

Wetting in Vacuum-Inert Gas Partial Pressure Atmosphere Brazing

The introduction of continuously flowing high purity inert gas into a vacuum effectively helps the brazing process at high temperatures

BY A. SAKAMOTO

ABSTRACT. The effects of introducing a continuous flow of high purity argon into vacuum on the wetting behavior of high-temperature brazing were investigated.

Contact angles, the spreading of the molten brazing filler metals on the base metals, and evaporation losses of filler metals were measured as a function of the pressure and the introducing gas flow rate. Introducing an adequate flow of argon effectively maintains the partial pressure of impurities in the brazing atmosphere at a very low level; it also reduces the evaporation of the volatile elements of filler metals.

Introduction

In the latter half of the 1950's, Huschke and Hoppin (Ref. 1) as well as Feduska (Ref. 2) described the advantages of high temperature brazing in vacuum vs. brazing in a dry hydrogen atmosphere when applied to superalloys, especially gamma prime type alloys containing reactive elements such as Ti, Al and Cb. Since then, high temperature brazing in vacuum— together with the cold wall vacuum furnace—has been developed to the point where it is now widely accepted in industry.

When brazing in hydrogen and argon atmospheres, gases having very low dew point are required to decrease the oxidation potential of brazing atmospheres. In the actual brazing process, the lower limits of dew point of brazing atmospheres are $-50 \sim -60^\circ\text{C}$ ($-60 \sim -75^\circ\text{F}$) due to the ability of gas dryer and to the outgassing from tubing, retort, works and brazing fixtures.

The partial pressure of water vapor at

the -60°C (-75°F) dew point for hydrogen is equivalent to about 10^{-2} torr. Also, a 10^{-4} torr level can be easily obtained in the vacuum furnace with an equivalent dew point of -90°C (-130°F) even if 10^{-4} torr is for water vapor. Therefore, the cleanliness of a vacuum atmosphere is expected to be better compared to that of a hydrogen atmosphere.

However, brazing in vacuum has an inherent disadvantage with respect to high vapor pressure elements such as Mn, Ag, Cu that may be present in brazing filler metals and/or the base metals; these evaporate because of low surrounding pressure—especially during high temperature, long brazing cycles. When the temperature is kept constant, the metal evaporation rate in the vacuum is a function of the vapor pressure of the metal at the temperature, and the evaporation losses increase with holding time. Therefore, when brazing large assemblies over several hours for the entire brazing cycle, it is possible for brazeability to undergo extreme deterioration in the vacuum due to the appreciable evaporation of high vapor pressure elements in the filler metals.

To overcome this problem, the partial pressure introduction of an inert gas is provided so as to minimize the evaporation of such elements. The choice of gas and its purity, proper settings of the furnace pressure, and gas flow rate are established in this treatment. However, the effects of these factors on the wetting of the base metals by the molten brazing filler metals or on brazeability are not well documented.

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

1. To provide a better understanding of the wetting behavior of typical high temperature brazing filler metals on several corrosion- and heat-resistant alloys in vacuum/inert gas atmospheres.
2. To evaluate the inert-gas introducing effect on brazeability.

Theoretical Considerations

Utilization of the evaporation effect in brazing has been demonstrated for the increased remelt temperature brazing with filler metals containing volatile melting point depressants (Ref. 3). It has also been proved in the aluminum brazing in a vacuum where magnesium vapor is produced during brazing cycle to getter the atmosphere and to react with oxide films present on the aluminum components (Ref. 4).

However, in general, the evaporation of filler metal elements results in poor braze quality because of the loss of molten filler metal and because of the increase of melting temperature range due to a change in composition of the molten filler metal. Therefore, should the selected temperature and pressure be such that appreciable evaporation can be expected, provision should be made to introduce a partial pressure of a dry inert gas into the furnace chamber to minimize the evaporation effect.

Reduction of the evaporation rate by inert gas atmospheres is expressed, according to Epstein (Ref. 5), by the following approximate formula:

$$\frac{V_g}{V_v} = \frac{2.736}{\lambda P} \cdot \left[\frac{1}{b} \right]^{2/3} \frac{[1 + Mm/Mg]^{1/2}}{[1 + \zeta g/\zeta m^{1/3}]^2} \quad (1)$$

where: V_g = evaporation rate of metal in introducing inert gas atmosphere, $\text{g}/\text{cm}^2 \cdot \text{second}$ (s); V_v = evaporation rate of metal in high vacuum, $\text{g}/\text{cm}^2 \cdot \text{s}$; P = pressure of inert gas atmosphere, dyne/cm^2 ; λ = amount related to length of heated area on metal surface, cm; Mg = molecular or atomic weight of inert gas; Mm = atomic weight of volatile metal; ζg = density of inert gas, g/cm^3 ; ζm = density of volatile metal vapor, g/cm^3 ; b = Van der Waals' constant b of inert gas, cm^3/mole .

