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High-Temperature Brazing of Stainless Steel with Low-Phosphorus Nickel-Based Filler Metal

Three experimental brazing filler metals are evaluated for suitability in industrial applications

BY E. LUGSCHEIDER AND T. COSACK

ABSTRACT. The demand for boron-free, low-melting, nickel-based filler metals led to the development of three high-temperature brazing alloys with low amounts of phosphorus and iron. The evidence of the industrial suitability of the proposed new brazing alloys was made with stainless steel AISI 321 using a combined test method. The results of the metallographic investigation were confirmed by tensile strength tests. The tests established optimum operating parameters such as brazing temperature, time and joint clearance.

Introduction

Nickel-based filler metals are desirable because of their alloying elements and simple method of production. Extensive composition possibilities lend these filler metals to a wide spectrum of applications. In addition to applications in specialized industries, the use of high-temperature brazing with nickel-based filler metals is increasing in conventional areas.

By using nickel-based brazing alloys, hard phases, which decrease tensile strength, are formed in the center of the braze where the maximum joint clearance is exceeded. This can be seen in metallographic investigations. The maximum joint clearances, depending on the brazing parameters, were determined by using a specially developed metallographic specimen. It has a linearly increasing joint clearance. Results of metallographic investigations are summarized in the figures. The investigations have been made with a computer-controlled micrograph analysis system, which has graphics. In the end, the results of the metallographic investigations were verified by tensile strength tests of a brazed butt joint tensile specimen.

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KEY WORDS

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Low Phosphorus
Nickel Base
Brazing Filler Metal
Max. Joint Clearance
Wedge Specimen
Investigation Method
Combined Methods
Braze Formation

Experimental Work

Brazing Alloys

The three silicide-phosphide high-temperature brazing filler metals were developed by modifying ternary Ni-Cr-Si alloys with phosphorus and iron.

Alloy 1 was developed by the addition of 0.5% phosphorus to the alloy 20.4Cr-11.6Si-bal Ni, which is almost the ternary eutectic point of the Ni-Cr-Si system. Hence, a melting range was reached between 1044° and 1060°C (1911° and 1940°F).

Alloy 2 was developed by alloying 3% phosphorus to the ternary alloy 27.1Cr-5.2Si-bal Ni, producing an alloy with a

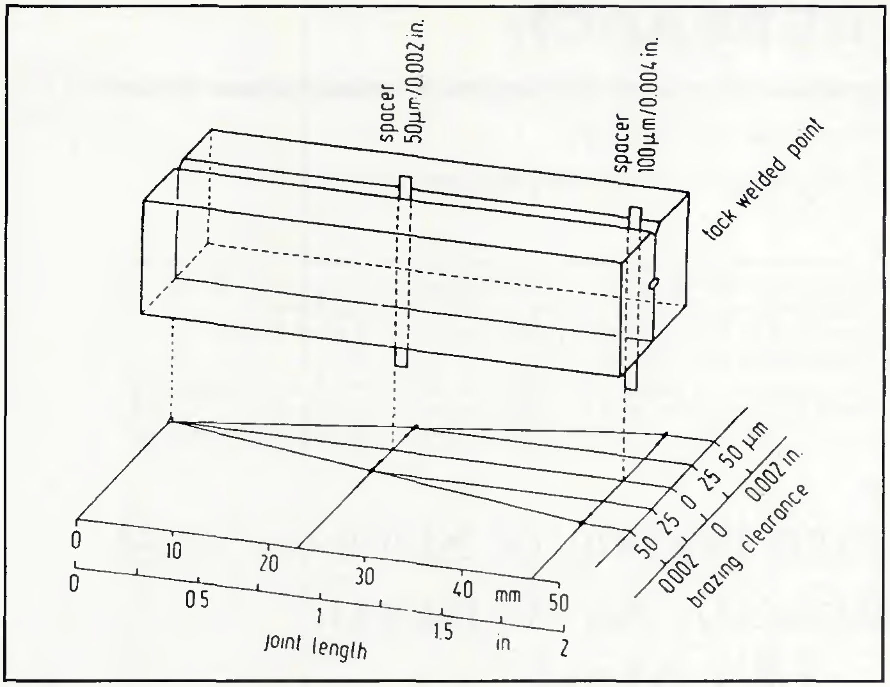

Fig. 1 — Wedge specimen used to vary the joint clearance

melting range of 952° to 1155°C (1746° to 2111°F). Alloy 2 has a high chromium content, which can be seen in the wide melting range and the high liquidus temperature.

