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Behaviour of Precipitates in Bond Region of Overlay Welding (Report Ⅱ) †

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Abstract

In the previous report[1], it was confirmed that the precipitation of carbide $M_{23}C_6$ can be occurred at the boundaries between austenite and ferrite grains. With the use of various means such as optical and electron microscopes, electron diffraction, and the state analysis of the extracted residues by X-ray diffraction, the examination was carried out at which niobium added can be detected.

It was confirmed that the massive carbides of niobium (NbC) having f.c.c. structure precipitated at the boundary between austenite and ferrite grains. From the ion micro-analysis (IMA) of the extracted residues, molecular ions of iron, chromium, molybdenum, nitrogen, and oxygen were detected.

1. Introduction

Overlaid weld metal of austenitic stainless steel with band arc method must be usually composed of two phase structure. The first layer is used for interference of elemental dilution from the base metal, and the second one is used for obtaining required chemical composition. To prevent weld crack occurrence, the weld metal must be adjusted to have ferrite structure in the range of 5 to 10% after post-heating.

Furthermore, various elements are added to the weld metal of stainless steel to improve hot workability, machinability, corrosion resistance and oxidation resistance. The respective effects were reported in the previous papers.[2)-5)] Especially, the element, which is apt to form carbide or nitride, such as niobium or tantalum is added.

It was determined that small amount of niobium or titanium showed to form easily the precipitates of carbide than chromium when the heat-treatment in the range of 850 to 930°C was applied. For this reason, it is considered that niobium has superior stabilizing characteristic for carbon in steel because the precipitating temperature of chromium carbide is 600°C.

Moreover, niobium despite of small addition in stainless steels has the following benefits:

1) It prevents inter-crystalline corrosion.
2) It makes austenite grain size to be finer.
3) It improves the mechanical properties such as yield strength, tensile strength, high strength at elevated temperature, creep strength and so on because of the precipitation or solid solution hardening.

During welding, the HAZ receives heat-treatment above 1000°C. Knife line attack phenomenon is the important problem for welding.[6)] Therefore the attention must aim to the process of post-heating after welding. In this report the studies were performed to determine the shape, the composition, the location of the precipitates deposition. Therefore, observation by optical and electron microscopes and analysis by electron and X-ray diffr-actions were carried out.

2. Esperimental Specimens

Chemical compositions of base metal and band electrode are given in Table 1. In this study, ASTM 387D as base metal was one layer overlaid. 347S steel was used as hoop, and flux was composed of the system CaO-Al_2O_3-MgO-CaF_2. The welding conditions and the overlaying size are given in the previous report.[1)]

Table 1 Chemical Composition of Base Metal and Filler Wire

		Elements (%)								
		C	Si	Mn	P	S	Ni	Cr	Nb	Mo
base metal	40 x 500 x 600	0.12	0.22	0.49	0.014	0.011	–	2.29	–	0.97
wire	0.4 x 75	0.014	0.35	1.72	0.014	0.013	11.24	21.22	0.91	–
flux	12 x 100	$CaO-Al_2O_3-MgO-CaF_2$								

† Received on Jan. 8, 1976

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Specimens for microscopical observation and extraction of the precipitates were heat-treated at 1100°C for 2 hour and water-quenched, continuously stress-released at 650°C for 20, 50, 65, 75, 120, hour respectively. Specimens of the required size were cut, cleansed with acetone and alcohol after buff-polished and continuously dried.

Further, chiplike specimen was prepared for acid extraction of the precipitates. Specimen for electron microscopical observation was cut and reduced the thickness with mechanical polishing. Afterward, it was polished with electrochemical method.

3. Experimental Procedures

Chemically, niobium carbide is thought as stable compound. For that reason, many reliable extracting methods have been developed for state analysis of niobium in steel.[7)−9)]

In this experiment, the acid extraciton method with HCl or HNO_3 and potentiostatic electrolysis, were carried out to extract niobium carbide from the weld. Extracted residues were washed with water and alcohol, then they were separated with a centrifuge and dried in vacuum dryer. The residues were identified with Debye-Scherrer method. (Cr Kα,32 kV x 10 mA, V filter)

Electron microscopical observation and electron diffraction were performed under the accelerated voltage of 200 kV (JEM 200 type), and the selected area electron diffraction was used to identify crystal structure of the specimens. Residues were burried in indium to analyze with IMA. Further, we attemped to determine gas components such as oxygen and nitrogen in the residues.