According to equation (1), when the temperature and the amount of the volatile metal are kept constant, the reduction of the metal evaporation rate is made

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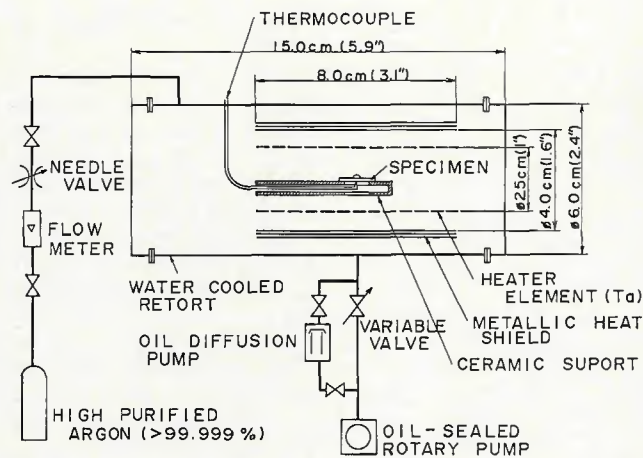


Fig. 2—Schematic of miniature vacuum-inert gas atmosphere furnace for brazing

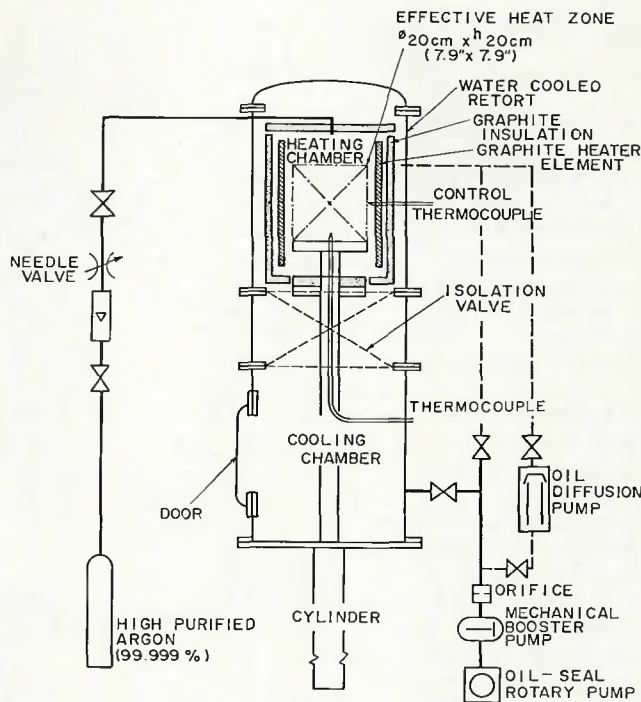


Fig. 3 (right)—Schematic of vacuum-inert gas atmosphere furnace for brazing

shield of the smaller furnace were metallic; each was equipped with evacuation and inert gas introducing systems. The chamber pressure could be set at $10^{-5} \sim 760$ torr by adjusting the needle valve of inert-gas introducing system and the variable valve of evacuation line. In introducing an inert gas, a rotary pump only was operated for evacuation.

The larger furnace was the two-chamber vertical type. Both the heater and the insulating materials were graphite. An isolation valve between the heating and the cooling chambers prevented gas absorption by the heaters, the insulating materials and the inside wall of the heating chamber during furnace loading and unloading. When the heating chamber was opened in air and the furnace not used for more than 24 hours (h), baking was carried out for 1 h at 1200°C

(2190°F) to eliminate absorbed gases.

The larger furnace, like the smaller one, was equipped with an evacuation system and an inert gas introducing system. The pressure in chamber could be varied, from 50 to 10^{-5} torr, and 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 by mechanical means only.

High purity 99.999% argon with a $< -70^{\circ}\text{C}$ (-94°F) dew point was used as the introducing gas in both furnaces. Concerning the pressure in the furnace, more than 10 torr was measured with "U" tube manometer, $10 \sim 10^{-3}$ torr was measured with McLeod gauge, and less than 10^{-3} torr was measured with ionization gauge.

Procedure

In the investigation of wettability, as shown in Fig. 4, 100 mg of filler metal was placed on the center of base metal platelet. After brazing, the spreading and solidified contact angle were measured.

Photographs were taken at $\times 5$ of all the wettability specimens. The boundaries of areas wetted by filler metal shown by the photographs were traced with a planimeter, and the areas of spread were measured. The area of spread (A) divided by the projected area (A_0) of a hypothetical sphere of filler metal 100 mg was defined as a spreading index ($= A/A_0$), which was one of the measures for wettability evaluation.

