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Weldability of High Nitrogen Austenitic Stainless Steel Reports 2 ---Nitrogen Absorption of 316L Weld Metal and Its fine particles in Extreme Environments---

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Abstract

The suitability of the high nitrogen pressure Metal Inert Gas (MIG) ARC welding process (constant arc length condition) for high nitrogen containing stainless steel was investigated. The formation of fine stainless steel particles by the high nitrogen pressure MIG arc welding process was examined. The welding atmosphere consisted of pressurized N_2 gas. Nitrogen absorption of 316L stainless steel weld metal in the high nitrogen pressure MIG process was studied. The effect of nitrogen in remelted weld metal and changes in the microstructure of the weld metal were observed.

The nitrogen content of weld metal and the number of fine particles increased with increasing pressure of N_2.

Approximately 0.64 and 2.4 mass % nitrogen was absorbed in the weld metal and fine particles at pressure of N_2 6Mpa and 3Ma nitrogen respectively. Nitrogen content of the weld metal was lower than that representing equilibrium solubility of 316L stainless steel at close to the melting point. Nitride in the fine particles could not be found by X-ray analysis.

Key words: nitrogen absorption, stainless steel, weld metal, fine particle.

1. Introduction

Structural materials must offer the properties of nonmagnetism, high resistance to corrosion, wear, high strength and ductility according to the demands of the electronics, the precision and the cryogenic industries.

Recently, as advanced materials, high nitrogen stainless steels have been introduced. For example, a new material of cold-rolled austenitic stainless steel containing over 1% of nitrogen is a candidate.[1)]

The characteristics of remelting and solidification of the high nitrogen stainless steel as a new material intended for welding are not yet sufficiently studied. In this study , the ability of high nitrogen(N_2) pressure Metal Inert Gas (MIG) arc welding process (constant arc length condition)for high nitrogen containing stainless steel was investigated.

New functional materials using fine metal particles have been studied and developed.
Fine particles of various metal alloys , ceramics and polymers are also required. The production process of fine metal particle by means of Arc melting is one route. But it is said that arc process do not show good production efficiency.

In previous reports[2,3)] the authors demonstrated that production efficiency of fine metal particles could be greatly improved by using the high pressure MIG process.

In this experiment, high nitrogen containing austenitic stainless steel fine particles were produced by high nitrogen pressure MIG arc welding. The nitrogen content of weld metals and stainless steel fine particles were discussed. The results of nitrogen content were discussed and related to thermodynamically equilibrium data. Microstructural observations and some metallographical tests on high nitrogen stainless steel weld metals were described.

2. Experimental Procedure

Welding apparatus designed to maintain a controlled high pressure arc atmosphere was used. A stainless steel chamber of about 1.8m^3 in volume maintained the desired gas pressure and composition.

The atmospheric pressure in the chamber can be changed from about 15pa to 6.5Mpa.

The head of an automatic TIG. MIG and sub-

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chamber. **Figure 1** shows the schematic illustration of the equipment for high pressure arc welding.

The size of base metal is 12×100×200(mm). A consumable electrode wire of 1.6 mm diameter was used. The chemical composition of the SUS 316L stainless steel plate and electrode wire used is shown in **Table 1**.

High N_2 pressure MIG arc welding was performed using direct current power source which had an open circuit voltage of 80V(1,000A). Drooping characteristics and electrode positive conditions were chosen.

The nitrogen contents of stainless steel weld metals and its fine particles were analyzed by Leco equipment.

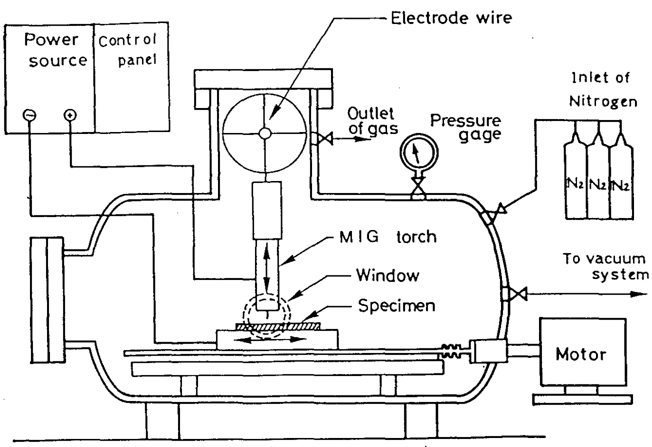

Fig. 1 Schematic illustration of apparatus

Table 1 Chemical composition of base metal and electrode wire (mass%)

