EXPERIMENTAL PROCEDURE

This chapter describes the experimental procedure followed during the course of the investigation and aims to examine the influence of the shielding gas composition, the base metal nitrogen content prior to welding and the surface active element concentration in the weld metal on the absorption and desorption of nitrogen during the autogenous arc welding of stainless steel.

3.1   STAINLESS STEEL ALLOYS STUDIED

During the course of this investigation, the influence of autogenous welding on the nitrogen content of eight stainless steel alloys was evaluated. The chemical compositions of these alloys are shown in Table 3.1. The first six alloys in the table are experimental alloys designed to have compositions similar to that of AISI type 310 stainless steel. Type 310 is a highly alloyed austenitic stainless steel that is normally produced without any deliberate nitrogen addition. It was selected as the base alloy for this investigation because it solidifies as austenite and remains fully austenitic down to room temperature. This prevents any bulk solid-state phase transformations, which can lead to changes in the solid-state nitrogen solubility in the alloy, from taking place after solidification.   The last two alloys listed in Table 3.1 are commercially available stainless steels included in this investigation as reference samples. Cromanite is a nitrogen-alloyed austenitic stainless steel produced by Columbus Stainless, and AISI 304 is a general-purpose austenitic grade with no deliberate nitrogen addition.

Table 3.1 Chemical compositions of the stainless steel alloys investigated (percentage by mass, balance iron).

Alloy	Comments	Cr	Ni	Mn	Si	C	S	N
VFA 657	Low N, low S	24,4	20,1	1,91	1,60	0,075	**0,023**	**0,005**
VFA 658	Medium N, low S	24,6	19,9	1,89	1,63	0,080	**0,023**	**0,105**
VFA 659	High N, low S	24,3	19,9	1,93	1,63	0,085	**0,022**	**0,240**
VFA 752	Low N, high S	24,6	19,5	1,99	1,51	0,087	**0,052**	**0,006**
VFA 753	Medium N, high S	24,5	19,3	1,89	1,61	0,082	**0,061**	**0,097**
VFA 755	High N, high S	24,5	19,3	1,90	1,55	0,079	**0,049**	**0,280**
Cromanite	-	18,1	0,59	9,74	0,29	0,036	0,004	0,511
AISI 304	-	18,2	8,10	1,75	0,48	0,029	0,009	0,078

In order to study the influence of the base metal nitrogen content on nitrogen absorption and desorption during welding, the experimental type 310 alloys listed in Table 3.1 were produced with three different nitrogen concentrations:

- a *low* nitrogen level (residual nitrogen content of approximately 0,005%),
- a *medium* nitrogen level (approximately 0,1%), and

- a *high* nitrogen level (approximately 0,25%). This nitrogen level corresponds to the equilibrium nitrogen solubility limit in this steel calculated at a temperature of 1600ºC and a nitrogen pressure of 1 atmosphere using Wada and Pehlke's activity coefficients and interaction parameters[1].

The influence of the surface active element concentration in the steel on nitrogen absorption and desorption during welding was evaluated by producing each of the low, medium and high nitrogen type 310 experimental alloys described above at two different sulphur concentrations:

- a *low* sulphur content (approximately 0,02%), and
- a *high* sulphur content (approximately 0,05%).

This provides for a matrix of six experimental alloys with varying nitrogen and sulphur contents, as shown in Table 3.1. As an alternative to varying the sulphur content of the base metal, small amounts of oxygen can be added to the shielding gas during welding, thereby varying the absorbed oxygen content of the weld metal. Varying the sulphur content of the base metal was preferred during this investigation because it provides more accurate control of the surface active element concentration of the weld metal.

