (\text{shielding gas}) \rightarrow 2\text{N (plasma)} \rightarrow 2\text{N (} \% \text{ Weld pool) (1)} \]

At a given temperature, Sievert’s law is the one that governed the equilibrium solubility of the nitrogen in the molten weld metal. According to Sievert, the concentration of nitrogen in the molten weld metal is proportional to the square root of the diatomic nitrogen partial pressure above the welding bath ‘as indicated in Equation. 2’ [21, 22].

\[ N_{eq} = K_{eq} \sqrt{P_{N_2}} \]

Where:

- \( N_{eq} \), is the molten weld metals nitrogen concentration at equilibrium with diatomic nitrogen (wt-%).
- \( K_{eq} \), is the equilibrium constant.
- \( P_{N_2} \), is the nitrogen Partial pressure in the shielding gases (atm).

This means that the solubility limit of nitrogen in molten stainless steel can be increased by raising the partial pressure of the diatomic gas above the melting point. Nevertheless, there are many doubts about Sievert's law's applicability when describing diatomic gas dissolution in molten weld metal in the presence of plasma [20, 21, 23].
To elaborate more on the solidified weld metal nitrogen content, it was imposed that the final nitrogen content is the result of complex processes input and output of nitrogen atoms in the molten weld pool during laser welding.

As for the nitrogen input and absorption in the molten metal from two sources:

- Nitrogen coming from the plasma atmosphere surrounding the weld pool[24].
- Nitrogen content of the base metal prior to melting during welding.

In addition, the process of removing dissolved nitrogen from the weld pool is done by recombining the nitrogen atoms to form nitrogen molecules (N$_2$) that may escape into the atmosphere, plus escaping of some atoms close to the surface during and immediately after solidification as represented by Eq. 2.

$$2N \text{ (% Weld pool)} = \rightarrow N_2 \text{ (gas)} \quad (2)$$

From this standpoint, increasing the turbulence of the weld pool increases the amount of escaping nitrogen due to the increase in the surface area of the weld pool. Also, in the case of using only argon as a shielding gas, means that no input of nitrogen atoms in the weld pool, this allows some of the nitrogen atoms already existing in the weld pool to combine to compose nitrogen molecules (N$_2$) that can getaway into surrounding atmosphere. This hypothesis is schematically illustrated in Fig. 7.

In laser welds with pure argon as the shielding gas, a significant amount of delta ferrite is observed in re-solidified fusion zones (Fig. 8a). As nitrogen is added to the shielding gas, ferrite percentages decrease, whereas austenite percentages increase in the fusion zone. As illustrated in Fig. 8b, the fusion zone microstructure can be observed when pure nitrogen is used as the shielding gas. Scanning electron microscopy (SEM) examination gives deep information on the microstructure of re-solidified fusion zones of the laser welds (Fig. 9). SEM imaging shows the diversity in the delta ferrite such as primary delta ferrite, skeletal delta ferrite and lathy delta ferrite in the matrix of austenite.

It is noteworthy that the researcher concluded in previous studies [2, 25] that weld metal's microstructure is significantly affected by nitrogen, as it limits the formation of delta ferrites and increases austenite.

### 4. Conclusions

SS 304 stainless steel welds have been implemented at diverse mixture ratios of nitrogen-argon shielding gas by fiber laser autogenous welding process.

- In fiber laser welding of austenitic stainless steel, X-ray transmission observation could be used to observe the formation and development of porosities. The results could provide an advance in understanding porosity formation.
- The mechanism of porosity formation induced by the keyhole can be summed up as follows: bubbles start from the tip of the keyhole, which increases in quantity and size in case of keyhole
instability, consequently are captured by the solidification front.

- Porosity may occur more easily in the welds with argon shielding gas compared with nitrogen shielding gas. Where nitrogen gas could react and dissolve in the molten weld pool, accordingly, it can restrain the propensity of porosity in the weld metal.

- The increase in nitrogen partial pressure in the shielding gas resulted in an increase in the nitrogen content of the solidified welded metal; on the other hand, the use of pure argon shielding gas decreased the nitrogen content of the solidified welded metal relative to the base metal.

- When laser welding is performed, the nitrogen content in the molten weld pool is the result of a complex series of processes involving the input and output of nitrogen atoms.

- Nitrogen has a significant effect on the microstructure of the re-solidified fusion zone, as it limits the formation of delta ferrites and increases austenite.

## Declarations

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### References


Figures

Figure 1

Illustration of micro-focus x-ray transmission in situ surveillance system.
Figure 2

location of the gas analysis sample.
Figure 3

High-speed photos of the molten pool at different shielding gas compassion show the keyhole formation process.

<table>
<thead>
<tr>
<th></th>
<th>100% Ar</th>
<th>50% Ar &amp; 50% N₂</th>
<th>100% N₂</th>
</tr>
</thead>
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<tr>
<td>X-ray Transmission images</td>
<td><img src="image1.png" alt="Image" /></td>
<td><img src="image2.png" alt="Image" /></td>
<td><img src="image3.png" alt="Image" /></td>
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</tbody>
</table>

Schematic illustration

![Image](image4.png)

Power = 5kW, Speed = 2m/min, fd = 0

Figure 4

Micro-focused X-ray transmission observation of keyhole behavior during 304 stainless steel fiber laser welding in different shielding gases.
Figure 5

X-Ray inspection image of welds made using different shielding gases.
Figure 6

SS 304 weld metal nitrogen contents for fiber laser welding with different Ar-N\textsubscript{2} gas ratios.
Figure 7

Graphic illustration of the autogenous laser weld pool, showing nitrogen absorption and desorption in the weld-pool throughout fiber laser welding.
Figure 8

Microstructure of fusion line showing d-ferrite distribution in the fusion zone of fiber laser welds made with (a) pure argon (b) pure nitrogen.

Figure 9

SEM image of laser weld showing d-ferrite distribution in the fusion zone of fiber laser welds made with (a) pure argon (b) pure nitrogen.