Nickel Brazing Below 1025 C of Untreated Inconel 718

Use of a getter makes nickel brazing of Inconel 718 and X-750 below 1025 C (1875 F) as easy as nickel brazing of stainless steels in vacuum

BY J. CHRISTENSEN AND K. RØRBO

ABSTRACT. Nickel brazing of untreated Ni-Cr-Fe high temperature alloys containing reactive metals such as Ta, Ti and Nb (Cb) normally requires brazing temperatures above 1075 C (1970 F) and a vacuum better than 10^{-5}mm Hg. Brazing of these alloys at lower temperatures and/or at higher pressures has previously involved a time consuming pretreatment in which the oxides formed by the reactive metals are eliminated.

This paper describes a method in which the dewetting effect of these oxides is eliminated during the brazing cycle by means of a getter material, e.g. zirconium.

The new process applied to Inconel 718 and X-750 and with BNi6 and BNi7 as brazing filler metals results in perfect wetting and joint filling at 1025 C (1877 F) under a vacuum of only 10^{-2}mm Hg. The only pretreatment necessary for Inconel 718 and X-750 is degreasing.

Corrosion tests on the brazed joints in water at 290 C (550 F) have shown a corrosion rate of 0.3-2.3 μm/year (0.01-0.09 mils/year); this may be compared with heat treated and unbrazed Inconel 718 which has shown a corrosion rate of less than 0.1μm/year (0.004 mils/year).

The authors are with the Metallurgy Department, Research Establishment Riso, Danish Atomic Energy Commission, Roskilde, Denmark.

Paper was presented at the 55th AWS Annual Meeting (5th International AWS-WRC Brazing Conference) held at Houston during May 7-9, 1974.

Introduction

The presence of reactive metals such as Ta, Ti, and Nb(Cb) in some Ni-Cr-Fe high temperature alloys — e.g. Inconel 718 and X-750* — produces in these nickel alloys surface oxides which are very difficult to reduce and wet in a vacuum or in a dry hydrogen atmosphere. Therefore brazing of these alloys normally requires a vacuum better than 10^{-5}mm Hg and brazing temperatures higher than 1075 C (1970 F).

Unfortunately brazing and diffusion at these high temperatures often cause grain growth in the base metal which cannot always be accepted. The mechanical strength of the parts to be brazed will often be so low at these high brazing temperatures that some kind of support is needed. Furthermore the aggressive effect of some brazing filler metals — e.g. BNi7 — is severe at these high temperatures (Ref.3).

Previously the only way to lower this brazing temperature has been to eliminate the surface oxides formed by the reactive metals — at least until the brazing filler metal has wetted and flowed. This is normally done in one of the following two ways:

1. Nickel plating of the base metal, either chemically (electroless nickel plating) or electrolytically to a thickness of 15-20 μm (0.6-0.8 mil).
2. Removing the surface oxides and the reactive metal alloying elements just below the surface to prevent new surface oxides from forming during heating to the brazing temperature.

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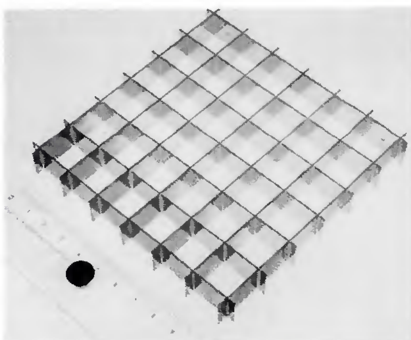
Of course both of these methods are very time consuming in the preparation work needed before brazing. Furthermore the surface treatments may change the final base metal composition in thin plates, e.g. honeycomb structures. With electrolytic nickel plating the plated layer is thicker at the edges; problems may arise in obtaining the tight fit which is required for maximum ductility of the nickel brazed joints.

Getter Brazing

A much easier way to overcome these problems could be the use of a very strong oxide reducing medium, which would eliminate the surface



The 49 cross joints in this untreated Inconel X-750 nuclear spacer were brazed simultaneously using BNi7 and a zirconium getter oxides of these Inconels directly during the brazing operation. Such a process would be very attractive if the metal-metal oxide reactions would take place at reasonable temperatures and vacuums.