Brazing Alloy 3 was developed from the ternary alloy 15.8Cr-8.5Si-bal Ni by alloying it with 3% phosphorus and 3% iron. It has a solidus of 996°C (1825°F) and a liquidus of 1058°C (1936°F).

At the beginning of testing, special key alloys were used. The commercial brazing alloy BNi-7 was the phosphorus supplier, the BNi-5 alloy and a nickel-silicon alloy were the silicon suppliers, and the rest was pure nickel and chromium powder. The alloys were made molten in a vacuum induction furnace and then mechanically pulverized. After production, the brazing filler metals were chemically analyzed and the melting range was investigated by differential thermo analysis (DTA). Only an insignificant deviation from the nominal composition occurred, and the melting range composition is shown in Table 1.

With SEM investigation, Ni solid-solution silicides and phosphides were discovered in the three brazing alloys. The undesirable hard phases, such as the silicides and phosphides, were evident only in a low-volume amount.

Test Method

It was shown that the brazing joint clearance depends on the development of hard phases in the center of the braze. Therefore, a special specimen was developed. The wedge specimen is composed of two joining halves, tacked together by GTAW after inserting spacers to make different joint clearances.

Figure 1 shows the structure of the wedge specimen with 50-µm and 100-µm spacers and the geometry of the joint clearance. The filler metal powder is placed in the V-groove, which then fills the joint through capillary flow during the brazing process.

Table 1—The Investigated Nickel-Based Filler Metals

Brazing Filler Metal	Nominal Composition wt-%	Melting Range °C (°F) T_{Sol}	Melting Range °C (°F) $T_{Liqu.}$
Alloy 1	20.3Cr-11.5Si-0.5P-bal Ni	1044 (1911)	1060 (1940)
Alloy 2	26.3Cr-5.1Si-3.0P-bal Ni	952 (1746)	1155 (2111)
Alloy 3	14.8Cr-8.0Si-3.0P-3.0Fe-bal Ni	996 (1825)	1058 (1936)

For metallographic preparation, the braze is sectioned. For the investigation in a computer-controlled micrograph analysis system, the wedge specimen is etched and surfaced by vapor-deposition. The computer-controlled micrograph analysis system is comprised of a microscope, TV camera, display monitor, digitizer table and computer. The brazing clearance and joint geometry are registered by measuring the wedge specimen. All measuring data are stored on discs in the format of tables. They are available whenever needed. A graphic working program takes the measuring data and draws a wedge diagram on the printer. The diagram gives the geometric structure of the wedge specimen with all its characteristics. A typical characteristic with the application of nickel-based filler metal is the dependence of the tensile strength on the amount of hard phases in the center of the braze joint, which in turn, depends on the joint clearance. With the beginning of the hard phase stabilization, the maximum brazing clearance (MBC) is defined. Below MBC, the micrograph of the braze joint displayed only Ni-solid-solution, indicating good tensile strength qualities. The MBC is indicated during the measurement of the wedge specimen. This information is used by the computer, and the maximum brazing clearances of every wedge specimen are drawn in accordance with the brazing temperature and brazing time.

The test method is a combined method, and the results of the metallographic investigations are compared with tensile strength tests. Brazed butt joint tensile specimens are prepared by using a fixed brazing temperature and holding time, but with different joint clearances.

