Metallography of Vacuum Brazed and Vacuum Heat Treated and Gas Quenched Materials

All of the materials studied—low alloy steel, aluminum, and stainless steel—had comparable properties and microstructures when conventionally heat treated and quenched and when heat treated in vacuum and quenched by gas

BY JAMES L. McCALL

ABSTRACT. This paper describes the results of metallographic studies that have been made on several materials included in a program directed toward developing a vacuum brazing/heat treating, gas quenching (VBGQ) process. A previous program[1] showed that the process was feasible for joining complex shapes of 6061 aluminum and indicated that it may be useful for other materials as well. Because of this, a program was initiated to develop the process for joining various aluminum alloys, low alloy steels, and corrosion resistant steel alloys. This program included mechanical property evaluations of specimens of these materials that were heat treated by processes intended to simulate those that would be encountered during the VBGQ process.

Mechanical properties of these specimens were compared with those obtained from conventionally heat treated specimens. Also, several of the materials were brazed by the process and the quality of the brazed joints were evaluated. Much of this work is described in a previous paper.[2] The present paper primarily is limited to descriptions of the results of rather detailed metallographic studies that were made of specimens of both types.

Materials Heat Treated

Specimens of three low alloy steels (4130, 4340 and 18Ni250 maraging steel), three aluminum alloys (2014, 2219 and 6061), and two corrosion resistant alloys (type 316 and type 321 stainless steel) which had been heat treated to simulate the conditions that would be experienced during the process were studied. These were all sheet material, but various gauge thicknesses were included in the studies to permit an evaluation of thermal response effects due to thickness variations. Also, the effects of different quenching gases were studied. The quenching gases included argon, carbon dioxide, helium, nitrogen and liquid nitrogen. Metallographic studies of the materials are described individually below.

2014 Aluminum

2014 aluminum is a copper, magnesium, silicon alloy normally heat treated by solution treating at 935 ±10F for 40 minutes, water-quenching and aging 18 hours at 320F. Specimens of material in this condition were compared to specimens given the same solution treating and aging conditions but with the intermediate quench accomplished by argon, liquid nitrogen or helium. For this material, as well as for all the materials studied, the data presented will be limited to a maximum of three quenching gases because of the similar results obtained from them.

The mechanical properties obtained from tensile tests are shown in Table 1. Three thicknesses were investigated: 0.125, 0.063 and 0.025 in. However, since the data from the various thicknesses were generally the same, the data for only one thickness, 0.125 in., are included in the table. Also included are quenching rates measured for the various gases from cooling time curves.

Metallographic examinations of the 2014 material are summarized in Fig. 1. From the photomicrographs contained in this figure the structures of all the gas quenched samples are shown to be nearly identical to that of the water quenched sample, except for the helium quenched sample which had more distinct grain contrast. Since

Table 1—Mechanical Properties of Aluminum Alloys Vacuum Heat Treated and Gas Quenched

Aluminum alloy	Thickness in.	Quenching medium	Quenching time from 935F to RT sec	Ultimate tensile strength ksi	Yield strength ksi	Elongation %
2014	0.125	Water	—	73.4	65.8	14.7
	0.125	Argon	17.5	67.2	59.2	9.7
	0.125	Liquid nitrogen	40.0	62.9	52.8	11.7
	0.125	Helium	11.2	74.3	67.9	10.7
2219	0.125	Water	—	61.3	42.0	13.0
	0.125	Argon	13.7	57.9	39.0	12.0
	0.125	Liquid nitrogen	45.5	50.4	32.5	11.0
	0.125	Helium	8.7	59.3	40.6	10.7
6061	0.125	Water	—	45.8	39.1	17.0
	0.125	Argon	11.2	45.7	38.3	14.0
	0.125	Liquid nitrogen	65.0	43.1	34.0	15.3
	0.125	Helium	8.7	45.5	38.1	16.7

