Study of Furnace Atmosphere for Vacuum-Inert Gas Partial-Pressure Brazing

The introduction of high-purity argon into a vacuum effectively suppresses the partial pressure of CO and retards the surface reactions on stainless steels

ABSTRACT. The effects of introducing a continuous flow of high-purity argon into a vacuum on the compositions in the furnace atmosphere, and on the surface reactions of several stainless steels, were investigated.

The increase of partial pressure of CO in the furnace atmosphere is suppressed and the surface reactions of the stainless steels are retarded as the flow rate of the introduced argon is increased.

Introduction

High-temperature vacuum brazing has made great strides during the last 30 years, and it is currently an important production process in many industrial fields. There is every reason to believe that the usefulness of this process will continue to grow (Refs. 1, 2). By brazing under high vacuum, in an atmosphere free of oxidizing gases, a superior product with greater strength and quality may be obtained.

While vacuum brazing has been used mostly to join base metals that are difficult or impossible to braze under other conditions, it can be used to braze most high-temperature alloys. Most high-temperature brazing filler metals are suitable for vacuum brazing also. However, brazing in vacuum has an inherent disadvantage with respect to high vapor pressure elements such as Mn, Ag, and Cu that may be present in brazing filler metals. These filler metals evaporate because of low surrounding pressure, especially during high-temperature, long brazing cycles.

To overcome this problem, the introduction of an inert gas under partial pressure is done to minimize the evaporation of such elements. The choice of gas and its purity, the proper settings of the furnace pressure, and the gas flow rate were established. The effects of these factors on the wetting of the base metal by the molten brazing filler metal and on the brazeability were studied in a previous investigation (Ref. 3).

Wetting and brazeability seem to re-

BY A. SAKAMOTO

late strongly to the surface conditions of the base metals during the brazing cycle, and the surface conditions may depend on the brazing atmosphere, *i.e.*, the elements in the atmosphere and their quantity. However, these relationships are not well documented.

The investigation described in this paper was planned to accomplish the following:

1) Determine the effects of the introduction of continuously flowing highpurity inert gas into a vacuum on the compositions in the furnace atmosphere.

2) Discuss the surface reactions of the several stainless steels brazed in a vacuum-inert gas partial pressure atmosphere.

Experimental

Equipment

The experiments were conducted in a vacuum-inert gas atmosphere furnace — Fig. 1. The furnace was a cold-wall, two-chamber vertical type. Both the heater and the insulating materials were graphite.

An isolation valve between the heating and cooling chambers prevented gas absorption by the heater, the insulating materials, and the inside wall of the heating chamber during furnace loading and unloading. When the heating chamber was opened to air and the furnace was not used for more than 24 h, baking was carried out for 1 h at 1200°C (2190°F) to eliminate absorbed gases.

The pressure in the chamber could be varied from 6.7×10^3 to 1.33×10^{-3} Pa

KEY WORDS

Brazing Stainless Steel Furnace Atmosphere Vacuum-Inert Gas Partial Pressure Brazing Surface Reactions Oxide Film High-Temperature Brazing (50 to 10⁻⁵ torr), and the inert gas flow from 20 to 0 L/min (42 to 0 cfh) was controlled by a needle valve in the inert gas line and an orifice in the evacuation system. Evacuation during the introduction of an inert gas was done by mechanical means only.

High-purity 99.999% argon with $a < 70^{\circ}C$ (94°F) dew point was used as the introducing gas.

The mass spectrometer used for analyzing the furnace atmosphere was a MAQ-150A quadrupole type of ULVAC with a resolution from mass 0 to 150 and a sensitivity of 7.5 X $10^{-3} \sim 7.5 \times 10^{-2}$ A/Pa (1 ~ 10 A/torr) with a minimum detective partial pressure of 5 X 10^{-11} Pa (4 X 10^{-13} torr) for nitrogen.

Test Procedure

The mass spectrometer was used to scan the furnace atmosphere during typical furnace cycles of heating and cooling, where the heating rate was 7.7°C/min (13.9°F/min) from room temperature up to 1150°C (2102°F). It was held at the temperature for 30 min and then cooling occurred in the furnace. The average cooling rate was 8.3°C/min (14.9°F/min).