Figure 2 shows the specimen used for tensile strength tests. The specimen consists of two halves with the brazing joint in the middle. The various joint clearances are made by spacers, which are removed after the brazing process. With a clearance below the maximum brazing clearance, the tensile strength of the

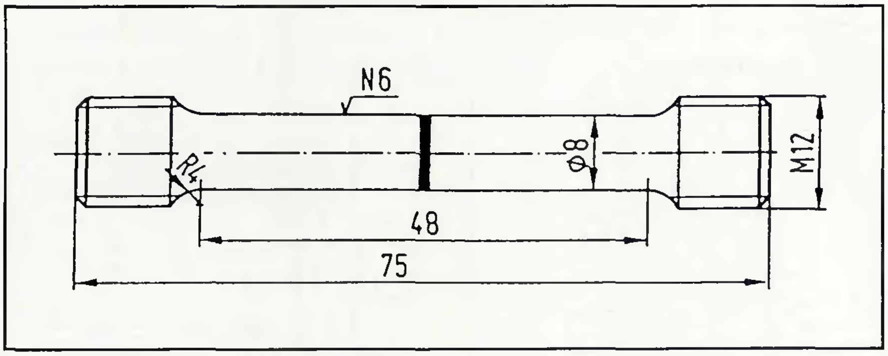

Fig. 2 — Brazed butt joint tensile specimen

Fig. 3 — Microstructure of a joint brazed with Alloy 1 filler metal. The base metal is 321 stainless steel, brazed at a cycle of 1070°C/60 min, with a joint clearance of 16 μm

Fig. 4 — Microstructure of joint brazed with Alloy 1 filler metal. The base metal is 321 stainless steel, brazed at a cycle of 1150°C/10 min, with a joint clearance of 20 μm

braze equals the tensile strength of the base metal. With a clearance above the maximum joint clearance, the tensile strength reaches only 20 to 50% of that of the base metal.

Braze Formation

For the brazing filler metals Alloy 1, Alloy 2 and Alloy 3, the wedge specimens and the tensile test rods were brazed at temperatures and times shown in Table 2.

All the specimens were stainless steel AISI 321 brazed in a vacuum furnace with a pressure less than 10^{-3} mbar (10^{-1} Pa).

Table 2—The Brazing Cycles for the Three Investigated Brazing Filler Metals

Brazing Cycle	Brazing Filler Metal Alloy 1	Alloy 2	Alloy 3
1040 °C (1904 °F)/10 min			•
/60 min			•
1070 °C (1958 °F)/10 min	•		•
/60 min	•		•
1090 °C (1994 °F)/10 min	•	•	•
/60 min	•	•	•
1120 °C (2048 °F)/10 min	•	•	•
/60 min	•	•	•
1150 °C (2102 °F)/10 min	•	•	
/60 min	•	•	
1190 °C (2174 °F)/10 min		•	
/60 min		•	

During the brazing process, the molten filler metal fills the clearance according to capillary force. This capillary force entails an interaction between the base metal and the filler metal. At first, there are heterogeneous chemical reactions at the solid-liquid interface, whereby atoms accumulate at the interface of the solid and liquid metal. This process is known as wetting. Secondly, there is a concentrated equilibrium behind a thin limit zone. The new brazing alloy reacts with the base metal elements such as iron, chromium and nickel. After the brazing process is completed, there is a chemical composition in the braze different from the filler metal. A micrograph indicates a solid solution in the base metal in which silicon and phosphorus are dissolved. The solid solutions grow on concentric circles from the base metal to the brazing filler metal, meeting each other in the center of the braze.

It becomes a braze without hard phases. At wide joint clearances, the diffusion distances are already too big. The hard phases formed cannot completely dissolve in solid solutions. Hard phases, such as silicides and phosphides, form in the center of the braze, reducing tensile strength.

Especially in solid solutions, iron amounts of up to 40% could be analyzed by SEM, although before the brazing cycle, iron was not in the filler metal. A base metal solid solution can also be seen with SEM analysis, but the difference between the braze, which is influenced by molten filler metal and the brazing joint clearance, is very big. This phenomenon is known as erosion. This material change is caused by material transport mechanisms between solid and liquid metals and by solid matter diffusion.

The joint in Fig. 3 was brazed with the filler metal from Alloy 1, at a temperature of 1070°C (1958°F) and a holding time of 60 min. The grain growth in the brazing joint can be seen and is contrasted with the microstructure of the base metal.