After the area of spread was measured, the specimens were sectioned to correspond to the center of the filler metal drop and photographs of the sec-

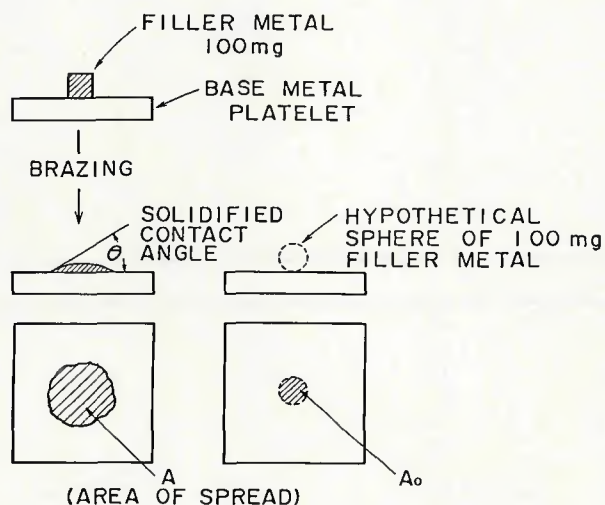


Fig. 4—Contact angle and spreading index determination. Spreading index $= A/A_0$

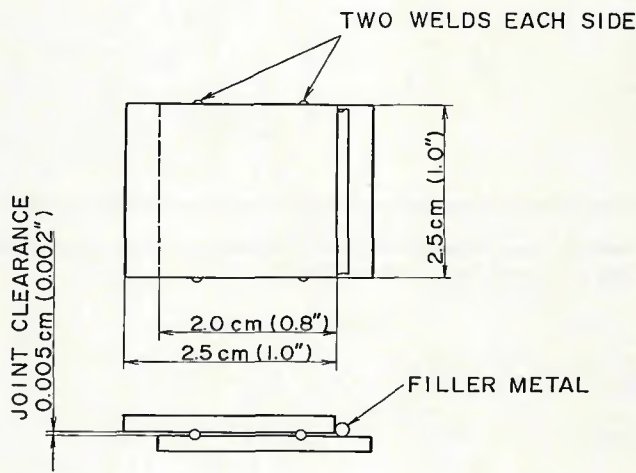


Fig. 5—Lap joint specimen for coverage measurement

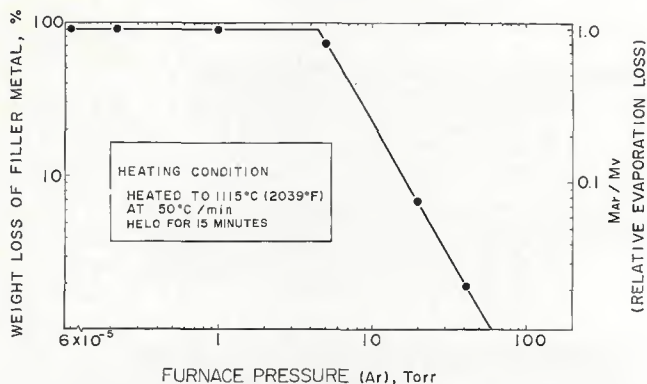


Fig. 6—Relation between evaporation loss of Ag in Ag-10Pd filler metal on Type 304 stainless steel vs. pressure for furnace in which Ar has been introduced. Ar flow rate—50 mL/min; M_v —evaporation loss in high vacuum; M_{Ar} —evaporation loss in Ar atmosphere

tions were taken at X50. The photographs were used to measure solidified contact angle between base metals and filler metals.

The wettability specimens (base metal) measured about 12 by 24 by 1.5 mm (0.5 by 1 by 0.06 in.) for the miniature furnace with the metallic heater, and about 25 by 25 by 2.5 mm (1 by 1 by 0.1 in.) for the larger furnace with the graphite heater. One face of the specimens (both sizes) was polished to a #400 emery paper finish after acid pickling (nitric-fluoric acid solution), and all surfaces were washed by ultrasonic cleaning with acetone.

In the smaller furnace, wettability vs. furnace pressure was determined for an argon gas flow rate at 50 mL/min (0.1 cfh). In this case, BAu-4 and BNi-2 were the brazing filler metals; A-286 and René

41 of the gamma prime type alloys (sensitive to the brazing atmosphere) were the base metals. The specimens were heated at 50°C/min (122°F/min) from room temperature up to brazing temperature, held at the temperature for 15 min and cooled in the furnace. The brazing temperature was 50°C (122°F) above the liquidus of the brazing filler metals.

For Ag-10Pd brazing filler metal, the base metal specimen was Type 304 stainless steel; evaporation losses of the brazing filler elements were measured after heating at 1115°C (2039°F) for 15 min in setting furnace pressures.

The heat-up rate was 50°C/min (122°F/min), and cooling occurred in the furnace. With the smaller furnace, the temperature of a specimen was measured by using a thermocouple attached to the bottom of the specimen.