Types 304 and 17-7 PH stainless steels and iron-based heat-resistant Alloy A286 were used to study the surface reactions during typical furnace cycles under various vacuum-argon gas, partial pressure atmospheres — Table 1. The Alloys 17-7 PH and A286 were selected because they contain the reactive elements aluminum or titanium, which develop extremely stable oxide, nitride and carbide on the surface of the base metals.

The specimens measured about 25 X 25 X 2.5 mm (1 X 1 X 0.1 in.). One face of the specimen was polished with No. 600 emery paper and all surfaces were washed by ultrasonic cleaning with acetone.

After heat treatment, the surface states of specimens were analyzed by electron spectroscopy for chemical analyses (ESCA). The Ar+ etching rate was 1 X $10^{-3} \mu m/min$.

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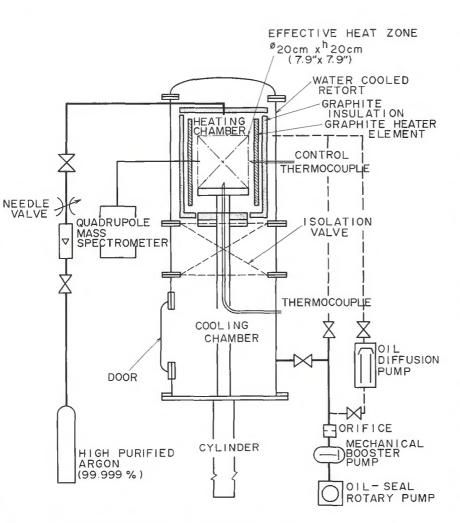


Fig. 1 — Schematic of vacuum-inert gas atmosphere brazing furnace.

Table 1—Chemical Compositions of Alloys (wt-%)											
	С	Si	Mn	Р	S	Ni	Cr	Мо	Ti	Al	V
Type 304	0.05	0.64	1.60	0.020	0.007	10.06	18,80	_		_	_
17-7 PH	0.09	0.19	1.00	0.017	0.011	7.09	17.69	_	_	0.92	-
A286	0.04	0.38	1.53	0.013	0.004	25.13	19.25	1.27	2.12	0.16	0.03

Results and Discussion

Furnace Atmosphere Analyses

For purposes of comparison, the first atmosphere analyzed was a high vacuum (1.3 X 10⁻³ Pa — 10⁻⁵ torr) produced by mechanical and diffusion pumping evacuation. The mass spectrometer readings and the furnace pressure are shown in Fig. 2. The ordinate is the reading of mass peak, which gives the relative value corresponding to the partial pressure of constituents, and the abscissa shows the furnace cycle time of heating and cooling.

The C+ (M/e = 12) and O_2^{2+} , O+ (M/e = 16) are considered to be fragments of CO due to their cracking patterns and mutual reactions among constituents. The OH+ is a fragment of H_2O . Therefore, residual gases in high vacuum were principally composed of CO, H_2 and H_2O .

The H₂O content reached the maximum value at 400°C (752°F) during heating and then decreased. On the other hand, the CO and H₂ contents increased with temperature and reached maximum values at the highest temperature, 1150°C (2102°F). The furnace pressure varied in the same manner as the CO and H₂ contents.

Above 400°C (752°F), the CO and H_2 gases showed almost the same behavior, but the H_2O showed a behavior opposite to them. Therefore, it is reasonable to think that the reaction between

moisture in the furnace atmosphere and graphite used as a heating element occurs above 400°C (752°F) and that reaction is as follows: $C + H_2O = H_2 + CO$.

According to Perdijk's thermodynamic calculations (Ref. 4), the principal residual gases in vacuum at the lower temperatures are H_2O and possibly CO_2 or CH_4 , while at the higher temperatures above 427°C (220°F), CO and H_2 prevail.

The results of residual impurity gas analyses of the 13 Pa (0.1 torr) chamber in which the continuous argon flow was introduced at the argon flow rate of 20 and 90 mL/min (0.04 and 0.19 cfh) are shown in Figs. 3 and 4. A noticeable difference was observed between Figs. 3 and 4 in the CO content behavior. The increase of CO content (CO+, C+ and CO2+ readings), which started around 400°C (752°F), during heating was suppressed as the argon flow rate increased. This was as expected from the theoretical considerations, described in previous work (Ref. 3). The argon flow rate dependencies, however, were not observed in the H₂O and CO₂.