The microstructure of the solid solution contains 41.8% nickel, 34.1% iron, 19.1% chromium and 5.1% silicon. The silicon amount was below the solution limit, so the formation of a complete solid solution was possible. The phosphorus content was below the detection limit.

Results of the Metallographic Investigations and the Tensile Strength Tests

AISI 321 stainless steel joints brazed with Alloy 1 were investigated at brazing temperatures between 1070° and 1150°C (1958° and 2102°F) with brazing times of 10 and 60 min. The selection of brazing temperatures was made according to the results of DTA.

Figure 4 shows a nickel solid solution micrograph without hard phases at a joint clearance of 20 μm. The grains grow into the brazing filler metal from the base metal. But the difference between base metal and filler metal is enormous with this specimen brazed at 1150°C and having a holding time of 10 min — Fig. 5. With an additional diffusion heat treatment, base metal assimilation can be expected.

The braze with a joint clearance of 93 μm displays one-third hard phase in the center of the joint. It can be clearly seen that the solid solutions are growing on concentric circles from the base metal into the filler metal. The different phases in the middle of the joint are nickel solid solutions, nickel-chromium silicides and nickel silicides. The grain boundary growth in solid solution has its origin at the grain boundary of the base metal. Already, the diffusion distances for a complete solubility of the hard phases formed with silicon were too great at these brazing parameters.

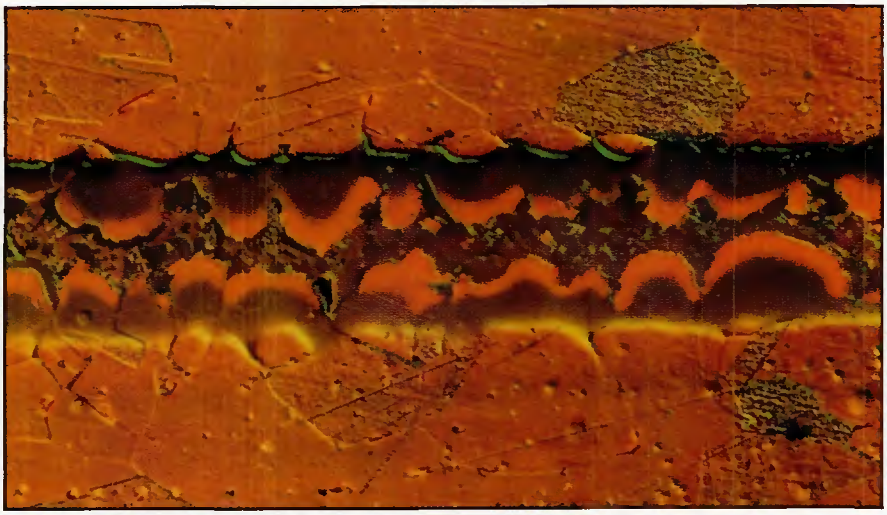

Fig. 5 — Microstructure of a joint brazed with Alloy 1 filler metal. The base metal is 321 stainless steel, brazed at a cycle of 1150°C/10 min, with a joint clearance of 93 µm

For all the brazed specimens, diagrams were drawn by the computer system schematically showing geometry of the wedge specimens.

The wedge specimen diagram (Fig. 6) shows a MBC of 29 µm with the given brazing parameters. The wide beam in the diagram illustrates the beginning and the extent of hard phases. The straight line pairs marked by crosses and stars show the variation in joint clearance. The base metal is not influenced by trans and intercrystalline precipitations as with boron-containing filler metals. The wedge specimen diagram shown is a typical example for all the wedge specimen diagrams, produced by the computer after analyzing data available. All wedge specimen diagrams with the MBC marked appear as MBC's in the filler metal-base metal characteristic diagrams.

Figure 7 shows the filler metal-base metal characteristic of the AISI 321 stainless steel joints brazed with Alloy 1. For the brazing time of 10 min, the MBC's appear almost temperature independent between 22 and 35 µm. At the brazing time of 60 min, MBC's were established between 35 and 42 µm. The filler metal-base metal characteristic data offer a basis for the estimation of manufacturing parameters for brazing temperature, time and joint clearance.