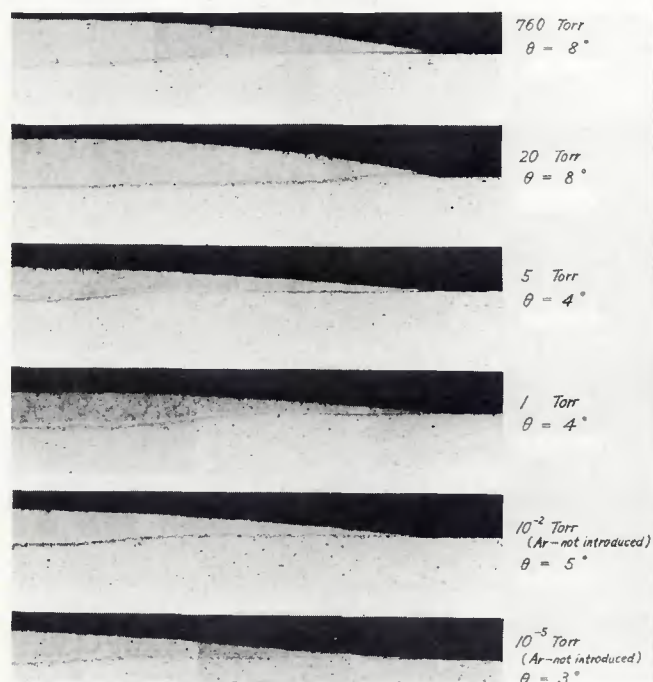


Fig. 8—Effect of Ar introduced furnace pressure on solidified contact angle θ for BAu-4 filler metal/A-286 base metal brazed at 1000°C (1832°F) for 15 min with an Ar flow rate of 50 mL/min. X35 (reduced 59% on reproduction)

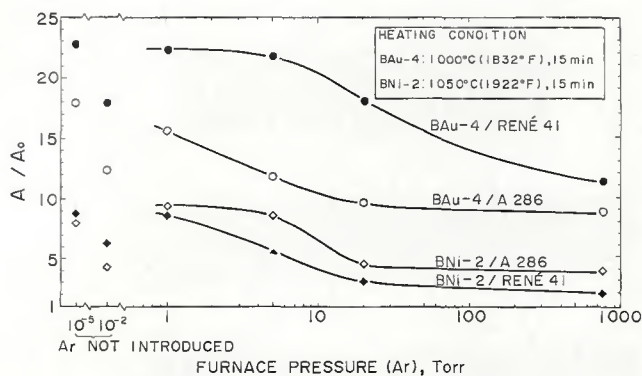


Fig. 7—Effect of Ar introduced furnace pressure on spreading index A/A_0 ; Ar flow rate—50 mL/min

The effect of argon on wettability was examined in the larger furnace with the graphite heater when the furnace pressure was fixed. Ag-10Pd was the filler metal selected to braze Type 347 stainless steel and A-286 at a furnace pressure of 15 and 40 torr. BAu-4 filler metal was selected for brazing Type 347 stainless steel, A-286, Hastelloy X and Inconel 718 at furnace pressures of 15, 1, 0.1, 1.5×10^{-2} and 1×10^{-4} torr. Brazing filler metal Ni-15Cr-3.5B was also used for the four base metals, with furnace pressures of 1, 0.1 and 1×10^{-4} torr. In the case of 1.5×10^{-2} and 1×10^{-4} torr, argon was not introduced.

To investigate the relevancy of the wettability and brazeability for the typical combination of the brazing filler metal and the base metal, the joint coverage was measured by brazing the single lap

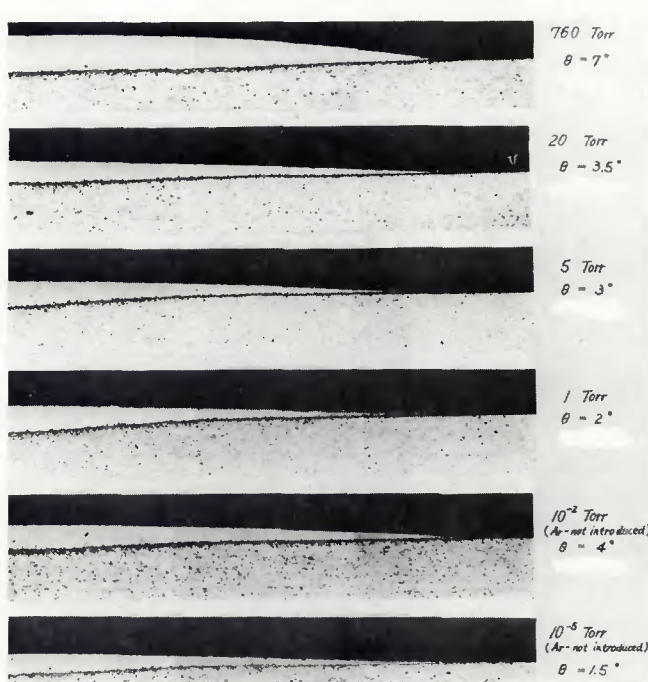


Fig. 9—Effect of Ar introduced furnace pressure on solidified contact angle θ for BAu-4 filler metal/René 41 base metal brazed at 1000°C (1832°F) for 15 min with an Ar flow rate of 50 mL/min. X35 (reduced 59% on reproduction)