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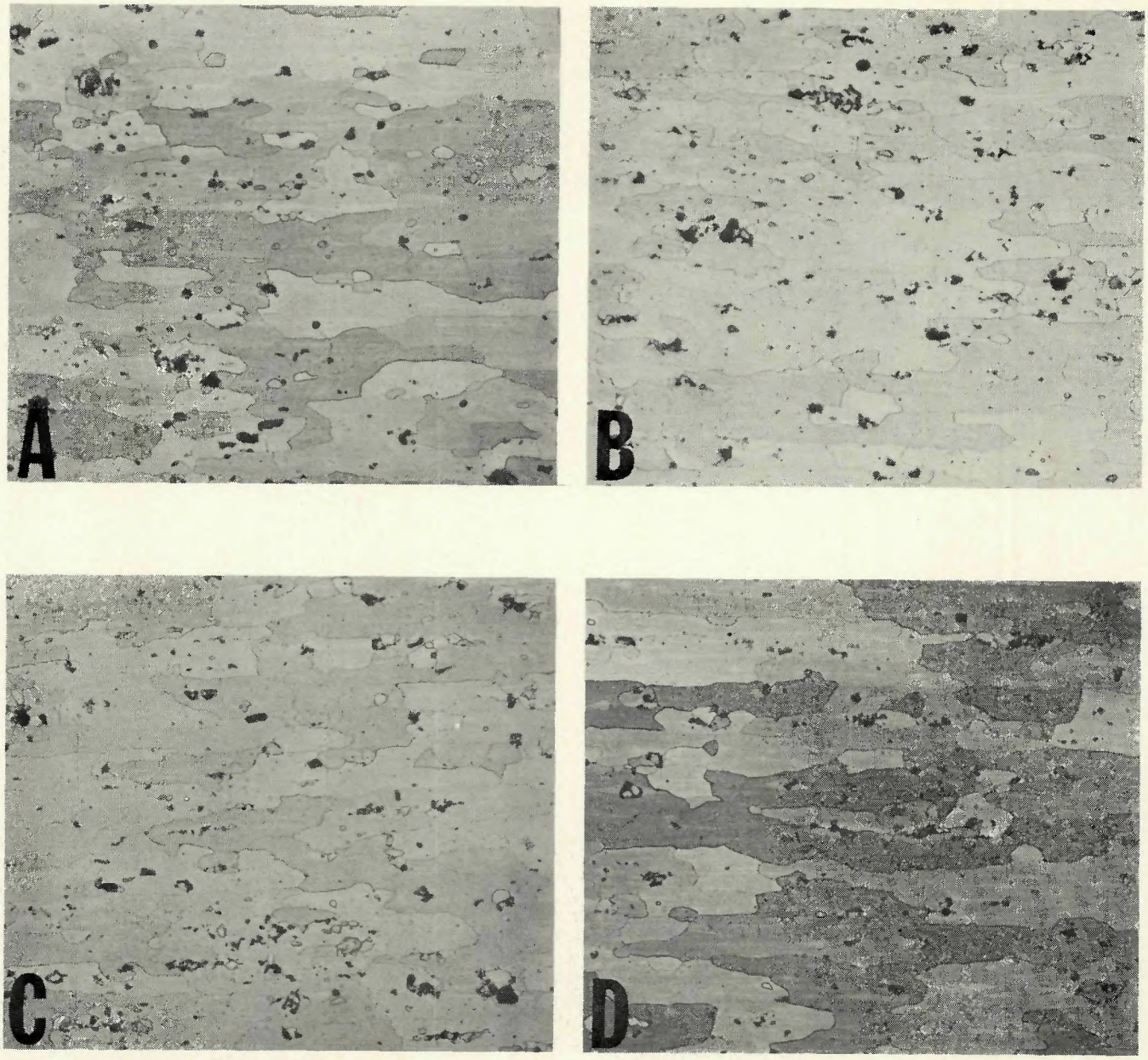


Fig. 1—Microstructures of 2014 aluminum alloy solution treated at 935 ± 10F for 40 minutes, water or gas quenched and aged 18 hours at 320F. (a) water quenched; (b) argon gas quenched; (c) liquid nitrogen quenched; and (d) helium gas quenched. Mag: 400X

all the samples were aged identically, and since the helium was found to be the most rapid quenching medium (Table 1), the grain contrast must be an effect of the quenching rate. The precipitate particles in the grains and in the grain boundaries are $CuAl_2$.

2219 Aluminum

2219 aluminum is an aluminum-copper alloy which is normally hardened by solution treating at 995 ±10F for 10 minutes, water quenching and artificially aging at 375F for 36 hours. For the present studies, however, a 46-hour aging time was used. This condition was compared with specimens exposed to the same solution treating and aging conditions but the intermediate quench was accomplished by argon, liquid nitrogen or helium. Three thicknesses were investigated: 0.125, 0.050 and 0.020 in. Table 1 contains mechanical property data for these samples as well as quenching time data for the three gases as determined from cooling time curves.