Material	C	Si	Mn	P	S	Cr	Ni	Mo	N	O
SUS316L	0.017	0.65	1.21	0.03	0.02	17.01	12.29	2.07	0.018	0.004
SUS316L,1.6mmφ	0.016	0.50	1.82	0.019	0.021	19.24	12.71	2.42	0.05	0.012

2.1 Weld metal

Weld metals were obtained by bead on plate deposition method. Surface ground and cleaned base metals were set in the chamber. It was evacuated to a pressure of 100Pa, then filled with N_2 gas at the desired pressure. Welding (bead on plate deposition on) parameters used in this experiment were as follows: current;I=200 A, base metal travel speed;3.3mm/s, distance from base metal surface to torch tip;35mm, visual arc length: approximately 10mm.

After welding the surface appearance was checked and the microstructure of the weld metal was observed by optical microscope or scanning electron microscope(SEM). Lattice parameter measurements and hardness tests were applied to the weld metals.

2.2 Fine particle

In order to collect the fine stainless steel particle produced by high N_2 pressure MIG arc welding stainless steel wishes(diameter 150mm) were set on a travelling stand around the welding torch. The weight of the dish was measured before and after MIG arc welding and the generation rate of fine particle was calculated. Welding parameters employed were as follows:current; I=300 A, base metal travel speed; 0.83mm/s, distance from base metal surface to electrode tip: approximately 15mm.

3.Experimental Results and Discussion

3.1 Nitrogen content of 316L austenitic stainless steel weld metal by means of high N_2 pressure MIG arc welding

In previous paper[4], the authors reported that welding conditions, such as constant arc length, electrode positive and direct current drooping characteristics power source were useful for high Ar pressure MIG arc welding of mild steel plate.

Instead of Ar gas, high N_2 pressure MIG arc welding with the conditions described above was applied to high nitrogen containing stainless steel in a fusion joining process.

The surface appearance of weld metals obtained at various nitrogen pressures from 0.1Mpa to 6Mpa using high pressure MIG arc welding are shown in **Fig.2**. These seem to be free of defects on the weld metal surface, such as blow holes or cracks. The geometry seems smooth and without peculiar shape.

As shown in the same figure, arc voltage varies with N_2 pressure from about 68 V at 0.1 Mpa to about 90 V at 6.0 Mpa. The same tendency of arc voltage change was reported in a previous paper[4].