The previous study (Ref. 3) showed that the wetting behavior of typical hightemperature brazing filler metals on several corrosion- and heat-resistant alloys is improved as the argon gas flow rate is increased at a given pressure level of the atmosphere.

Figure 5 depicts the mass spectrometer readings of a 13 Pa (0.1 torr) furnace atmosphere with an argon flow rate at 90 mL/min (0.19 cfh). Much outgassing has occurred after the furnace has been unused for about 40 h. In this unbaked dirty chamber, a higher evolution of CO and higher levels of H₂O and CO₂ were observed compared with the normal process results shown in Fig. 4.

Figures 6 and 7 depict the mass spectrometer readings of 133 Pa (1 torr) atmospheres with the argon flow rate set at 200 and 2000 mL/min (0.42 and 4.2 cfh). Much outgassing has occurred after the furnace has been unused for about 40 h. The evolution of CO, H_2 and CO₂ gases was observed in the unbaked dirty chamber when the argon flow rate was lower (Fig. 6), while the increase of these gases was well depressed even in the dirty chamber with the higher argon flow rate - Fig. 7. This indicated that the introduction of a continuous flow of highpurity inert gas acted effectively also in the dirty chamber. This tendency was found in the previous brazeability study (Ref. 3).

Surface Reactions

Typical examples of heat-treated specimens are shown in Fig. 8. The surface of



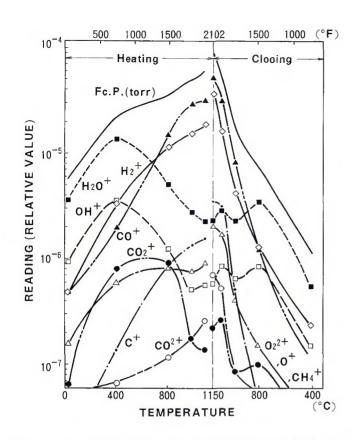


Fig. 2 — Mass spectrometer readings and furnace pressure (Fc. P.) of 1.3×10^{-3} Pa (10⁻⁵ torr) high-vacuum furnace atmosphere.

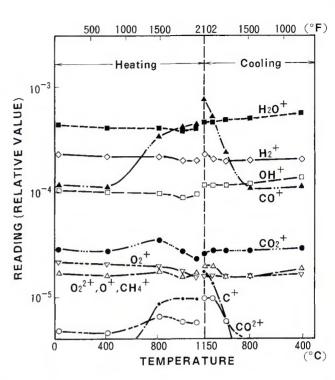


Fig. 3 — Mass spectrometer readings of 13 Pa (0.1 torr) Ar atmosphere with argon flow rate of 20 mL/min (0.04 cfh).

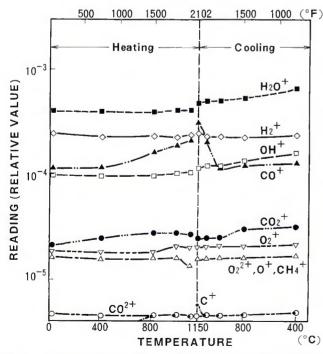


Fig. 4 — Mass spectrometer readings of 13 Pa (0.1 torr) Ar atmosphere with argon flow rate of 90 mL/min (0.19 cfh).

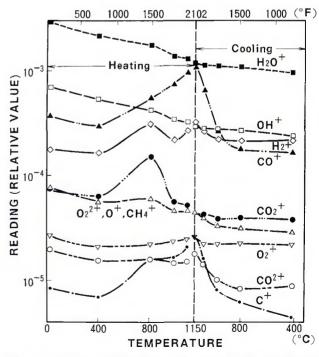
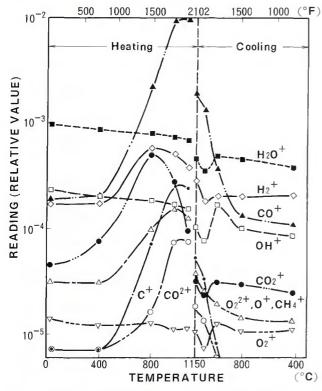


Fig. 5 — Mass spectrometer readings of 13 Pa (0.1 torr) Ar atmosphere with argon flow rate of 90 mL/min (0.19 cfh) in unbaked dirty chamber.