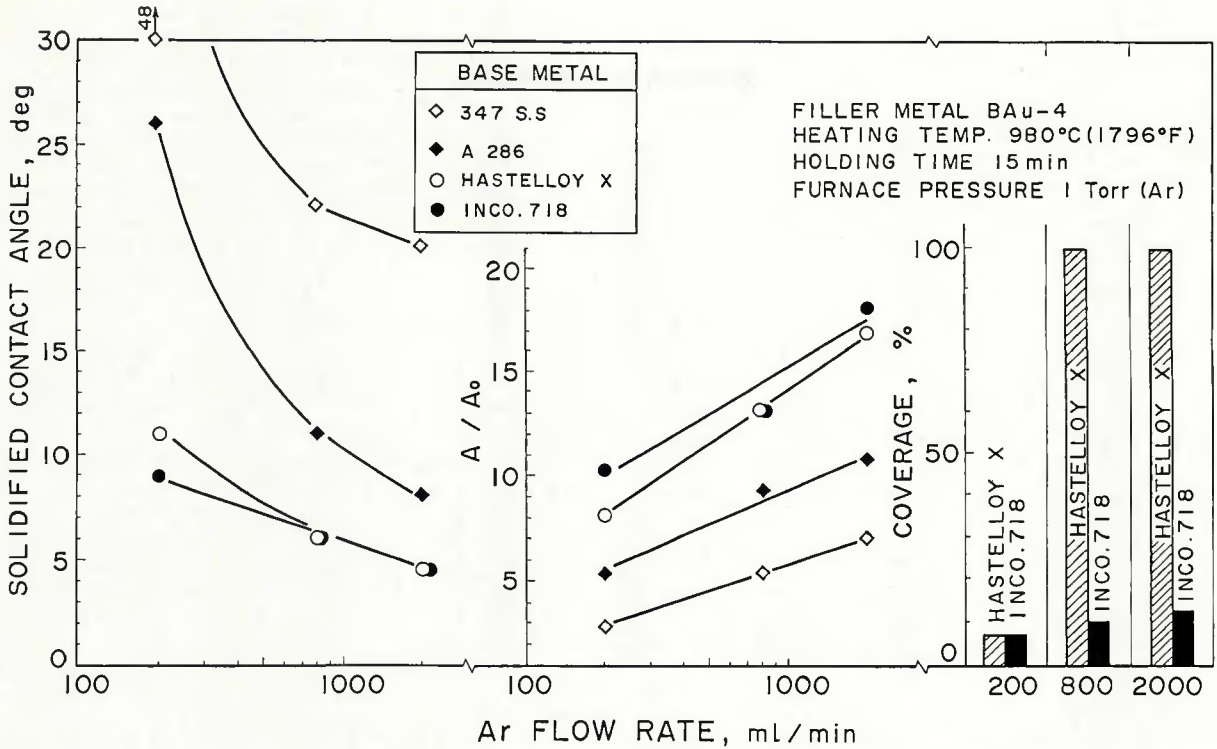


Fig. 20—Effects of Ar flow rate on solidified contact angle, spreading index (A/A_0) and coverage of joints—heated in unbaked dirty chamber

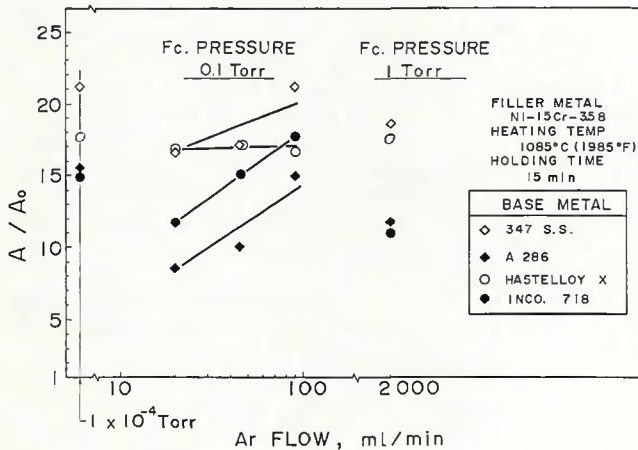


Fig. 21—Effects of Ar flow rate and furnace pressure on spreading index (A/A_0) for Ni-15Cr-3.5 B filler metal

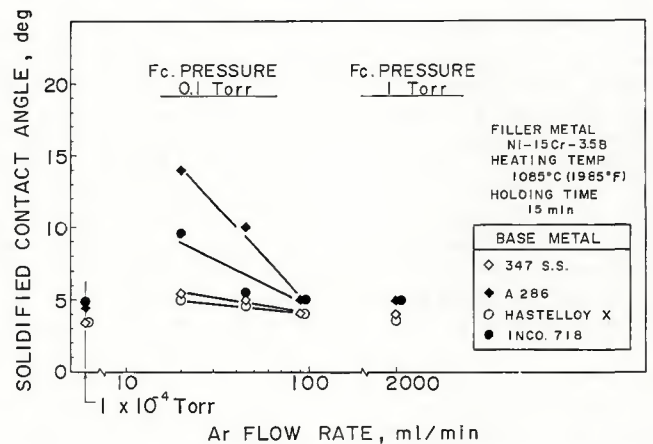


Fig. 22—Effects of Ar flow rate and furnace pressure on solidified contact angle for Ni-15Cr-3.5 B filler metal

pared with obtained gamma prime type alloys, A-286 and Inconel 718. Hastelloy X had the best wettability.