3.2 WELDING PROCEDURE

All the stainless steel samples were rolled to a thickness of 6 mm, thoroughly ground to remove any scale or surface oxides and degreased using acetone. Welding was performed using an automatic autogenous (i.e. no filler metal was added to the weld pool during welding) gas tungsten arc welding (GTAW) process in an enclosed glove box. Direct current electrode negative polarity and a 2% thoriated tungsten electrode, ground to an included angle of 90°, were used. The experimental arrangement is shown in Figure 3.1. The glove box was flushed with pure argon for at least fifteen minutes prior to welding, and a low argon flow rate was maintained through the box during welding to ensure a slight positive pressure inside the glove box. This was deemed necessary to avoid contamination from the surrounding atmosphere due to air entrapment in the arc during welding. Shielding was supplied by shielding gas flowing through the welding torch at a pressure of 1 atmosphere and a flow rate of 20 ℓ/minute. Welding was performed using a welding current of 150 A and an arc length of 2 mm (corresponding to an arc voltage of approximately 17 to 20 V, depending on the shielding gas composition). The welding speed was maintained at a level of 2,7 mm/s using an automatic tractor that moved the plate to be welded at a constant speed relative to the stationary welding torch.

3.3 MEASURING THE WELD POOL TEMPERATURE DURING WELDING

In order to compare the actual nitrogen content of each weld with the calculated equilibrium nitrogen solubility limit, the weld pool temperature is required for substitution into Wada and Pehlke's equations[1] described in §1.2.3 of the literature survey. An indication of the temperature of the molten weld metal during welding was obtained by measuring the temperature of the centre of the weld pool, based on the assumption that rapid convection in the weld pool ensures a fairly homogeneous temperature distribution in the molten metal during welding. The temperature measurement was performed by inserting a

thermocouple into the weld pool behind the arc during welding. The thermocouple was shielded from exposure to the arc by a ceramic sheath that left only the fused end of the wires uncovered. Accurate placement of the thermocouple in the center of the pool was facilitated by an adjustable steel guide tube attached to the main body of the welding torch and the thermoelectric signal from the thermocouple was recorded using a calibrated XY-recorder. The experimental arrangement is shown in Figure 3.2.

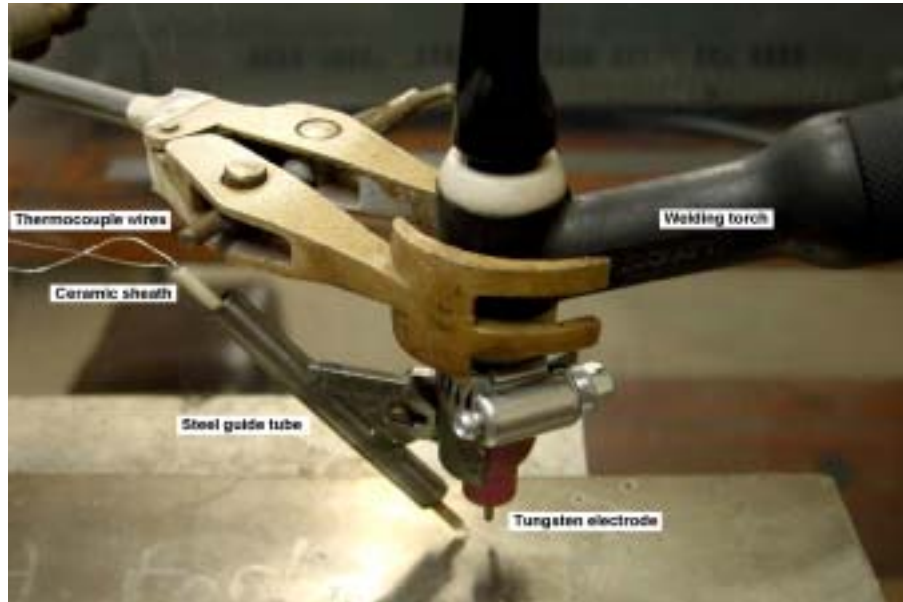**Figure 3.1** Experimental arrangement for autogenous welding.

Figure 3.2 Experimental arrangement for measuring the weld pool temperature.