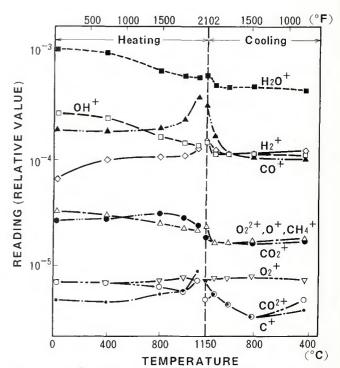


Fig. 6 — Mass spectrometer readings of 133 Pa (1 torr) Ar atmosphere with argon flow rate of 200 mL/min (0.42 cfh) in unbaked dirty chamber.

Type 304 stainless steel had a metallic shine independent of the furnace atmosphere conditions in this study. However, on the 17-7 PH and A286 specimens, there were degrees of discoloration depending on the furnace conditions.

The discolorations of the 17-7 PH specimens heat treated in an unbaked furnace at 13 Pa (0.1 torr) argon, in a baked furnace at 13 Pa (0.1 torr) argon,

and in a 1.3 X 10⁻² Pa (I1X 10⁻⁴ torr) vacuum were gray, light gray (low shine) and metallic shine, respectively.

The discoloration of A286 was significant; changing from purple to light brown depending on the furnace atmosphere conditions, as the impurity gases in the furnace atmosphere were decreasing.

Results of ESCA surface analysis of Type 304 stainless steel annealed at

Fc. C	CONDIT	TION	BASE METAL				
	Pressure Ar Flow (Torr) (ml/min)		<i>TYPE 304</i>	17-7 PH	A286		
0.1	90	Unbaked					
0.7	90	Baked					
10-4	_	Baked					

HEATING CONDITION 1150 °C, 30 min

Fig. 8 — *Typical examples of the appearance of heat- treated specimens.*

Fig. 7 — Mass spectrometer readings of 133 Pa (1 torr) Ar atmosphere with argon flow rate of 2000 mL/min (4.2 cfh) in unbaked dirty chamber.

1150°C (2102°F) for 30 min in a 13 Pa (0.1 torr) argon atmosphere are shown in Figs. 9 and 10. A surface layer thickness of about 1 μ m was formed, consisting mainly of chromium and iron oxides, with chromium oxide the most prominent.

The surface analysis of Type 304 stainless steel annealed in a vacuum of 1.3×10^{-2} (1 X 10^{-4} torr) at 1150° C (2102° F) for 30 min. showed similar results except that the depth of surface oxide was a little thinner at about 0.8 µm.

Even though all three specimens of Type 304 stainless steel were heat treated in different atmospheres, they all showed the same metallic shine on their surfaces. Oxidation took place on the surfaces during the heating cycles, and it is clear that the more impure gases in the furnace atmosphere, the more the oxidation.

It is known empirically that the oxide film, discoloring the surface when heating through a lower temperature range, gradually disappears with an increase in temperature and/or heating time during the vacuum heating of low-alloy steels and stainless steels. In Type 304 stainless steel, heated for 30 min. under a pressure of 1.3 X 10⁻² Pa (1 X 10⁻⁴ torr), the oxide film formed on heating to 750°C (1382°F) disappeared gradually with increasing temperature up to 900°C (1652°F); consequently, the appearance