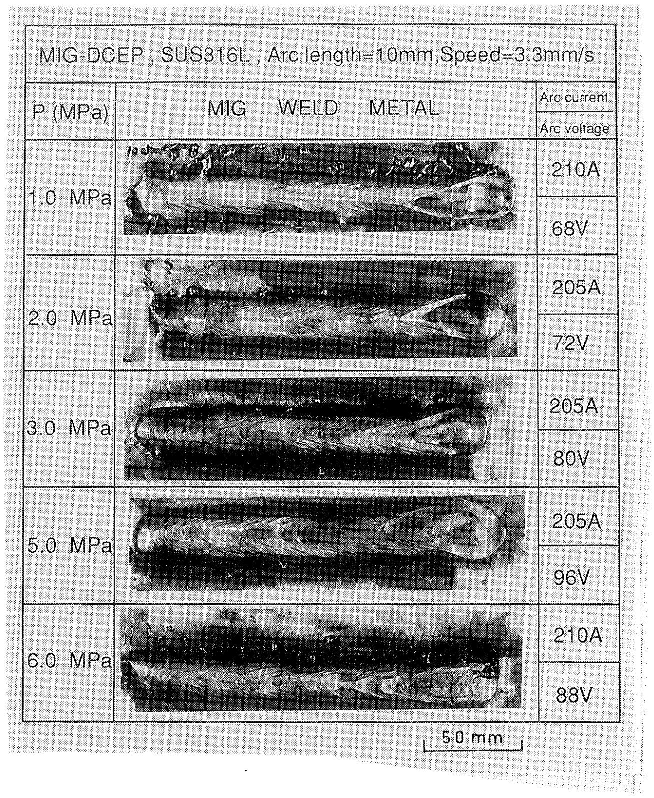

Fig. 2 Morphologies of bead appearance

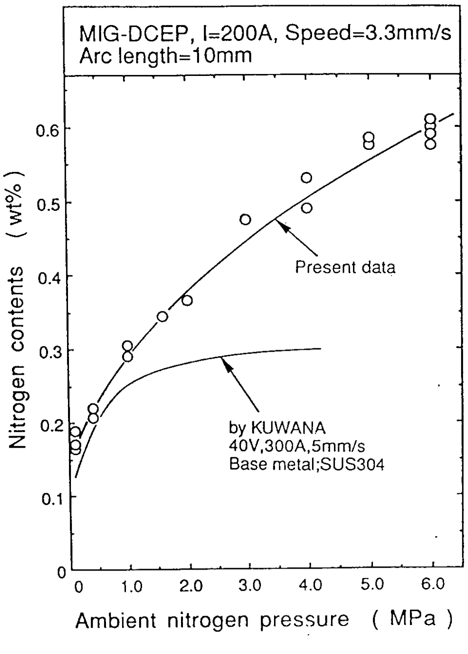

Fig.3 Effect of ambient nitrogen pressure on the nitrogen content of weld metal

Nitrogen content(N) of the weld metal as a fusion of a ambient N_2 pressure is shown in **Fig.3** N increases with increasing nitrogen pressure and it shows approximately 0.6 mass % at 6 Mpa. N content reported by T. Kuwana et al.[5] is plotted in the same figure as a solid line (experimental conditions: constant arc voltage;V=40V, current;I=300A, base metal SUS 304 stainless steel). Compared with Kuwana's results, the present results show higher nitrogen contents. This reason can be explained as follows. Constant arc voltage condition had been applied by Kuwana's works. In his case, it had been observed that arc length(distance between electrode tip and molten metal surface)decreased with increase of pressure. This means that the reaction time of the molten metal and nitrogen became shorter. But, in the case of constant arc length conditions as in this study, nitrogen absorption time does not change.

Using equilibrium results by Humbert,[6] Phelke et al[7]. and Nelson's equation [8], thermodynamical principles were applied to the nitrogen absorption of the weld metals. The nitrogen solubilities in the alloys that it had the same chemical composition as the specimens used were calculated. These solubilities , as a function of the square root of N_2 pressure, are shown in **Fig.4** Results of the present work are marked by open circles in the same figure. Nitrogen contents(N) obtained by these experiments do not represent saturation solubilities. It appears that the reaction of nitrogen gas and molten metal usually lasts for a few seconds. It is too short a time to reach saturation.

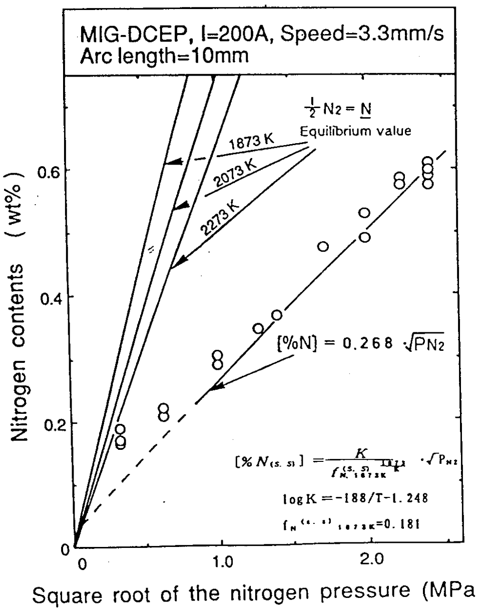

Fig.4 Relationship between sauare root of the nitrogen pressure and nitrogen content of weld metal and caluculated solubility

Beyond the 1 $Mpa^{1/2}$ level on, the linear relationship between N and N_2 pressure is observed in Fig. 4. The relation between weld metal nitrogen content and base metal nitrogen content is shown in **Fig.5** N content of the base metal was generated by high N_2 pressure MIG welding. N_2 pressures changed from 0.1Mpa to 6.0Mpa. From Fig.5 can be estimated the minimum N_2 pressure for MIG arc welding required for a given base metal nitrogen content. It can be noted that weld metal with was free of defects as a results of consists multi pass high pressure arc welding.