A dry hydrogen atmosphere is very often used as an oxide reducing atmosphere in high-temperature brazing. But considering the composition of two commonly used high-temperature nickel alloys — Inconel 718 and X-750 — given in Table 1, it is seen from Fig. 1 that the hydrogen atmosphere must be very dry, if the brazing temperature is to be kept below 1050 C (1922 F). From the curves in Fig. 1 it is also seen, that NbO (CbO) and Ta_2O_5 theoretically only require a dew point of -65 C (-85 F) or 6 ppm of water vapor, while TiO and Al_2O_3 require a dew point of the hydrogen atmosphere which is so low that it will be prohibitive under manufacturing conditions.

Another solution to the problem could be the use of an element which is more oxide reducing than those which are present in the base metal. Such an element should at least prevent the Inconels from further oxidation and might perhaps also reduce the surface oxides that are present already before the brazing operation. A look at Fig. 2 and other special tables for the standard free energies of formation of oxides reveals that zirconium could be a possibility. The standard free energy of formation of ZrO_2 is seen in Table 2 to be lower than that of the oxides of the alloying elements in the Inconels stated in Table 1. Zirconium has a melting point of 1860 C (3380 F) and a boiling point of nearly 3000 C (5400 F). It should therefore give no problems in a high vacuum at 1000 C (1832 F) to 1200 C (2192 F). Furthermore zirconium is easy to form and machine. Zirconium metal was therefore chosen for an investigation of its possibility to act as a getter for nickel brazing of the Inconels 718 and X-750.

Materials and Equipment:

The experiments were carried out in two different high vacuum furnaces; a small one which could be kept perfectly clean with a good control of the vacuum and the temperature, and a 1.3 cubic metre (46 cubic foot) industrial high vacuum furnace for brazing of larger components.