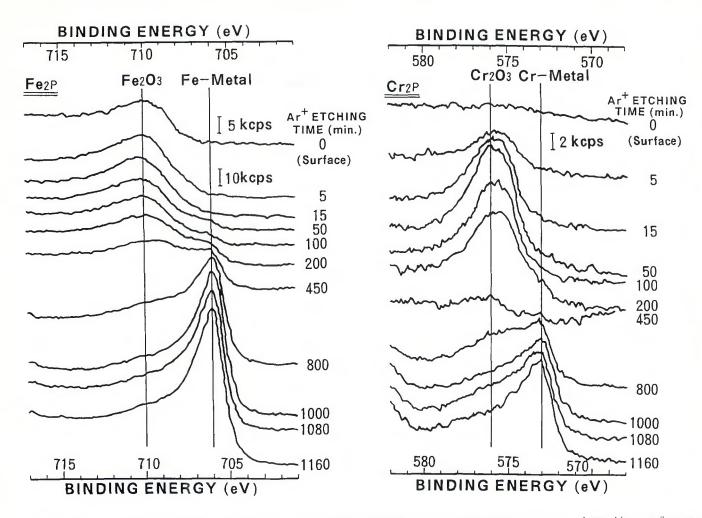


Fig. 9 — ESCA spectra of Type 304 stainless steel annealed at 1150°C (2102°F) for 30 min in 13 Pa (0.1 torr) Ar atmosphere with argon flow rate of 90 mL/min (0.19 cfh).

of the specimen surfaces changed from brownish light blue to metallic shine.

A mechanism of the disappearance, that is a reduction of oxide film by solute carbon in the steel to produce carbon monoxide, was reported on the basis of a thermodynamic analysis (Ref. 5) and experimental evidence (Refs. 5 - 7). Therefore, the surface oxide films are expected to be more easily reduced for the steels when the partial pressure of CO in a furnace atmosphere is lower.

Figures 11 and 12 depict the results of ESCA surface analysis of 17-7 PH stainless steel, after annealing at 1150°C (2102°F) for 30 min under a 13 Pa (0.1 torr) argon atmosphere. As mentioned above, this specimen had a dull appearance. The surface layer consisted of the oxides of aluminum and chromium and of very little iron oxide. Aluminum oxide was the most abundant and its depth was about 1.5 μ m, while the layer of chromium oxide remained at 1.0 μ m in depth.

The ESCA spectra of the 17-7 PH specimen annealed at $1150^{\circ}C(2102^{\circ}F)$ for 30 min. under a vacuum of 1.3 X 10^{-2} Pa (1 X 10^{-2} torr) is shown in Fig. 13. The specimen had a clear metallic shine. The surface

layer was composed predominantly of chromium oxide and a very small amount of aluminum oxide.

From the results of Type 304 stainless steel, the oxidation potential of a 1.3 X 10⁻² Pa (1 X 10⁻⁴ torr) vacuum seems to be almost the same as that of a 13 Pa (0.1 torr) argon atmosphere. An aluminum oxide could be expected to be formed more readily than a chromium oxide. Nevertheless, an aluminum oxide was not formed on the surface of 17-7 PH annealed under high vacuum.

The reason seems to be the pressure level of the furnace atmosphere. The aluminum oxide was considered to be reduced to a volatile aluminum suboxide by the base metal carbon. Lugscheider, et al. (Ref. 7), reported that during the brazing of nickel alloys containing titanium and aluminum, an aluminum oxide layer was predominantly formed at temperatures up to approximately 1130°C (2066°F) and above 1150°C (2102°F). The distinct reduction of the oxygen and aluminum contents in the surface layer was conspicuous. They concluded that the absence of an aluminum oxide (AI_2O_3) surface layer on the base metal annealed in a vacuum at 1150°C (2102°F) can be attributed to aluminum-suboxide volatilization due to carbon reduction, on the basis of the experimental evidence and thermodynamic consideration. On the other hand, the aluminumsuboxide volatilization was considered to be suppressed under 13 Pa (0.1 torr) argon atmosphere.

The ESCA spectra of A286 specimens after annealing at 1150°C (2102°F) under four atmosphere conditions are shown in Figs. 14 to 17. One of the A286 specimens was annealed in a high-purity argon atmosphere (>99.9995%, dew point <70°C) of a hot-wall quartz tube furnace in order to compare with the specimens heated in the cold-wall vacuum furnace. In this heat treatment, the specimen was heated at 50°C/min (122°F/min) from room temperature up to 1150°C (2102°F), and held at the temperature for 15 min.

The ESCA spectra showed the formation of titanium oxide (TiO_2) and titanium carbide (TiC) in the surface layer of all of these four specimens. Little formation of chromium oxide was detected.