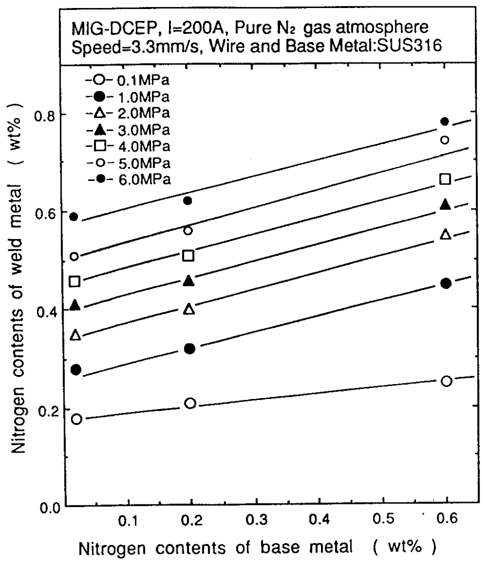

Fig. 5 Relationship between nitrogen content of weld metal and base metal nitrogen content

The structure of the weld metal consists of an austenitic single phase. Precipitates are observed above 0.68 mass % N. **Figure 6** shows micrographs of SEM (A) and TEM(B), respectively . The structure of the weld metal with about 0.78 mass % N by SEM observation is shown in (A). Fine films like precipitates, forming lamellar patterns, were detected clearly at grain boundaries. The microstructure of the specimen containing approximately 0.84 mas % N using a thin foil technique is shown in (B). Film -like precipitates were identified by selected area diffraction and shown to be nitrides, Cr_2N. Precipitated Cr_2N are thin and usually parallel oriented. Also , the formation of Cr_2N on twin boundaries is also observed.

The hardness of the weld metals was measured by Vicker's hardness testing. The hardness increased with increasing nitrogen content.

For example, weld metal containing about 0.6 mass % N indicated 250 HV or higher (the load 200gf). The hardening behavior seems to relate to solid-solution hardening by nitrogen. So, the variations of lattice parameter were measured by X-ray diffraction method. The results are shown in **Fig. 7**. The lattice parameter increases with increasing nitrogen content. Dissolved nitrogen cases strain in an austenitic structure. It is considered that most of nitrogen exists in solid solution in the weld metal. An interstitial form of nitrogen gives a hardening effect to the weld metal. But , in case of from about 0.7 mass % N , it is estimated that there is the possibility of precipitation hardening by Cr_2N.

3.2 Nitrogen content of 316L austenitic stainless steel fine particles produced by high N_2 pressure MIG arc welding

It was known that after arc welding, the base metal surface was covered by many fine particles[4]. The effect of ambient N_2 pressure on the generation rate(G.R.) of fine particles was determined. The relation between G.R. and ambient N_2 pressure is shown in **Fig.8**. the G.R. increases with increasing N_2 pressure. The formation fine stainless steel particles by the high N_2 pressure MIG arc process is superior to that by the TIG arc process.[2,3] Typical TEM photographs showing the fine particles are displayed in **Fig.9** The fine stainless steel particles have two types of shape, sparkle and polygonal. The sizes of fine particles have two types of shape, sperical and polygon at. The sizes of fine particles are not uniform, their diameters are distributed from about 40nm to several hundred nm. The fine particles are no magnetic property.

Based on the TEM observation results, the relation between size distribution and ambient nitrogen pressure was examined.

Figure 10 shows the percentage of particles as a function of particle size. The distribution of particle sizes has a wide range from several nm to hundreds of nm in diameter. With increasing welding pressure , the diameter of particles becomes slightly bigger.