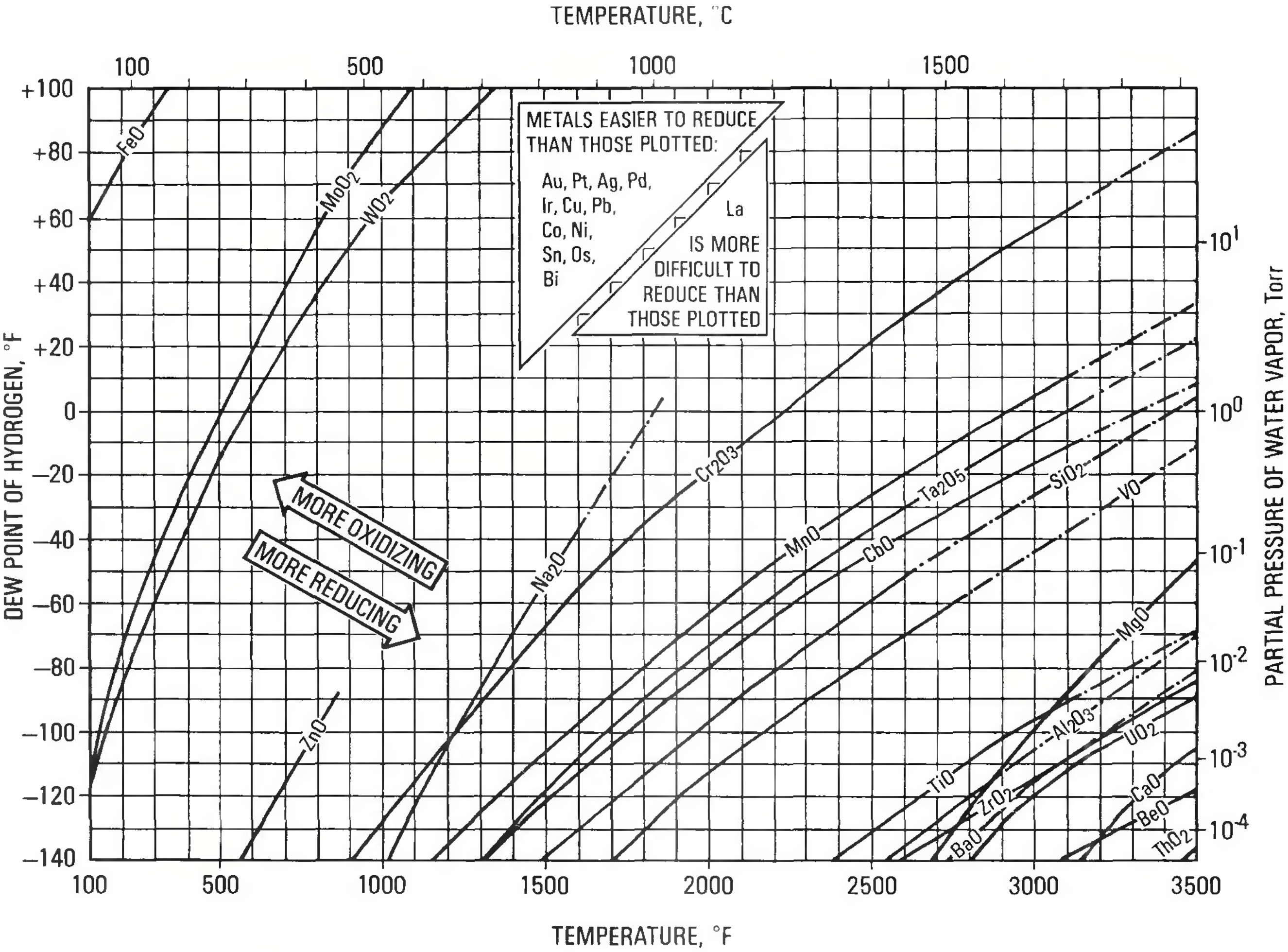


Fig. 1 — Metal-metal oxide equilibrium in pure hydrogen atmospheres (Ref. 2)

Table 1 — Chemical Composition of Inconel 718 and X-750

	Ni	Cr	Fe	Mn	Cu	Si	S	Co	Mo	C	Nb Ta	Ti	Al
Inconel 718	50 55	17 21	Bal		max. 0.30	max. 0.35	max. 0.05	max. 1.0	2.80 3.30	max. 0.08	4.75 5.50	0.65 1.15	0.20 0.80
Inconel X-750	70 min.	14 17	5 9	max. 1.0	max. 0.5	max. 0.5	max. 0.01			max. 0.08	0.7 1.2	2.25 2.75	0.4 1.0

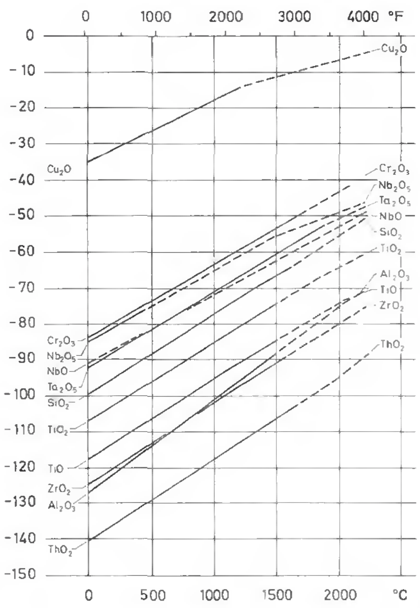


Fig. 2 — Free energy of formation in kcal/gram-atomic wt of oxygen (Ref. 1)

Specimens

Two different types of specimens — both Inconel 718 and X-750 — were used. The wetting effect of the two brazing filler metals was measured by means of the contact angles — Fig. 3 — on 0.8 mm (0.03 in.) thick platelets.

The joint filling characteristics were investigated on actual joints — each cross in the spacer which is shown in the lead photo (see title page).

Brazing Filler Metals

Initially two brazing filler metals, BNi6 and BNi7, were used — Table 3. It soon became clear, however, that the BNi7 alloy required better oxide reducing conditions than the BNi6, and the experiments were therefore continued using only the more difficult brazing filler metal BNi7.

The brazing filler metal, in powder form, was placed on the degreased platelets or in one corner of the cross-specimens and fixed there by means of a cement which volatilizes completely in the vacuum at a few hundred degrees Centigrade.