Figure 18 shows the ESCA depth profiles of these four specimens. The reacted surface layer had a lack of iron, chromium and nickel, and the layer consisted of ti-

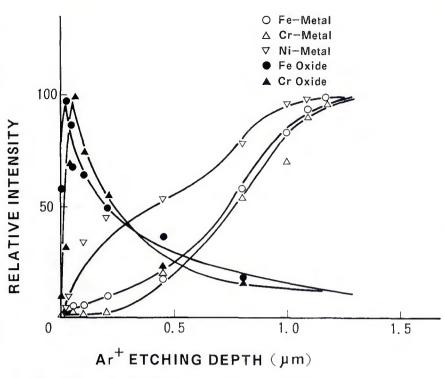
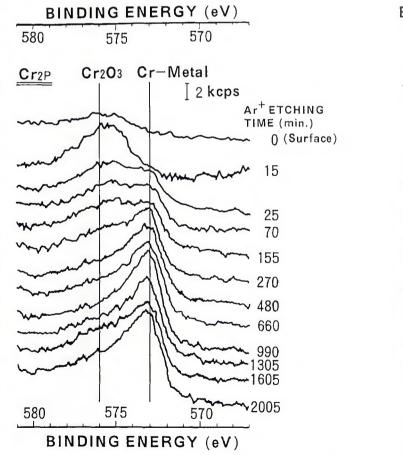


Fig. 10 — ESCA depth profile of the same specimen as seen in Fig. 9.



tanium oxide and titanium carbide in all of the four cases. The concentration of titanium oxide was higher in the exterior layer area, while an increase in titanium carbide was observed in the interior zone of the reacted layer.

After annealing in a vacuum of 1.3×10^{-2} Pa (1 X 10⁻⁴ torr) at 1150°C (2102°F) for 30 min, the surface layer of about 1.3 μ m in thickness was formed. After a 13 Pa (0.1 torr) argon atmosphere annealing, the compositions of surface layer were the same as those of 1.3×10^{-2} Pa (1 X 10⁻⁴ torr) vacuum annealed specimen and the surface layer thickness was about 1.5 μ m, somewhat thicker than that of the vacuum annealed specimen.

After annealing in an unbaked dirty chamber at a 13 Pa (0.1 torr) argon atmosphere, the thickness of the surface layer increased remarkably up to $2.0 \,\mu$ m, and distinct increases of titanium oxide and titanium carbide were observed in the exterior area and in the interior zone, respectively.

A thinner surface layer, about 1 µm in depth, was formed on the surface of the argon-annealed A286, and the profile of titanium oxide showed a narrow

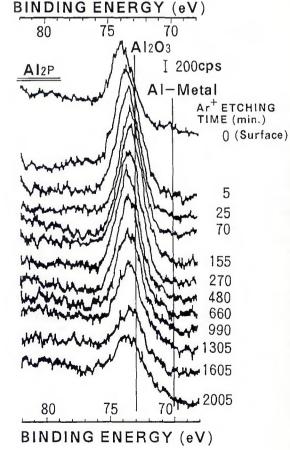


Fig. 11 — ESCA spectra of 17-7 PH stainless steel, annealed at 1150°C (2102°F) for 30 min in 13 Pa (0.1 torr) Ar atmosphere with argon flow rate of 90 mL/min (0.19 cfh).