4. Experimental Results and Discussion

In Photo. 1, X-ray diffraction result of the precipitate from as-weld and SR-treated are shown. The residues extracted from the specimens were confirmed to NbC as shown in Table 2 of as-weld and SR-treated. Imai et.al.,[10)] said that the niobium carbide has f.c.c. structure

Table 2 d-values of niobium carbide, NbC (compared with ASTM CARD 10-181)

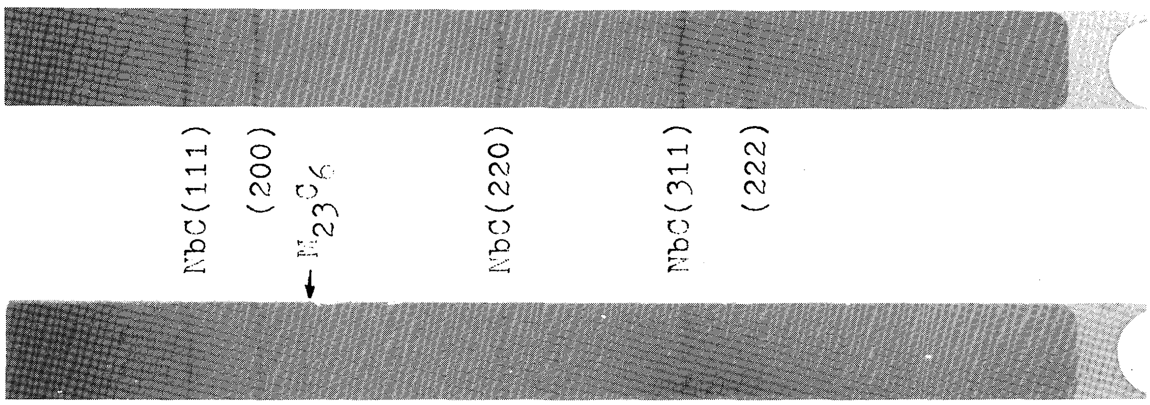
Residues	NbC a_o = 4.4702	hkl
2.57	2.58	111
2.22	2.23	200
1.57	1.58	220
1.337	1.348	311
1.28	1.290	222
1.10	1.117	400

and lattice parameter a_0=4.457 Å when contained a small quantities of nitrogen. Mori et.al.,[11)] said that carbonitride, $Nb_{0.8}N_{0.07}$ and $NbN_{0.65}C_{0.24}$ were isolated as the precipitate when 6N HCl extraction was used. Kanazawa et.al.,[12)] found that the precipitate (Nb, Mo) (C, N) has NaCl type and lattice parameter of a_0 = 4.441, 4.443 Å.

By Kawamura et.al.,[9)] said that the precipitates of niobium-carbides and -carbonittrides were identified to be f.c.c. δ-Nb (C, N) and lattice parameter a_0 = 4.437 − 4.453 Å. From analysis with IMA of extracted residues the molecular ion of iron, chromium, molybdenum, nitrogen and oxygen were determined distinctly. It was considered that the nitride or oxide of niobium coexisted in the extracted residues.

It is necessary to do more precise X-ray diffraction analysis to discuss in detail. In Photo. 2, metallographic textures of base metal and weld metal are compared. At the position apart 3 mm from bond, a massive precipitate having f.c.c. structure was observed. From the selected area electron diffraction analysis and X-ray diffraction, it was determined to NbC. With the use of IMA, it is

A)



B)