Figure 2 contains photomicrographs of the microstructure of these samples. Because of the longer than normal aging time (46 hours versus 36 hours), the microstructures of all the conditions contain a rather heavy precipitate structure and indistinct grain boundaries. The amount of precipitate in the microstructures of the specimens quenched using argon and helium were similar to the water quenched sample, whereas the liquid nitrogen quenched sample had a much greater amount of precipitate. This was confirmed by electron microscopy. The greater amount of precipitate undoubtedly is accounted for by the slower quenching rate of the liquid nitrogen (Table 1).

6061 Aluminum

6061 aluminum is a magnesium, silicon, copper, chromium alloy which normally is hardened by solution treating at 985 ±10F, water quenched and artificially aged at 320F for 18 hours. Specimens in this condition were compared with specimens exposed to the same solution treating and aging conditions but the intermediate quench was accomplished using

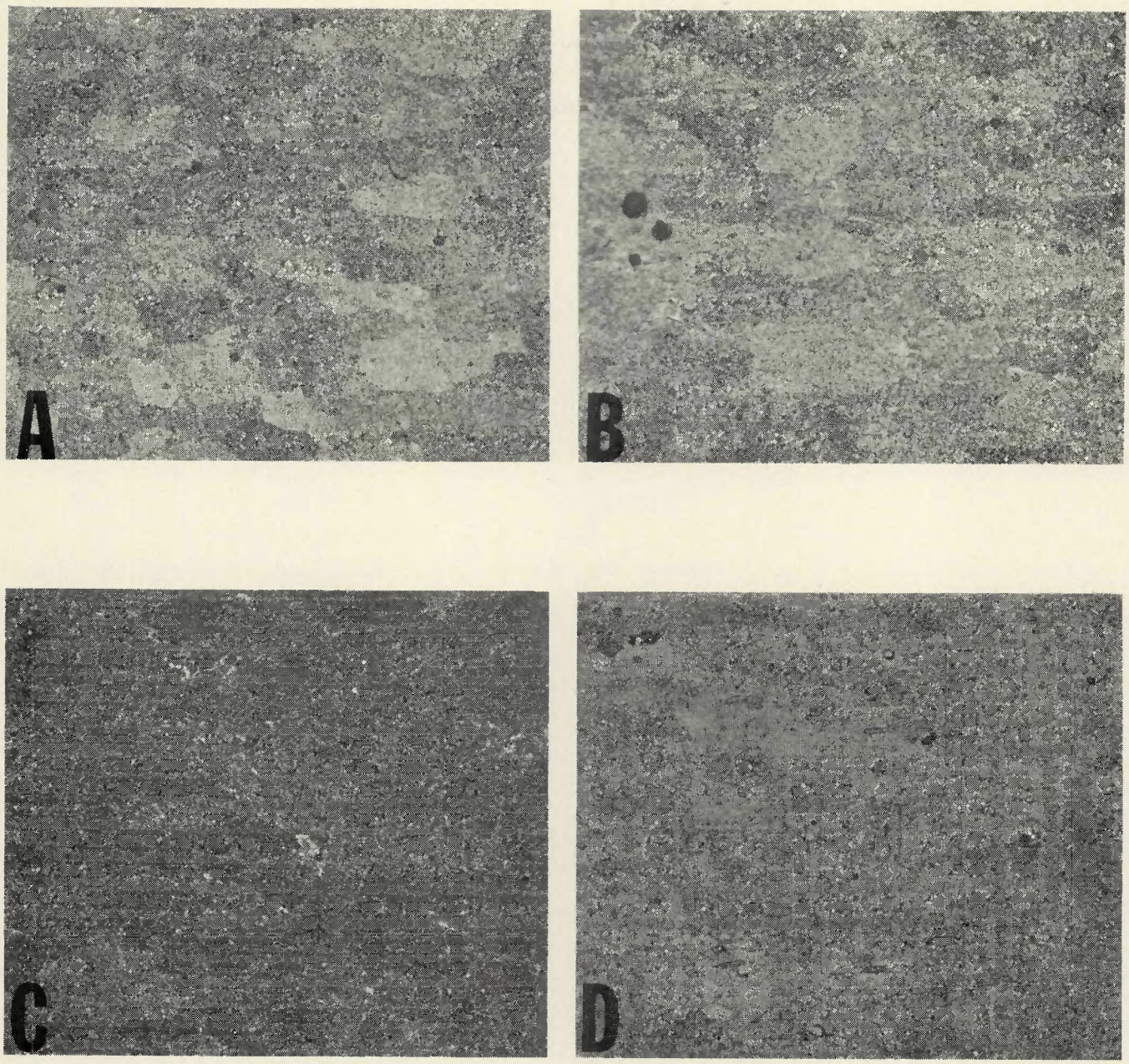


Fig. 2—Microstructures of 2219 aluminum alloy solution treated at 995 ± 10F for 10 minutes, water or gas quenched and aged 46 hours at 375F. (a) water quenched; (b) argon gas quenched; (c) liquid nitrogen quenched; and (d) helium gas quenched. Mag: 400X

argon, liquid nitrogen or helium. Three thicknesses were investigated: 0.125, 0.063, and 0.032 in. Again, since the data did not differ significantly among the thicknesses, results for only the 0.125-in. thickness is included in this paper.