Results found by metallographic investigations are comparable to the tensile strength tests conducted on brazed butt joint specimens.

At joint clearances below the MBC, which is marked as a tolerance area, tensile strengths near the base metal tensile strength were reached. The microstructure of the brazing filler metal was a nickel solid solution with good tensile strength characteristics. The fracture area ran through the filler metal and the base metal with shearing lips. At the brazing clearance of 12 microns, the fracture area in the base metal was separate from the filler metal. If the joint clearance value is above the MBC, the tensile strength decreases in proportion to the increase of hard phases in the center of the filler metal. The base metal tensile strength for 321 stainless steel is shown in Fig. 8.

Figure 9 shows a specimen in which the tensile test was interrupted at 565 N/mm^2, and an untested specimen as a comparison. The elongation reached nearly 30%, with very little shrinkage.

Brazing tests were conducted with Alloy 2 filler metal on 321 stainless steel at temperatures between 1090° and 1190°C (1994° and 2174°F), with holding times of 10 and 60 min — Fig. 10.

The brazing joint with a clearance of 44 microns shows no continuing hard phases and possesses solid solution grains, a condition which extends throughout the whole braze. The fine branchings are remarkable. Through SEM investigations, they proved to be phosphide grain boundary precipitations. The precipitations show a high chromium content.

With the investigation of the maximum brazing clearance, only the hard phases in the center of the filler metal were considered. The phosphide grain boundary precipitations of finely shaped branchings could not be detected.

All investigated MBC's for the Alloy 2 system are shown in Fig. 11. They range from 5 to 27 µm for the holding time of 10 min and from 30 to 42 µm for 60 min.

At these ranges, the necessity of the combined test method is especially clear.

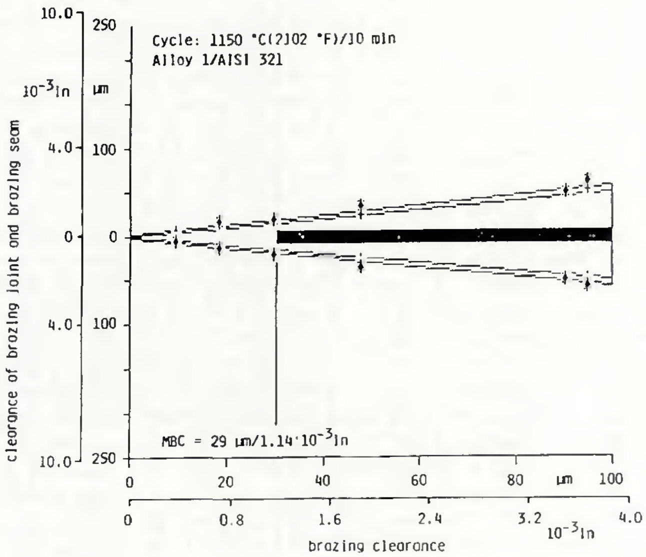

Fig. 6 — Wedge specimen diagram for Alloy 1 filler metal, brazed at a cycle of 1150°C/10 min

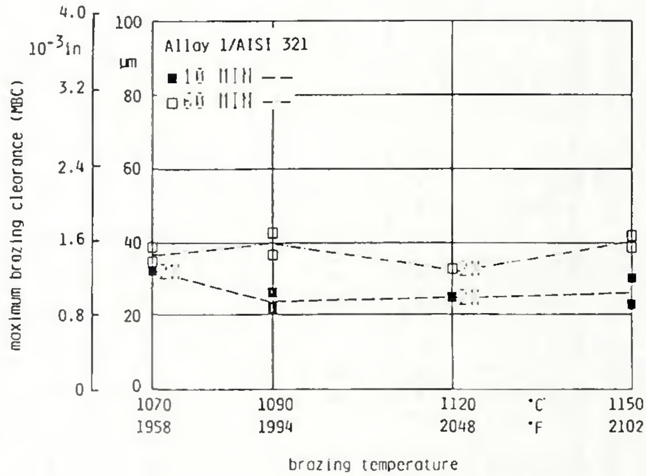

Fig. 7 — Alloy 1 filler metal and 321 stainless steel base metal characteristic diagram