The measurement results of the coverage for single lap joints brazed with the same batch as the wettability specimens are given in Fig. 18. As expected from wettability test results, the coverage increased as the argon flow rate was increasing at the same pressure. On the other hand, the ability of brazing filler metal to fill joints in Inconel 718 was extremely poor even though its wettability was good. Conversely, the joint filling of Type 347 stainless steel was considered to be satisfactory even though its wettability was poor.

The effects of brazing variables on joint filling (i.e., brazeability) can be eval-

uated by the wettability determined with the contact angle or by filler metal drop spreading when the base metal-filler metal combination remains the same. However, the data suggested that the joint filling or the brazeability cannot always be evaluated by contact angles or the spreadability for any base metal-filler metal combinations.

Figures 19 and 20 depict the results of specimens brazed in the atmosphere having much outgassing and without furnace baking after the furnace has been unused for about 40 hours. In this case, wettability and the coverage depended on the argon flow rate as indicated by Fig. 13 and by the data for the 1 torr furnace pressure in Figs. 16 and 18. Notable degradation at the lower flow rate

was found, and the difference from the normal process disappeared at the higher flow rate—that is, introducing a continuous flow of high purity inert gas acted effectively in the dirty chamber.

Results obtained with Ni-15Cr-3.5B filler metal are shown in Figs. 21 to 23. Wettability increased, and the coverage of the brazed joints was improved as the argon flow rate increased. Wettability obtained with this brazing filler metal was poor for the gamma prime type alloys A-286 and Inconel 718, and good for the solid solution type alloys—that is, for Type 347 stainless steel and Hastelloy X.

The results obtained with Ag-10Pd filler metal are shown in Figs. 24 to 26. Brazing was performed at furnace pressures of 15 and 40 torr to reduce silver evapora-

FILLER METAL Ni-15Cr-3.5B
 BRAZING TEMP. 1085°C (1985°F)
 HOLDING TIME 15 min

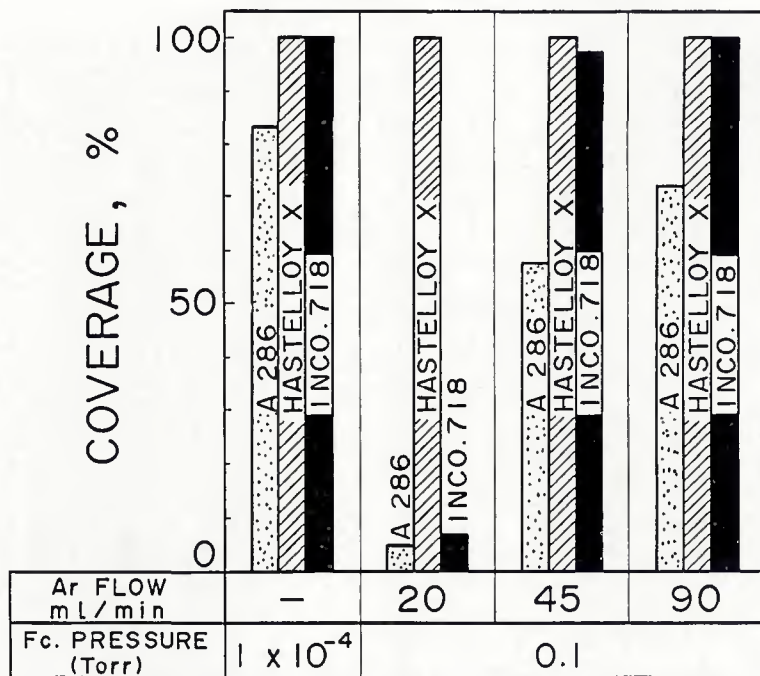


Fig. 23—Effects of Ar flow rate on coverage of lap joints brazed with Ni-15Cr-3.5 B alloy filler metal

tion. As with BAu-4 and Ni-15Cr-3.5B, wettability increased and the coverage was improved when the argon flow rate was increased. It is obvious from the comparison of data for the 15 and 40 torr furnace pressures that the low pressure of 15 torr had a good brazeability at the same argon flow rate.

The Ag-10Pd filler metal did not wet the gamma prime type alloy A-286 and did not fill the joints at all. Evaporation losses of the brazing filler metal detected

from wettability specimen weight losses were 2.4–3.6% at 15 torr and 0.6–1.0% at 40 torr; their dependence on the argon flow rate was not detected.

Strength of Joints Brazed in Vacuum-Inert Gas Introducing Atmosphere

Type 304 stainless steel single-lap joint specimens were brazed in several atmospheres with BAu-4 and Ag-10Pd. This was done to clarify the effects of the brazing atmosphere pressures on the

strength of the brazed joints. The test was performed in accordance with AWS C3.2 (Ref. 8), except that the joint clearance was 0.05 mm (0.002 in).