Table 2 — Free Energy of Formation in kcal/gram-at. wt. of Oxygen (Ref. 1)

Me ⇄ MeO	900 C (1652 F)	1000 C (1832 F)	1100 C (2012 F)	1200 C (2192 F)	1300 C (2372 F)
ZrO_2	-104	-101.5	-99.5	-97	-95
Al O_3	-103.5	-101	-98.5	-96	-93.5
TiO	- 97	- 95	-92.5	-90.5	-88.5
TiO_2	- 87	- 85	-82.5	-80.5	-78.5
Ta_2O_5	- 73	- 71	-69	-67	-65
NbO	- 74.5	- 72.5	-70.5	-68.5	-66.5
Nb_2O_5	- 67.5	- 65.5	-63.5	-61.5	-59.5
Cr_2O_3	- 66	- 63	-60	-56.5	-53.5

Table 3 — Nominal Composition of Brazing Filler Metals

	Ni	Cr	P
BNi6 (NB10) [a]	Bal		11
BNi7 (NB50) [a]	Bal	13	10

(a) Product of Wall Colmonoy Corp., Detroit, Mich.

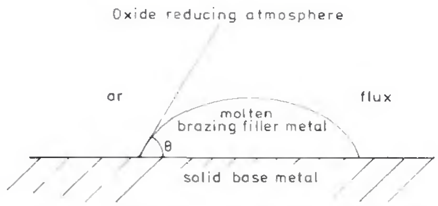


Fig. 3 — Contact angle for measuring the wetting effect

Table 4 — Contact Angles Measured after Brazing for 1 h at 1065 C (1950 F) with Brazing Filler Metal BNi7

	For brazing atmospheres of		
Base metal	10^{-5}mm Hg	10^{-2}mm Hg	10^{-2}mm Hg + Zr-getter
Inconel 718	1 deg	No Wetting	3 deg
Inconel X-750	1 deg	No Wetting	5 deg

Table 5 — Contact Angles Measured after Brazing for 1 h at 1000 C (1832 F) with Brazing Filler Metal BNi7

	For brazing atmospheres of		
Base metal	10^{-5}mm Hg + Zr-getter	10^{-5}mm Hg + Ti-getter	10^{-5}mm Hg
AISI 304	—	—	1.5 deg
Inconel 718	1 deg	1 deg	15 deg
Inconel X-750	3 deg	3 deg	No Wetting

Brazing Cycle

After the specimens had been placed in the furnaces, the laboratory furnace was evacuated to 5 × 10^{-6}mm Hg, while the larger manufacturing furnace was evacuated to 10^{-5}mm Hg. The temperature was then raised to the brazing temperature and held there for one hour. Finally the specimens were allowed to cool under vacuum to 200 C (392 F).

Equipment

The small furnace was a quartz tube, high vacuum furnace, Fig. 4. The 43 mm (1¾ in.) 10 by 0.5 m (20 in.) quartz tube is connected to a high vacuum pumping unit which gives a vacuum better than 5 × 10^{-6}mm Hg around the specimens. A resistance heated tube furnace which is kept at the brazing temperature ±5 C (±9 F) is pushed forward to bring the specimens in the quartz tube up to temperature. The temperature is measured by thermocouples in or a few millimetres above the specimens which are placed on Fiberfrax*. When getter brazing is used, the Fiberfrax will be placed in an open zirconium tube inside the quartz tube.

The larger furnace was an induction heated high vacuum furnace, Fig. 5. The 250 mm (10 in.) Teflon coated induction coil is placed in the 1.3 cubic metre water-cooled tank which is evacuated, using a 2000 l/s (71 cu ft/s) oil diffusion pump to a vacuum which was measured to be better than 10^{-5} mm Hg at the top.

All experiments were carried out with a molybdenum tube 150 mm (6 in.) ID by 300 mm (12 in.) as susceptor.

A special arrangement was tried to obtain a very good vacuum localized at the brazing zone. This was necessary because there was a rather long

**Product of the Carborundum Company, N.Y., U.S.A.*

Table 6 — Contact Angles Measured after Brazing in a Vacuum of 10^{-2}mm Hg (forepump) and a Zr-getter for 1 hr at 1025 C (1877 F) and 1065 C (1950 F) with Brazing Filler Metal BNi7

	Brazing temperature	
Base metal	1025 C (1877 F)	1065 C (1950 F)
AISI 304	1 - 2 deg	—
Inconel 718	2 deg	3 deg
Inconel X-750	3 deg	5 deg

distance between the susceptor and both the vacuum measuring instruments and the connection to the vacuum pumps. The arrangement involved an extra oil diffusion pump mounted directly underneath the molybdenum susceptor, which in this case was furnished with a lid.

When the small specimens were getter brazed, they were placed inside an open zirconium tube in the susceptor.