Photo. 1 X-ray diffraction patterns of residues extracted by HCl method

(A) As weld

(B) SR treated at 650°C, 75 hr

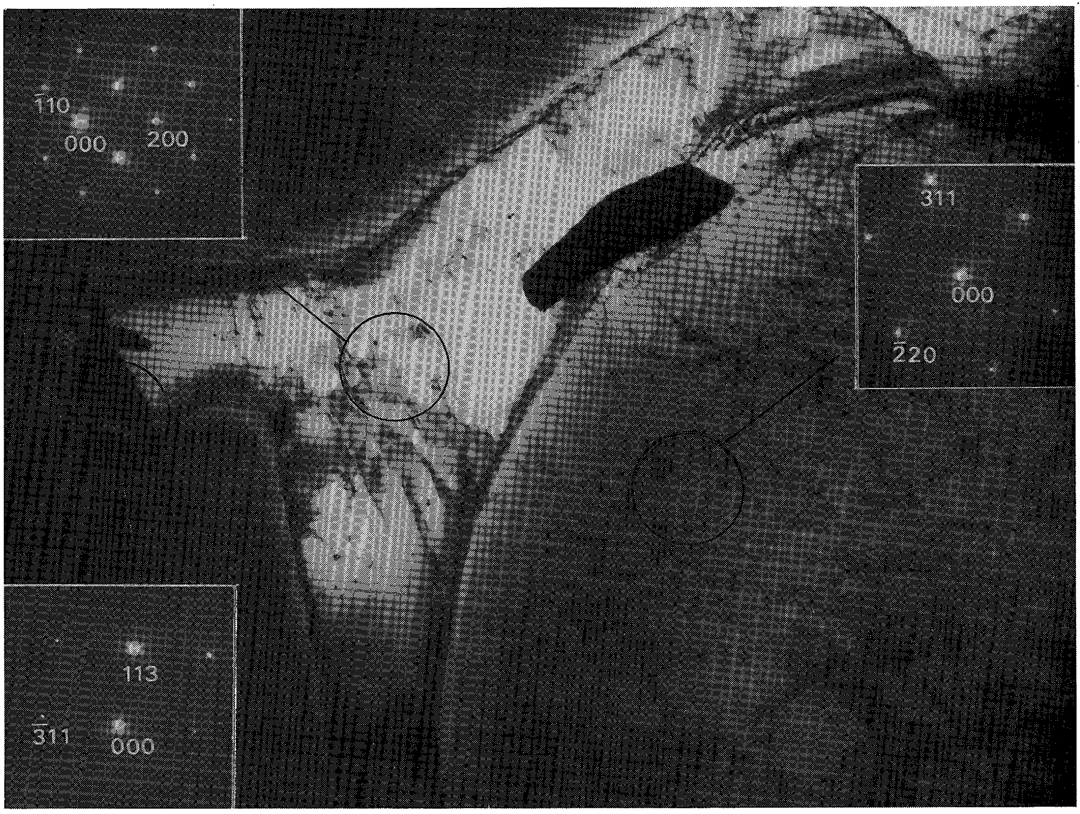
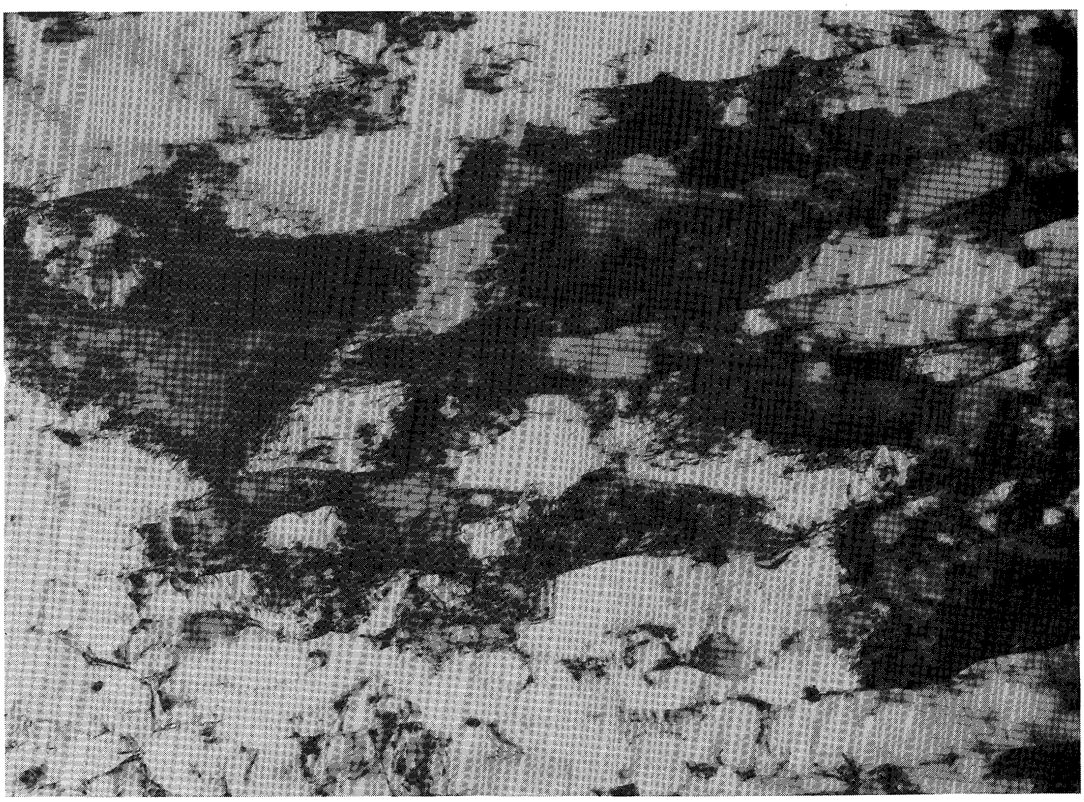


Photo. 2 (A) Transmission electron micrograph of overlaying stainless steel, showing a ferrite grain, NbC and $M_{23}C_6$ type carbides. (36000 x 4/5)



(B) Transmission electron micrograph of base metal take from the part apart 3 mm from the weld bond. (34000 x 4/5) estimated that zonal finely precipitates occurring at the houndaries between austenite and ferrite would be composed from $M_{23}C_6$ containing small amounts of chromium, iron and molybdenum. However the carbide of M_6C type[13] could not be observed by X-ray as well as electron diffraction.

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