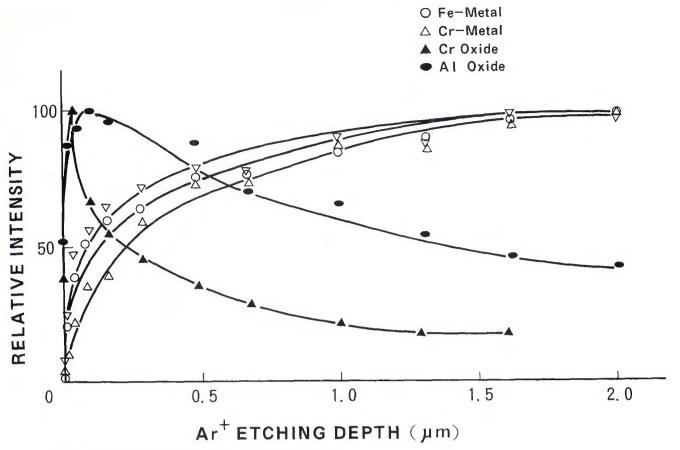
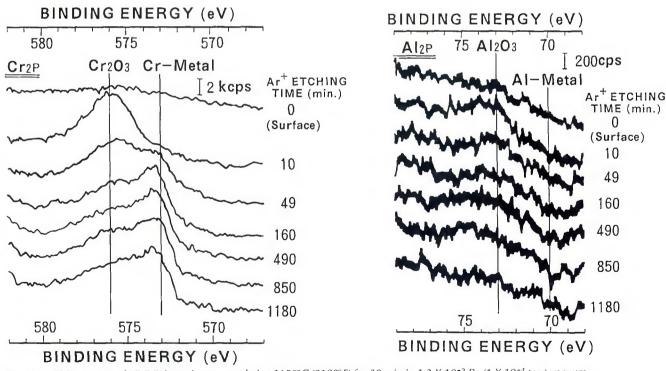


Fig. 12 — ESCA depth profile of the same specimen as seen in Fig. 11.





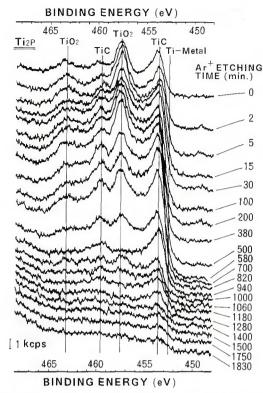


Fig. 14 — ESCA spectra of A286 specimen annealed at 1150°C (2102°F) for 30 min in 1.3 X 10⁻² Pa (1 X 10⁻⁴ torr) vacuum.

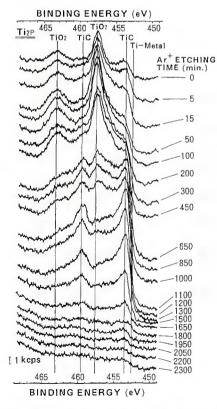


Fig. 16 — ESCA spectra of A286 specimen annealed at 1150°C (2102°F) for 30 min in 13 Pa (0.1 torr) Ar atmosphere with argon flow rate of 90 mL/min (0.19 cfh) in unbaked dirty chamber.

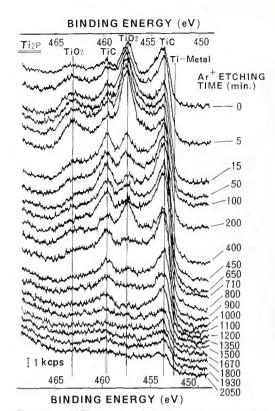


Fig. 15 — ESCA spectra of A286 specimen annealed at 1150°C (2102°F) for 30 min. in 13 Pa (0.1 torr) Ar atmosphere with argon flow rate of 90 mL/min (0.19 cfh).

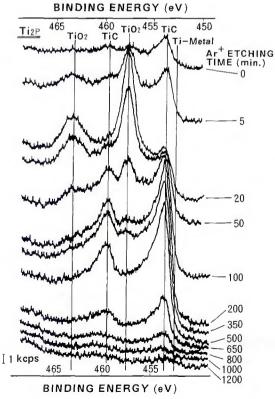


Fig. 17 — ESCA spectra of A286 specimen annealed at 1150°C (2102°F) for 30 min in 101325 Pa (760 torr) Ar atmosphere of hot-wall quartz-tube furnace.