The final spacer constructions, 113 mm (4½ in.) by 113 mm (4½ in.) by 13 mm (½ in.), were getter brazed in the same way in an open rectangular zirconium tube, 125 mm (5 in.) by 125 mm (5 in.) by 37 mm (1½ in.) — Fig. 6.

All experiments utilizing a zirconium tube as getter were carried out without the extra oil diffusion pump underneath the susceptor.

Results

The brazing filler metal BNi7 showed a very good wetting behavior on the Inconel 718 and X-750 specimens — Table 4 — with contact angles of only a few degrees when brazed in the small laboratory furnace at 1065 C (1950 F) and at a vacuum better than 10^{-5}mm Hg.

Unfortunately it proved impossible to obtain wetting of identical materials at the same temperature when brazing was carried out in the larger induction heated manufacturing furnace, even though the vacuum was measured as 10^{-5}mm Hg. There was still no wetting after very careful cleaning and degassing of the inside of the furnace. Even the arrangement where an extra oil diffusion pump was mounted directly on a closed susceptor was not sufficient to get the BNi7 to wet the Inconels 718 and X-750 at 1065 C (1950 F).

The idea of utilizing the effect of a getter material was then investigated in the small laboratory quartz tube furnace.

The results seen in Tables 4 and 5 show that zirconium is very efficient in promoting the wetting effect of the BNi7 brazing filler metal on both of the Inconels.

When brazing was carried out at 1065 C (1950 F), Table 4 shows that the wetting effect is practically the same in a high vacuum (10^{-5}mm Hg) without a getter as it is in a primary vacuum (10^{-2}mm Hg) which contains a zirconium getter. No wetting at all was obtained in a primary vacuum without a getter at 1065 C (1950 F).

When the brazing temperature is lowered to 1025 C (1877 F), Table 6 shows that there is still perfect wetting in a primary vacuum containing a zirconium getter.

With these results in mind some small specimens were prepared for brazing in the manufacturing furnace

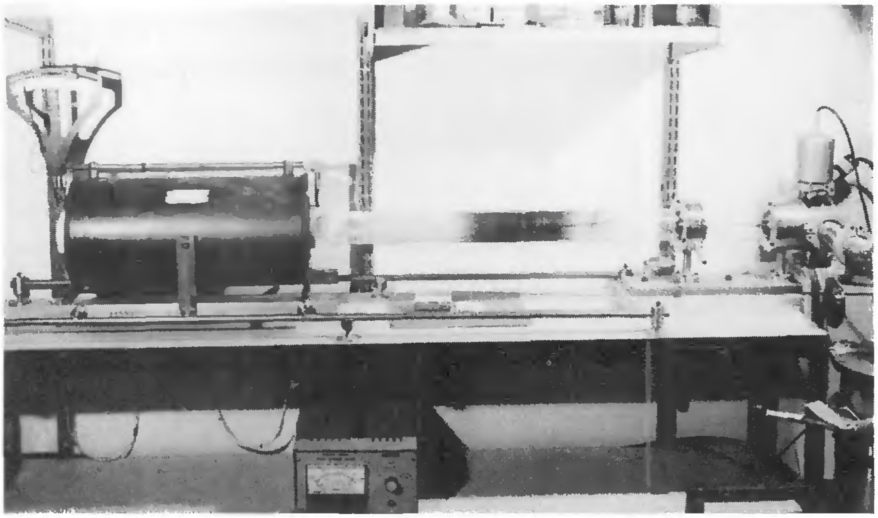


Fig. 4 — Quartz tube high-vacuum furnace with furnace withdrawn from the quartz tube. The zirconium tube shown on the table is placed in the quartz tube for getter brazing