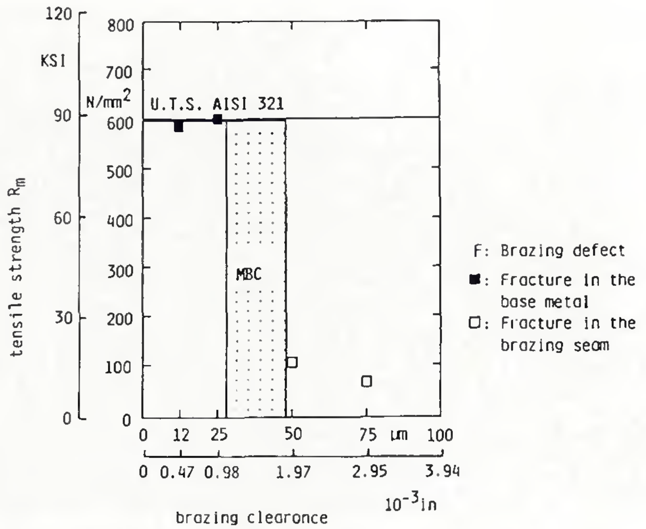

Fig. 8 — Tensile strength of brazed butt joint tensile specimens. Alloy 1/321 stainless steel, 1090°C/60 min

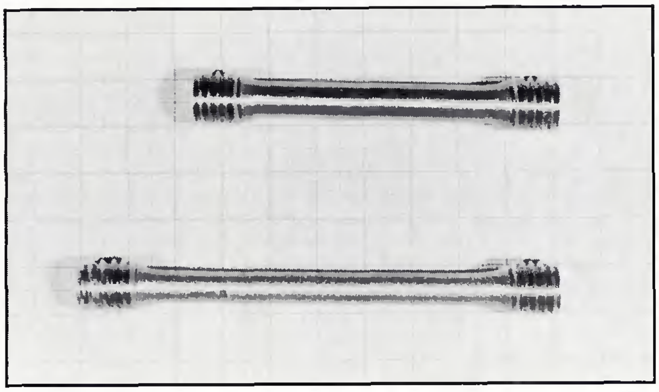

Fig. 9 — An untested tensile specimen compared with one that displays a 30% elongation, but very little reduction of area

Tensile strengths were reached at 400 N/mm² (58 ksi) with joint clearances below the maximum joint clearance in the diagram.

The diagram presented in Fig. 12 shows that there is no conformity between the metallographic investigations and tensile strength tests. Obviously, the phosphorus amount of 3.0% is too high for this brazing alloy. With this application, phosphide precipitations collect mostly on the grain boundary, a condition which can only be identified by SEM investigation.

Figure 13 shows the irregularly distributed phosphide precipitations.

According to its melting characteristics, the lowest brazing temperature was expected from Alloy 3. Brazing temperatures between 1040° and 1120°C (1904° and 2048°F) were selected, along with brazing times of 10 and 60 min. Fine

Fig. 10 — Microstructure of a joint braze with Alloy 2 filler metal. The base metal is 321 stainless steel, brazed at a cycle of 1190°C/60 min, with a joint clearance of 44 µm

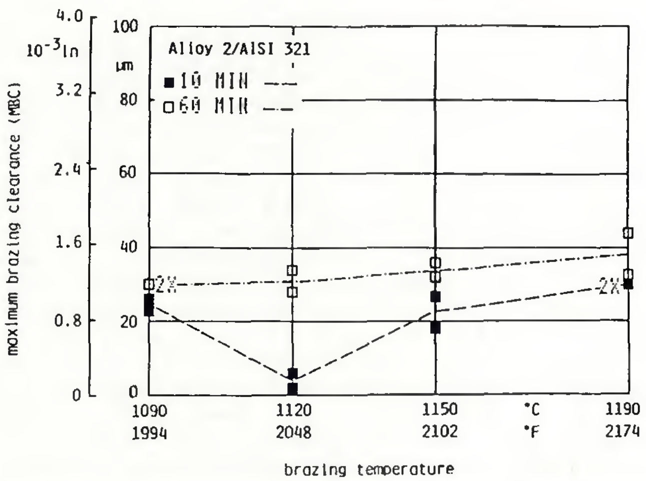

Fig. 11 — Alloy 2 filler metal and 321 stainless steel base metal characteristic diagram