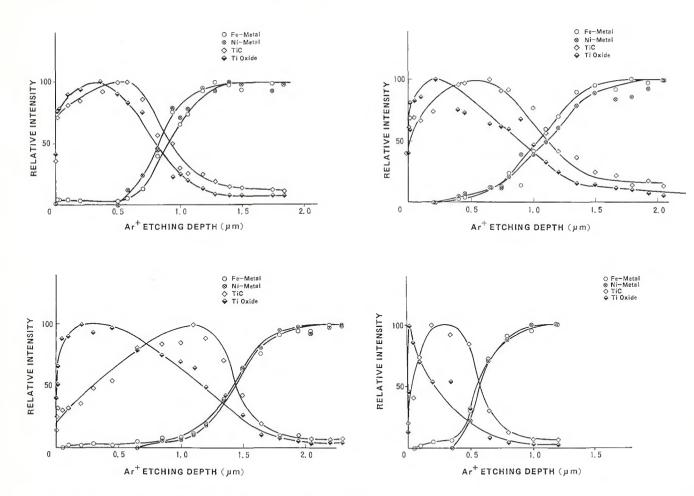


Fig. 18 — ESCA depth profiles of A286 specimen annealed at $1150^{\circ}C$ (2102°F) for 30 min. in four atmosphere conditions. A — 1.3 X 10⁻² Pa (1 X 10⁻⁴ torr) vacuum; B — 13 Pa (0.1 torr) Ar; C — 13 Pa (0.1 torr) Ar, unbaked dirty chamber; D — 101325 Pa (760 torr) Ar, hot-wall quartz tube-furnace.

peak near to the surface. This thinner surface layer may be attributed to the annealing cycle of higher heating rate and shorter holding time at 1150°C (2102°F) rather than to the 13 Pa (0.1 torr) argon atmosphere and the vacuum annealing.

Chromium and iron oxides were not observed in the surface layer of A286 specimens because it is not like Type 304 where the chromium and iron oxides might form when heating through a lower temperature range and disappeared at the higher temperatures due to the reduction by solute carbon in the base metal.

In the titanium-containing Alloy A286, it is more difficult for the oxide film to be reduced during heating compared to a steel which contains no titanium, such as Type 304 stainless steel. This is due to the lack of solute carbon, since most carbon in A286 is consumed in forming titanium carbide.

The formation of titanium carbide in the interior zone of the surface layer of A286 specimens may be the result of the following: 1) The titanium oxide forms on the surface of the steel through the lowering of the concentration of solute titanium in the surface layer. Simultaneously, the reduction of oxide at the surface by solute carbon takes place. 2) Titanium and carbon, which were dissolved in the bulk, diffuse to the surface layer of the steel. 3) Titanium and carbon are combined into titanium carbide and precipitate in the surface layer. There is very little titanium carbide precipitation in the exterior zone of the surface layer, where carbon is consumed in reducing the oxides.

As mentioned earlier, the oxides formed during heating through a lower temperature range are reduced by the carbon in the steel: $MO + \underline{C} = M + CO^{\wedge}$. As the partial pressure of CO in the furnace atmosphere is lowered, the formation of oxides can be suppressed, resulting in a thinner surface reactive layer. Therefore, in the vacuum-inert gas partial pressure atmosphere, the lowering of the partial pressure of CO, as the introduced argon gas flow rate is increased at a given pressure level, can decrease the oxidation potential of the atmosphere.

Conclusion

1) The introduction of a continuous flow of high-purity argon into a vacuum furnace effectively suppresses the increase of CO content, which starts around 400°C (752°F) during heating. This suppresses the surface reactions of the stainless steels, because the surface oxide films that once formed on heating through a lower temperature range are expected to be more easily reduced by the carbon in the steels when the partial pressure of CO in a furnace atmosphere is lower.

2) The reactive surface layers of Type 304 stainless annealed in a vacuum of 1.3 X 10^{-2} Pa (1 X 10^{-4} torr) and in a 13 Pa (0.1 torr) argon atmosphere were composed of the oxides of chromium and iron, with the chromium oxide being the most prominent.

3) In the case of 17-7 PH stainless steel, the reactive surface layer annealed in 13 Pa (0.1 torr) argon was composed of aluminum and chromium oxides. While the reactive surface layer annealed in a vacuum of 1.3×10^{-2} Pa (1 X 10⁻⁴ torr) consisted predominantly of

chromium oxide, the absence of an aluminum oxide seemed to be attributed to aluminum-suboxide volatilization.

4) In the case of A286 heat-resistant steel, the reactive surface layer consisted of titanium oxide and titanium carbide. The concentration of titanium oxide was higher in the exterior layer area, while an increase in titanium carbide was observed in the interior zone of the reheated layer.

Acknowledgment

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