The test results are given in Figs. 27 and 28. With BAu-4 filler metal, (Fig. 27), evaporation of the brazing filler metal was not detected at the brazing temperature, and the effects of the atmospheres on the joint strength were not found.

With Ag-10Pd filler metal as indicated by Fig. 28, high joint strength was obtained using argon at 4 torr as well as argon at 16 torr + helium at 4 torr, compared with the joint brazed at 760 torr in a hydrogen atmosphere. Under these low pressures, the strength of the joint would be improved as the result of the palladium enrichment in the filler metal due to the silver evaporation.

The evaporation losses of filler metal, measured with the wettability specimens (500 mg of filler metal) brazed at the same time, were 16% at 4 torr (Ar) and 3.5% at 16 torr (Ar) + 4 torr (He). The braze quality of the joint specimens brazed even in the 4 torr argon atmosphere was sound.

Conclusion

The introduction of a continuous flow of the high purity argon into vacuum furnace effectively maintains the partial pressure of impurities in the brazing atmosphere at a very low level. This can be obtained in the pumping range of mechanical evacuation and it reduces the evaporation of volatile filler metal elements if the adequate controls of the furnace pressure and the argon flow rate are provided. Specific points to be noted are:

1. At a given argon flow rate, wetting improves with decreasing pressure of the

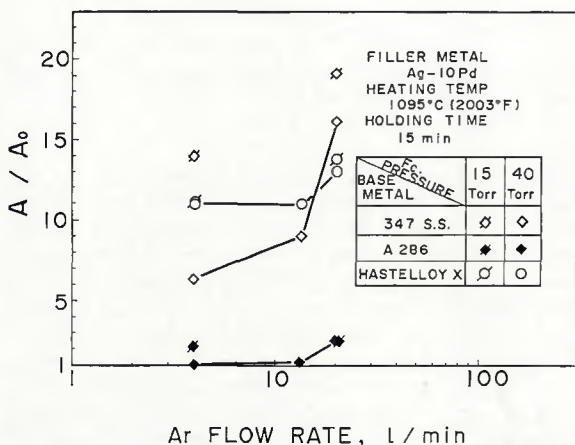


Fig. 24—Effects of Ar flow rate and furnace pressure on spreading index (A/Ao) for Ag-10Pd filler metal

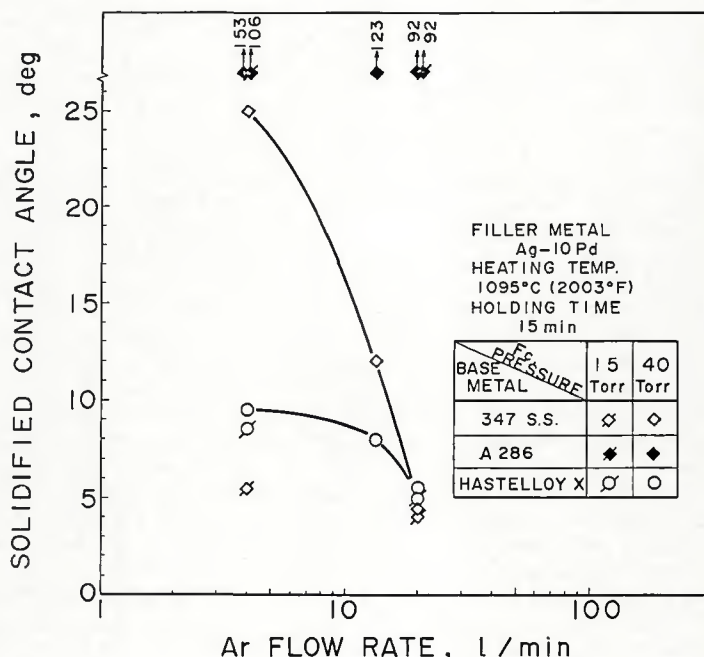


Fig. 25 (right)—Effects of Ar flow rate and furnace pressure on solidified contact angle for Ag-10Pd filler metal

brazing atmosphere. Furthermore, at a given pressure level of the atmosphere, wetting improves when the introducing gas flow rate is increased.

2. In the case of Ag-10Pd filler metal/solid solution type alloys, evaporation losses of silver in the brazing filler metal could be limited to less than several wt-%, if the brazing atmosphere pressure exceeded 15 torr argon; brazing could be carried out when using an adequate gas flow rate above the 15 torr pressure level.

3. With BAu-4 and BNi-filler metal/gamma prime type alloys, argon at an atmosphere pressure of 0.1 ~ 1 torr seemed to give the same wettability and joint filling as brazing in a 10^{-4} torr vacuum, provided gas flow was adequate.

4. The joint strength of Type 304 stainless steel brazed with Ag-10Pd filler metal increased as the pressure of brazing atmosphere was lowered; this occurred because of palladium enrichment of the filler metal due to the silver evaporation.