The nitrogen content of the fine particles(Np) as a function of ambient N_2 pressure is shown in **Fig.11**. N content of weld metals is plotted by black circles in the same figure.

Np increases with increasing nitrogen pressure and it reaches approximately 2.4 mas % as 3Mpa. From about 3Mpa, Np is almost constant. It is clear that fine 316L stainless steel particles absorb nitrogen from the arc atmosphere. As compared with N weld (bead), Np is significantly greate. A rapid absorption reaction by time stainless steel particles is clearly involved.

Np was compared with the results of equilibrium studies previously shown in Fig. 4. Np, N(bead) and calculated nitrogen solubilities as a function of the square root of N_2 pressure are shown in **Fig.12**. Np and N are marked by open circle or black circle,

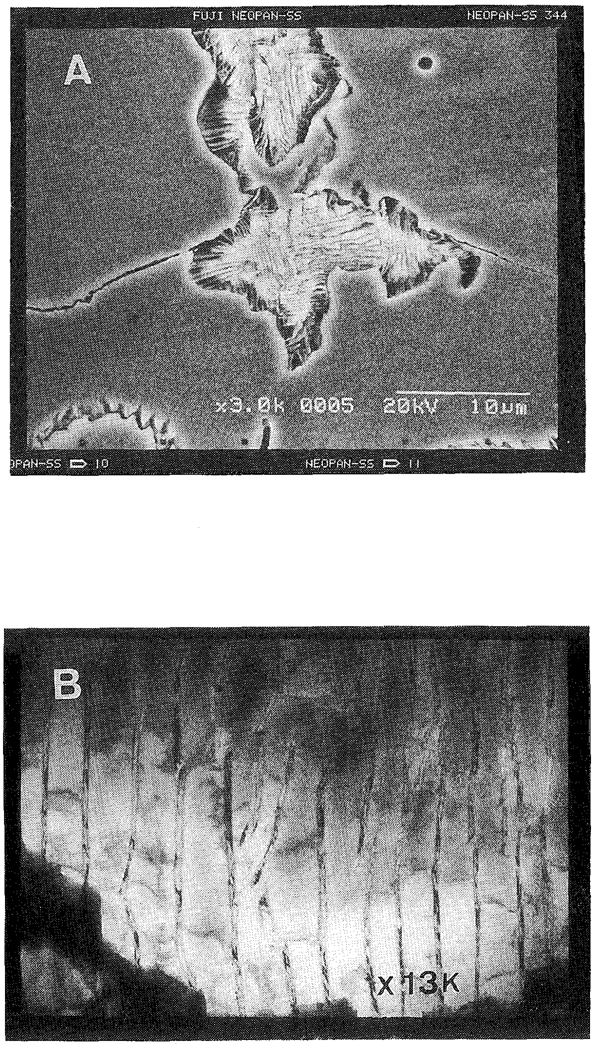

Fig. 6 SEM(A) and TEM (B) micrographs of nitrides

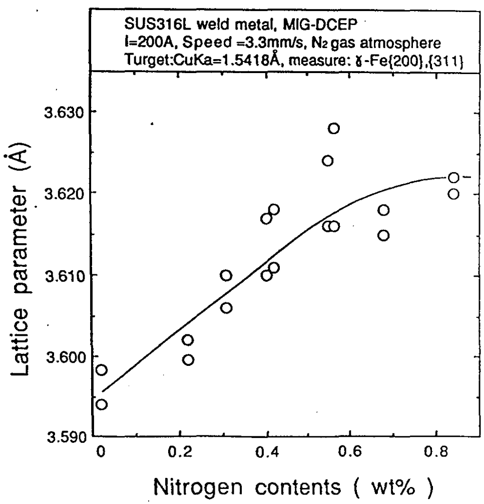