Initial experiments using this arrangement and the welding parameters described in §3.2 above were unsuccessful since the weld pool proved to be too shallow to allow full insertion of the thermocouple without fusing the wires to the bottom of the pool. Kuwana *et al*[2] performed similar experiments and concluded that the weld pool temperature during autogenous arc welding is not influenced to any significant extent by the welding current, but is determined principally by the liquidus temperature of the

alloy (as illustrated by equation (1.23) in Chapter 1). Based on the conclusions of these authors, the welding current was raised in an attempt to increase the size and depth of the weld pool, while maintaining the arc length and travel speed at the values described above. In order to confirm Kuwana *et al*'s[2] conclusions, the weld pool temperature was measured and compared at two different welding currents, 300 A and 350 A. Two measurements were performed at each current setting. The results proved to be very repeatable and no significant differences were detected between the temperatures measured at the different welding currents. The conclusion can therefore be drawn that the weld pool temperature during autogenous gas tungsten arc welding is not a strong function of the welding current, and that the temperatures measured at high current values will be equally valid at a current of 150 A.

3.4 THE INFLUENCE OF THE SHIELDING GAS NITROGEN CONTENT ON THE NITROGEN SOLUBILITY OF STAINLESS STEEL WELDS

Pure argon and four premixed shielding gases, listed below, were used during the course of the project to evaluate the influence of nitrogen additions to argon shielding gas on the absorption and desorption of nitrogen by stainless steel alloys:

- pure argon,
- argon – 1,09% N_2 shielding gas mixture,
- argon – 5,3% N_2 shielding gas mixture,
- argon – 9,8% N_2 shielding gas mixture, and
- argon – 24,5% N_2 shielding gas mixture.

All the stainless steel alloys listed in Table 3.1 were welded using these shielding gases and the welding procedure described in §3.2. After welding the beads were examined visually and the weld metal nitrogen content of each sample was analysed using an inert gas fusion analysis technique, taking care to remove the metal drillings required for analysis only from the weld. At least two analyses were performed on each sample to ensure adequate repeatability.

3.5 THE MINIMUM SHIELDING GAS NITROGEN CONTENT THAT LEADS TO THE EVOLUTION OF NITROGEN BUBBLES FROM THE WELD POOL DURING WELDING

As illustrated in §1.3, nitrogen dissolution during arc welding does not obey Sievert's law. The weld metal nitrogen content increases rapidly with an increase in the shielding gas nitrogen content at low nitrogen partial pressures, followed by steady-state behaviour where the weld reaches saturation and nitrogen absorption from the arc is balanced by nitrogen evolution from the weld pool[3]. This steady-state region is generally associated with violent degassing and bubble formation. In order to avoid severe nitrogen losses from nitrogen-alloyed stainless steels and to prevent the formation of nitrogen-induced porosity in welds, the shielding gas nitrogen content has to be limited to avoid initiating steady-state behaviour. The minimum shielding gas nitrogen content associated with the onset of steady-state behaviour was determined during the autogenous welding of each of the alloys listed in Table 3.1 by using a range of argon-nitrogen shielding gases, mixed using a system of flow meters. The flow meters

were calibrated to supply shielding gas with the desired argon-nitrogen ratio at a total flow rate of 20 ℓ/min to the welding torch. The shielding gas nitrogen content was increased from 0,5% to 5% in 0,5% increments, and the arc, weld pool and completed weld examined visually during and after welding to determine the minimum shielding gas nitrogen level associated with the onset of significant desorption during welding. This point was generally characterised by severe degassing, spattering and violent metal expulsion from the molten weld metal. The nitrogen contents of the welds corresponding to the onset of steady-state behaviour in each alloy were determined using inert gas fusion analysis techniques.

The series of experiments described above therefore aims at determining the influence of the shielding gas composition (argon-nitrogen ratio) and the weld metal chemical composition (base metal nitrogen and surface active element concentration) on nitrogen absorption and desorption during the welding of stainless steel. The results obtained during the investigation are described in Chapter 4.

3.6 REFERENCES

1. H. Wada, and R.D. Pehlke, "Solubility of nitrogen in liquid Fe-Cr-Ni alloys containing manganese and molybdenum". Metallurgical Transactions B, vol. 8B. December 1977. pp. 675-682.

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