The mechanical properties of the various conditions are contained in Table 1. The properties of the gas quenched specimens are comparable to those for the water quenched material.

Figure 3 contains photomicrographs of the microstructures of these samples. The undissolved precipitate in the microstructures is Mg_2Si. As can be seen, all the microstructures are essentially identical.

4130 Steel

The 4130 alloy is a low alloy steel containing 0.27 to 0.33% carbon and having chromium and molybdenum as principal alloying elements. It is generally hardened by austenitizing at 1575F for 15 minutes, oil quenched and tempered. Tempering for 1 hour at 1050F yields a strength of about 150,000 psi. This condition was compared to samples given the same austenitizing and tempering treatments but gas quenched using nitrogen. Only one quenching gas was investigated for all the steel materials.

As with the aluminum alloys, three thicknesses (0.125, 0.070, and 0.040 in.) were evaluated but since the data did not differ much among the thicknesses, only the 0.125-in. material is considered in this paper.

To obtain a fully martensitic structure, 4130 steel must be quenched fast enough from the austenitizing temperature to below the temperature where the 'nose' of the transformation curve from the T-T-T diagram occurs so that no transformation products are formed. This requires cooling to below about 1000F in less than about 1.5 seconds.[3] The time for nitrogen gas to quench 4130 steel to 1000F was found to be 7 seconds which means that the resultant structure should be a mixture of martensite and bainite. This was the case as shown in Fig. 4. However, the microstructure of the oil quenched specimen also