Acknowledgment

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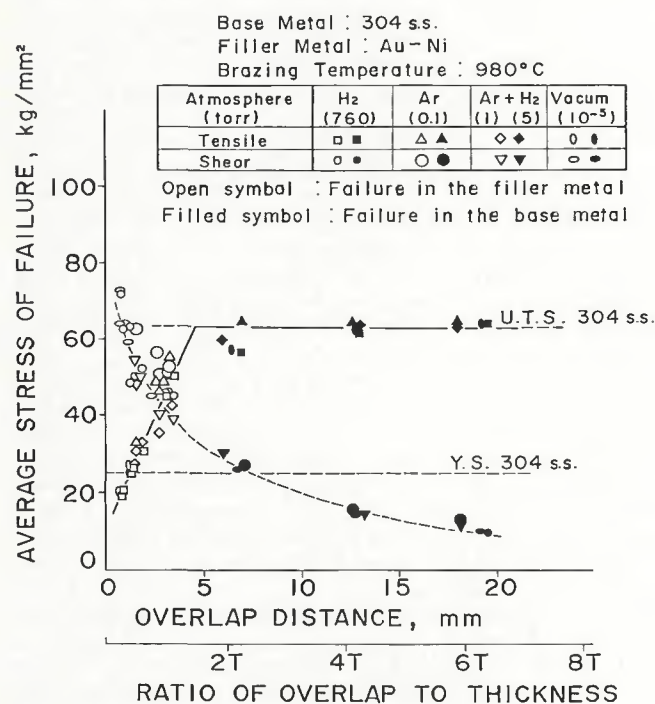


Fig. 27—Shear test data for Type 304 stainless steel joints brazed with BAu-4 filler metal in various atmospheres

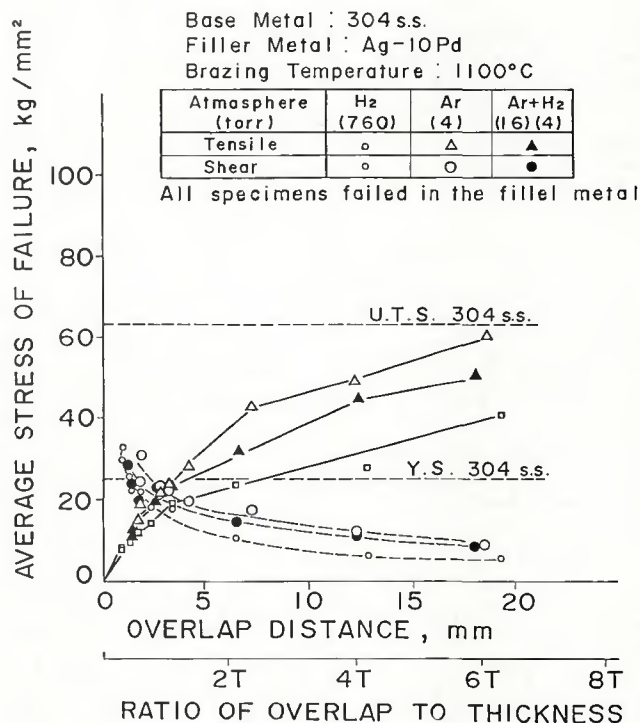


Fig. 28—Shear test data for Type 304 stainless steel joints brazed with Ag-10Pd filler metal in various atmospheres

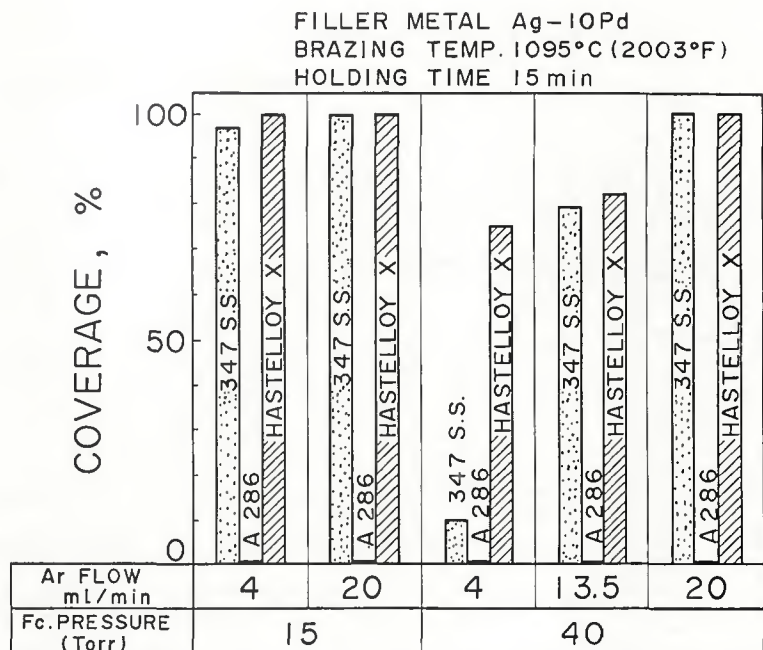


Fig. 26—Effects of Ar flow rate and furnace pressure on coverage of lap joints brazed with Ag-10Pd alloy filler metal