Fig. 7 Effect of the nitrogen content on the lattice parameter of weld metal

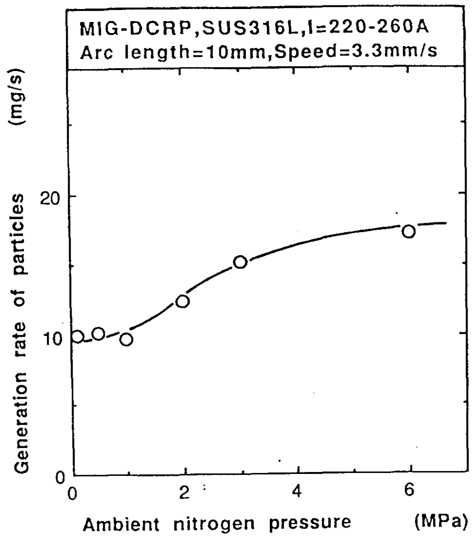

Fig. 8 Effect of ambient nitrogen pressure on the generation rate of fine particle

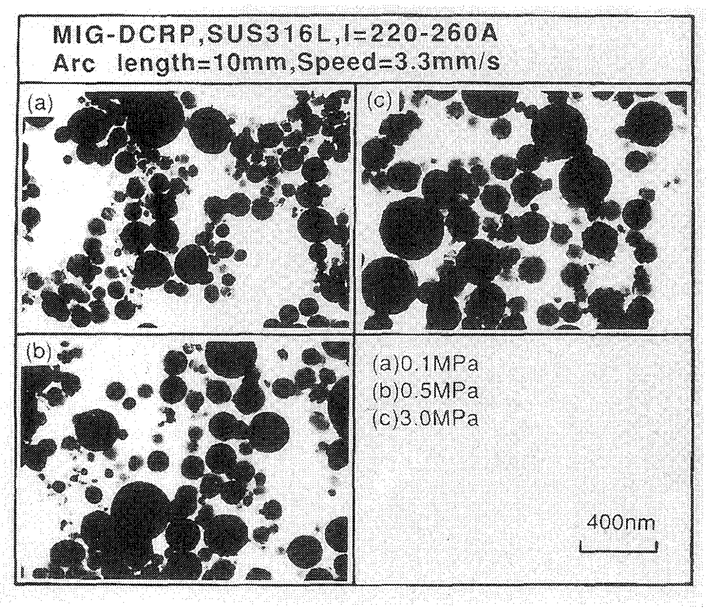

Fig. 9 Morphologies of stainless steel fine particles by TEM micrographs

respectively. The linear relationship between Np and the square root of nitrogen pressure is not demonstrated Nitrogen contents(Np) of fine stainless steel particles correspond to a nitrogen solubility at 1273K-1473K and 4Mpa N_2.

The mechanism of nitrogen absorption by 316L stainless steel particles can be understood from to scheme shown in Fig.13[9]. Nitrogen content of the

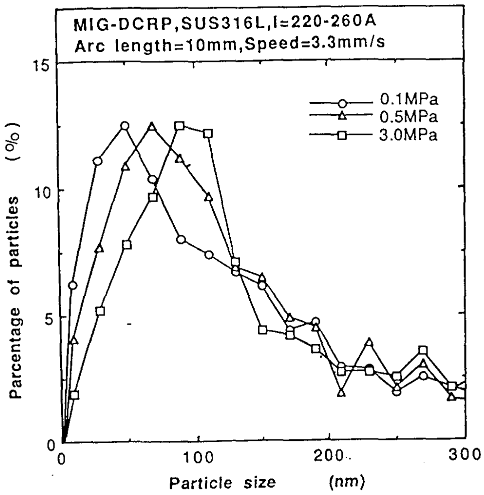

Fig.10 Comparison of fine particle sizes vs. ambient nitrogen pressure

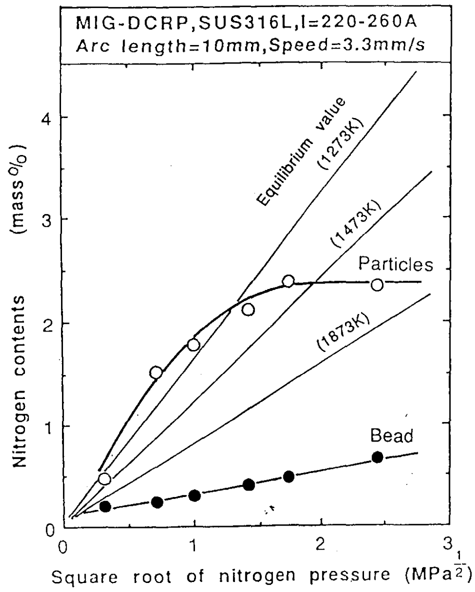

Fig.12 Relationship between square root of nitrogen pressure and nitrogen content of fine particles , weld metal(bead) and calculated solubility