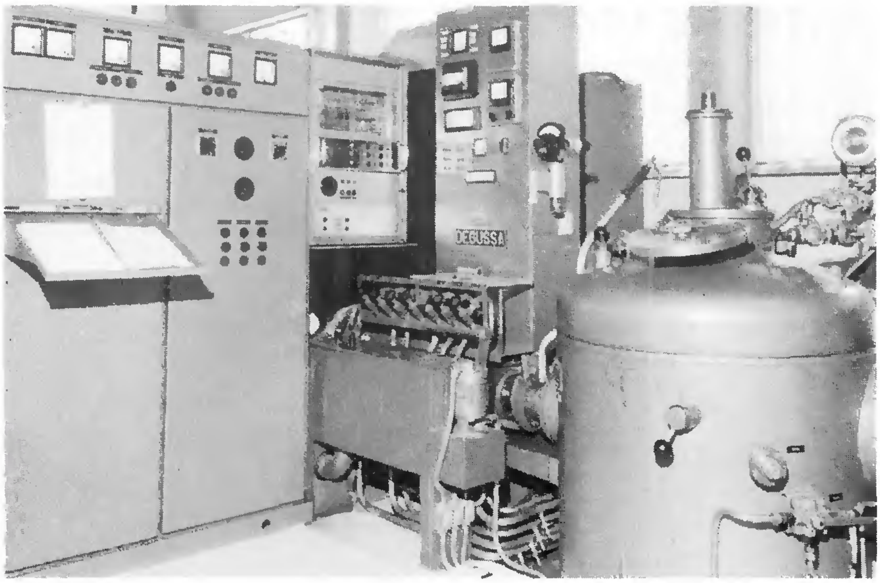


Fig. 5 — Induction high-vacuum furnace

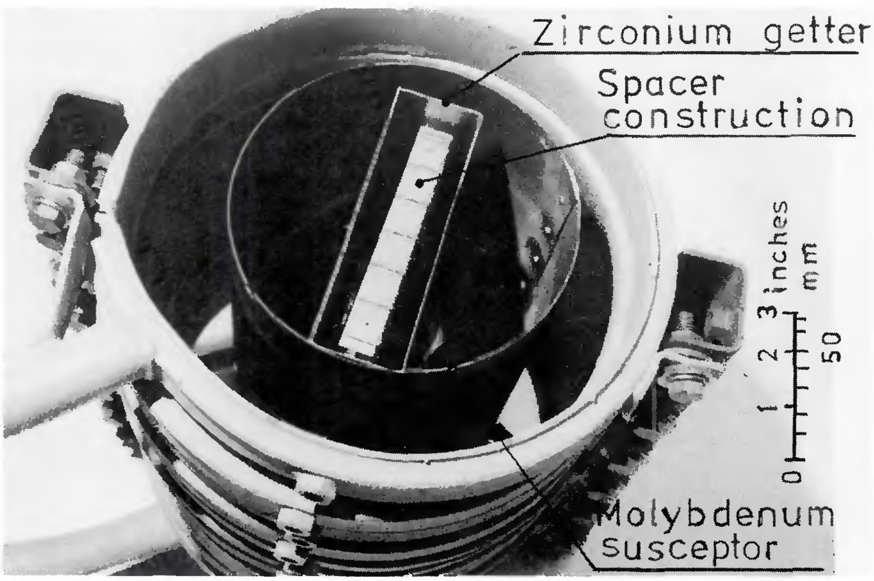


Fig. 6 — Arrangement for getter brazing of the spacer construction shown in the lead photo (title page)

Table 7 — Corrosion Rates in Water at 290 C (550 F)

Material	Corrosion rates mg/dm² per month	μm/year	mils/year
BNi6 brazed with a Zr-getter	8	1.1	0.043
BNi6 brazed on a nickelplate	17	2.3	0.090
BNi7 brazed with a Zr-getter	2.5	0.3	0.013
BNi7 brazed on a nickelplate	2.7	0.4	0.014
Nickel plate (from Watts bath)	7	0.9	0.037
Inconel 718	1	0.1	0.004

which was now furnished with a zirconium tube inside the susceptor. With exactly the same brazing conditions as earlier it was now found that perfect wetting of the BNi7 brazing filler metal on both of the Inconels 718 and X-750 could be obtained. The contact angles were only a few degrees.

Complete spacer constructions were then brazed in the arrangement — Fig. 6 and all of the 49 joints in each spacer showed perfect wetting and filling during their first brazing cycle which was 1050 C (1922 F) for one hour at a vacuum measured to be 10^{-5}mm Hg.

Discussion

From the above results it is evident that zirconium improved the wetting characteristics of these Inconels very drastically. Besides it was also observed that the appearance of the Inconel surfaces had changed as after a bright annealing.

It seems quite natural that zirconium will prevent the Inconel specimen placed inside the zirconium tube from oxidation as zirconium has a higher affinity to oxygen than any of the alloying elements in the Inconels.