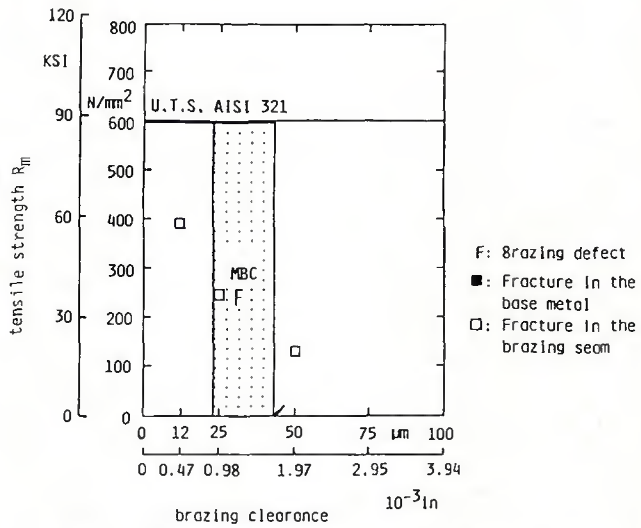

Fig. 12 — Tensile strength of 321 stainless steel butt joints brazed with Alloy 2 at a cycle of 1150°C/60 min

Fig. 13 — Microstructure of phosphide precipitations

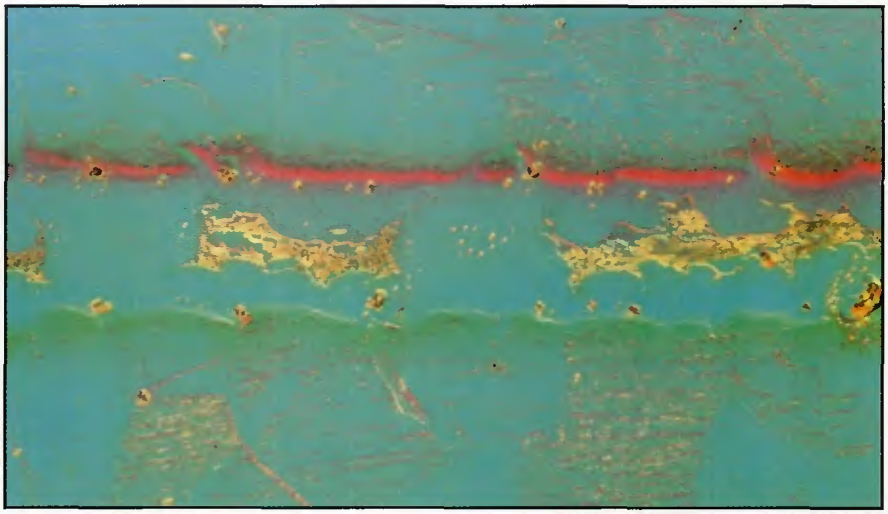

Fig. 14 — Microstructure of joint brazed with Alloy 3 filler metal. The base metal is 321 stainless steel, brazed at a cycle of 1090°C/60 min, with a joint clearance of 40 μm