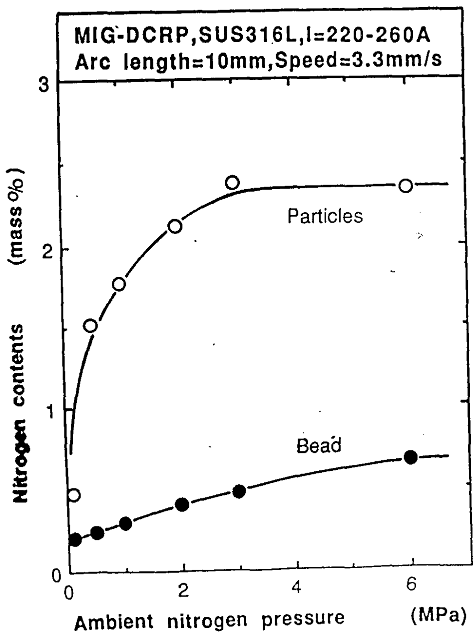

Fig.11 Effect of ambient nitrogen pressure on the nitrogen content of fine particle and weld metal (bead)

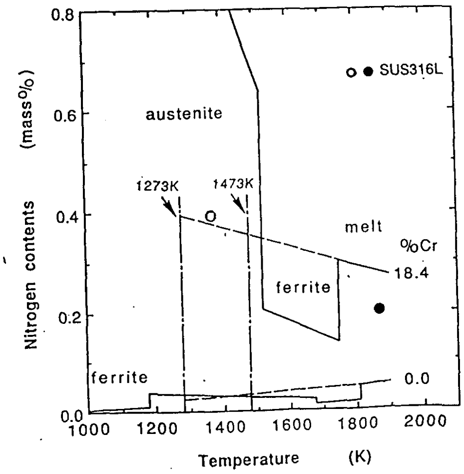

Fig.13 Schematic illustration of nitrogen content of Fe-Cr alloy as a function of temperature and I atm N_2 pressure

melt , ferrite and austenite phase as a function of temperature is shown here (N_2 pressure ;0.1Mpa). The fine particles contain about 18-19mass %Cr. Marked as lack circle, is the solubility of nitrogen 18.4 % Cr-Fe alloy and corresponds with the solid line as a function of temperature is lowered , nitrogen content increases.

316L austenitic stainless steel fine particles produced by high N_2 pressure arc welding, pass into the environment of nitrogen . The temperature of the fine particles is lowered. But if the fine particles pass quickly through the temperature range between 1530-

1750K, they will reach the austenitic phase. This areas has a high nitrogen solubility as shown in the same figure. Thus, 316L stainless steel fine particles can absorb faster nitrogen by this process.

It has been shown in this study high nitrogen containing particles are produced by high N_2 pressure MIG arc process. Compaction of high nitrogen fine particles is interesting and the investigation of mechanical and chemical properties of the compacts is important also.

Conclusions

(1) MIG arc welding under high N_2 pressure with a constant arc length condition is a useful fusion welding process for high nitrogen containing stainless steel.
(2)The nitrogen content of the weld metal is lower than the content for equilibrium solubility.
(3)The nitrides observed in the weld metal beyond about 0.64 mass % nitrogen.
(4)Lattice parameter and Vicker's hardness number of the weld metal are changed linearly by nitrogen content..
(5)Large amounts of nitrogen are absorbed in the 316L stainless steel fine particles, the content corresponds to the amount of equilibrium solubility during 1273K-1473K up to 3Mpa N_2 pressure.
(6)Nitride can not be found in the high nitrogen containing fine particles by x-ray analysis
(7)The generation rate of fine particles increased with increasing N_2 pressure.

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