On the other hand it is not immediately obvious that zirconium should even be able to reduce the surface oxides of the Inconels considering the relatively low temperatures 1000-1050 C (1832-1922 F) and the not particularly high vacuum 10^{-5} - 10^{-2}mm Hg. However, this must apparently be the case because it has been possible to refute other explanations of this effect such as dissolution of the oxides or a deposition of elements capable of improving the wetting characteristics.

Tables 4 and 5 show that dissolution of the surface oxides did not take place either at 1065 C (1950 F) and 10^{-2}mm Hg or at 1000 C (1822 F) and 10^{-5}mm Hg on account of the lack of any significant wetting under those conditions. Exhaustive microprobe analysis of both the diffusion zone and the surfaces confirmed that there had been no deposition of zirconium or other elements than those already present in the Inconels — Table 1.

It is therefore concluded that zirconium will prevent oxidation of Inconels 718 and X-750 under the conditions described by means of a getter effect, which locally will lower the partial pressure of the oxygen. This getter effect is also supposed to be so strong that the zirconium getter might even be able to reduce the surface oxides already present on the Inconels before the brazing operations.

Corrosion

Since the experiments were carried out for the brazing of spacers for nuclear fuel elements, corrosion tests were necessary. Earlier planned corrosion tests on nickel plated Inconels brazed with BNi6 were therefore extended to include getter brazed specimens.

The corrosion behavior of the two brazing filler metals BNi6 and BNi7 of the base metal Inconel 718, and of nickel plated Inconel 718 was tested in water at 290 C (550 F)

The brazed specimens were circular discs of Inconel 718 with a diameter of 10 mm (0.394 in.) and covered on one side with the brazing filler metal. The nonbrazed specimens (Inconel 718, as received or electrolytically nickel plated) were of the same geometry and had been given a heat treatment identical to that of the brazed specimens — 1025 C (1877 F), 1 h, 10^{-5}torr.

The corrosion testing was performed in a high pressure autoclave and lasted 226 days. The specimens were inspected (visually and by weighing) at intervals of about 30 days. After a small initial weight gain the weight of the specimens decreased linearly with time. The corrosion rates calculated from the weight changes are given in Table 7.

It is seen from the table that both of the brazing filler metals show excellent corrosion resistance in water at 290 C, although neither of them is as resistant as the base metal Inconel 718. It also appears that the chromium containing alloy BNi7 is significantly better (by a factor of three to six) than BNi6. While the brazing method seems to have only a negligible influence on the corrosion resistance of BNi7, the BNi6 specimens brazed with getter show a corrosion rate which is a factor of two lower than that of the BNi6 specimens brazed on a nickel plated surface.

Conclusion

1. Getter brazing of untreated Inconel 718 and X-750 with the brazing filler metals BNi6 and BNi7:

a) The use of a getter, zirconium, reduces the temperatures and/or vacuum requirements necessary. For example the brazing temperature can be lowered from 1065 C (1950 F) to 1000 C (1830 F) in a vacuum of 10^{-5} mm Hg or better , or down to 1025 C (1875 F) in a primary vacuum (10^{-2}mm Hg), and the flowing and wetting characteristics remain the same.

b) The pretreatment is only a degreasing. Time consuming treatments such as nickel plating are avoided.

c) The getter material, zirconium, which must surround the workpiece or the joints to be brazed is easy to form and machine and may even be used as susceptor in induction heating.

2. Corrosion properties in water at 290 C (550 F):

a) Both BNi7 and BNi6 show excellent corrosion resistance. The corrosion rates are 0.3-2.3 μm/year (0.01-0.09 mils/year). However, the chromium alloyed BNi7 is a factor of 3-6 better than the Ni-P alloy BNi6. This may be compared with heat treated heated and unbrazed Inconel 718 which has shown a corrosion rate of less than 0.1 μ m/year (0.004 mils/year).

b) Getter brazed BNi7 shows about the same corrosion rate as BNi7 brazed to nickel plated Inconel 718 — 0.3 and 0.4 μm/year (0.013 and 0.014 mils/year) — whereas getter brazed BNi6 shows a corrosion resistance which is a factor of 2 better than that obtained from BNi6 brazed to nickel plated Inconel 718 — from 1.1 to 2.3 μm/year (from 0.043 to 0.090 mils/year).

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