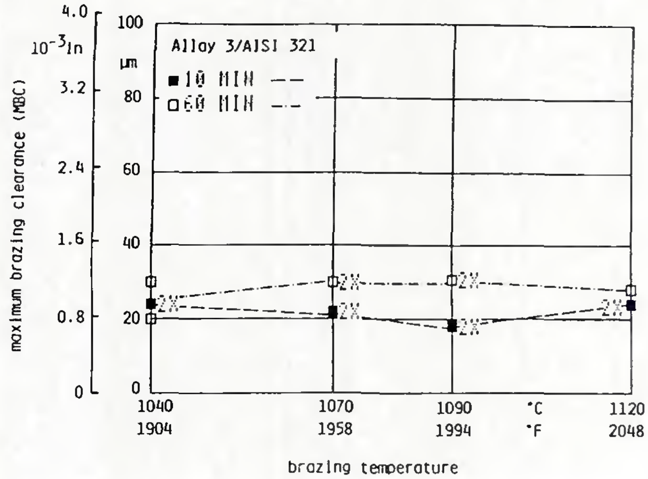

Fig. 15 — Alloy 3 filler metal and 321 stainless steel base metal characteristic diagram

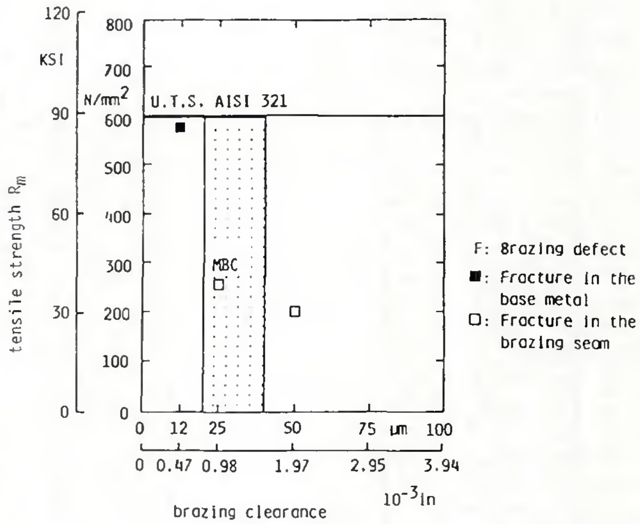

Fig. 16 — Tensile strength of 321 stainless steel butt joints brazed with Alloy 3 filler metal at a cycle of 1090°C/60 min

branchings are in the areas of the solid solution without hard phases.

As seen in Fig. 14, solid solution bridges are present throughout the braze. An intermediate area of hard phases exists, decreasing tensile strengths.

The results of metallographic investigations show that joint clearances have to be adjusted between 20 and 30 μm (see Fig. 15) to produce brazements without hard phases. Tensile strength tests show that acceptable tensile strengths can be reached at brazing temperatures above 1070°C. Indeed, all tensile strength specimens brazed at 1040°C have discontinuities, which can be traced to poor flow and wetting of the brazing alloy. Furthermore, satisfactory results were attained at brazing temperatures above 1090°C.

The tensile strength diagram in Fig. 16 shows that tensile strengths of the filler metal almost reach the strength of the base metal at joint clearances below the MBC.

Conclusion

The demand for boron-free nickel-based filler metals led to the development of silicide-phosphide brazing alloys, of which the suitability for industrial applications was so far unknown.

Using a combined test method, the interrelationship between joint clearance and hard phase formation was determined by metallographic investigations and tensile strength tests. The practical possibilities can be appraised under application conditions. The data from the tests show that only through the use of the two combined investigation methods is it possible to come to a final conclusion about the suitability of a filler metal-base metal combination.

Alloy 1 filler metal showed the best match between metallographic investigation and tensile strength test. With brazed butt joint tensile specimens, the filler metal reached the base metal tensile strengths at a brazing temperature of 1070°C and a brazing time of 10 min. This particular filler metal might be used in a similar manner as the commercial filler metal BNi-5, where an 80°C (144°F) lower brazing temperature is possible.

Alloy 2 filler metal is not suitable for industrial applications. At tight joint clearances and with brazing temperatures of 1190°C, satisfactory tensile strengths could not be reached. This brazing alloy is not an acceptable alternative to commercial filler metals or the other two experimental filler metals.

Alloy 3 filler metal can be used at brazing temperatures above 1070°C.

High tensile strengths can be reached at temperatures of 1090°C, making it a satisfactory alternative for industrial use. Minimum brazing temperature of 1090°C is about 60°C (108°F) lower than the brazing temperature of BNi-5.

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