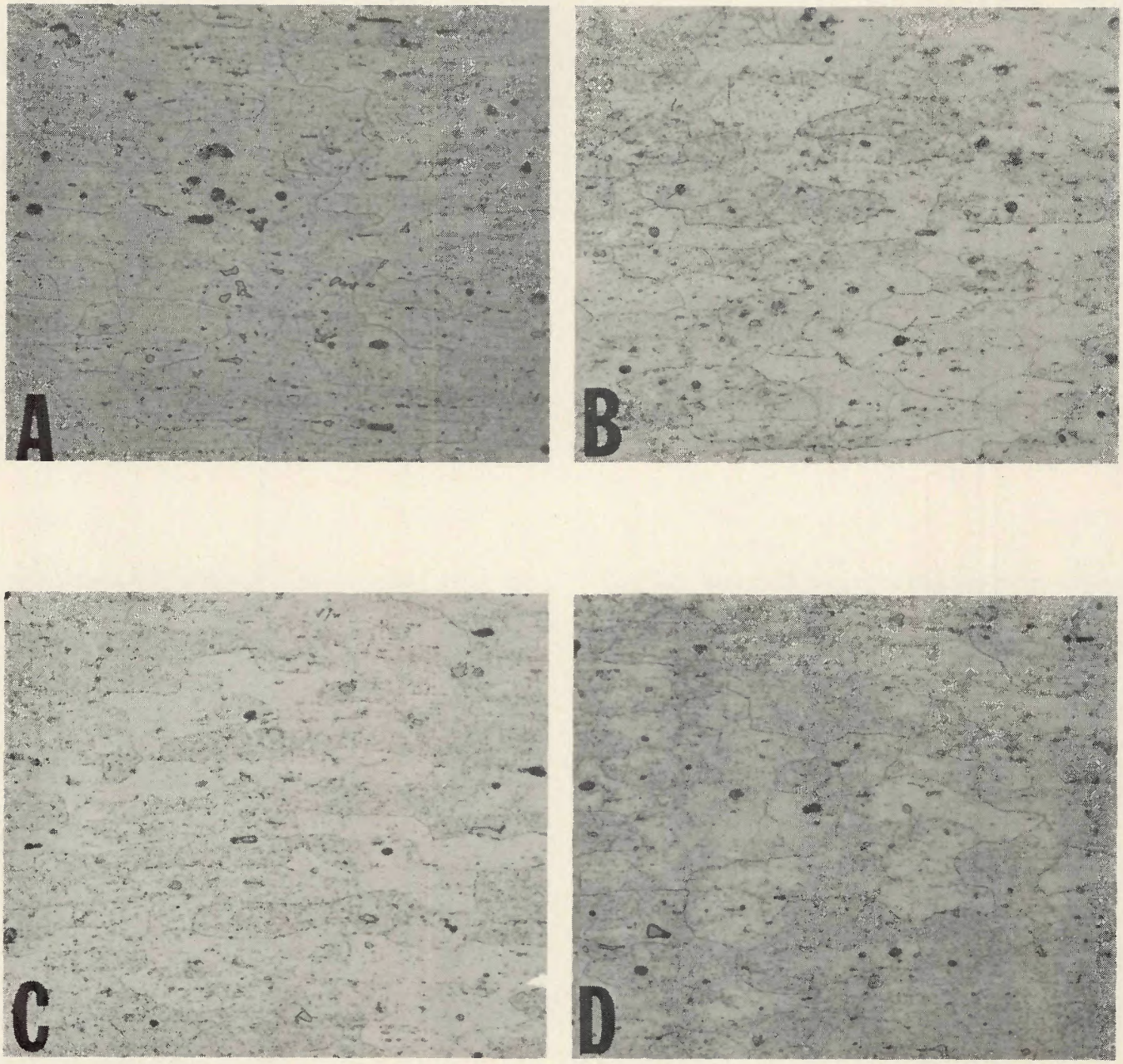


Fig. 3—Microstructures of 6061 aluminum alloy solution treated at 985 ± 10F, water or gas quenched and aged 18 hours at 320F. (a) water quenched; (b) argon gas quenched; (3) liquid nitrogen quenched; and (d) helium gas quenched. Mag: 400X

contained a mixture of martensite and bainite as shown in Fig. 4. Electron microscopic studies of the structures revealed both conditions to be essentially identical. The mechanical properties of the samples are shown in Table 2. As can be seen, the mechanical properties of the nitrogen quenched material actually exceed those of the oil quenched material.

Table 2—Mechanical Properties of Low Alloy Steels

Alloy	Thickness in.	Quenching medium	Ultimate tensile strength ksi	Yield strength ksi	Elongation %
4130 steel	0.125	Oil	156.4	146.0	14.0
	0.125	Nitrogen	162.0	147.0	13.0
4340 steel	0.125	Oil	187.0	173.0	9.0
	0.125	Nitrogen	186.0	172.4	8.5
18Ni250 maraging steel	0.125	Air cooled	268.4	260.7	7.0
	0.125	Nitrogen	267.1	255.0	8.0

4340 Steel

The 4340 material is a low alloy steel containing 0.38 to 0.43% carbon and principal alloying elements of chromium, nickel and molybdenum. It is generally hardened by austenitizing at 1500F for 30 minutes, quenching in oil and tempering. A temper at 900F for 1 hour produces a strength of about 180,000 psi. This condition was compared with samples given the same austenitizing and tempering treatments but gas quenched with nitrogen. As with 4130 steel, 4340 steel must be quenched rapidly past the 'nose' of the transformation curve to obtain a fully martensitic material. The nose occurs at about 800F and the allowable time to reach this temperature is about 10 seconds.[3] A time of about 7 seconds was measured for

the nitrogen gas to quench the 4340 samples to 800F so a fully hardened martensitic structure would be expected. This was obtained as shown in Fig. 5. A fully martensitic structure also was obtained in the oil quenched sample. The structure of the martensite in the nitrogen quenched sample was found from electron microscopic studies to be slightly finer than that in the oil quenched samples, which suggests that gas quenching is more rapid.

Mechanical properties of these materials are shown in Table 2. As can be seen, similar properties were obtained for both material conditions.

Maraging 18Ni250

Maraging 18Ni250 steel is an alloy containing about 18% nickel, 8% cobalt, 5% molybdenum and smaller amounts of titanium and boron. Carbon content is about 0.03%. The material is heat treated by annealing at 1500F for 30 minutes, air cooling and reheating to 900F for 3 hours. This condition was compared with samples given the same annealing and reheating treatments but cooled by nitrogen gas quenching. The quenching rate of the nitrogen for this material was found to be about 25 to 50 seconds from 1500F to room temperature.

Mechanical properties for these material conditions are shown in Table 2. As can be seen, both conditions have similar properties.

The microstructures of the materials are shown in Fig. 6. Both materials are quite acicular with some banding. This is common for maraging steels of this type. Electron microscopic studies of the samples revealed that the nitrogen gas quenched sample had a finer acicular structure than the air cooled sample, which apparently reflects the faster quenching rate of the gas.

Type 316 and Type 321 Stainless

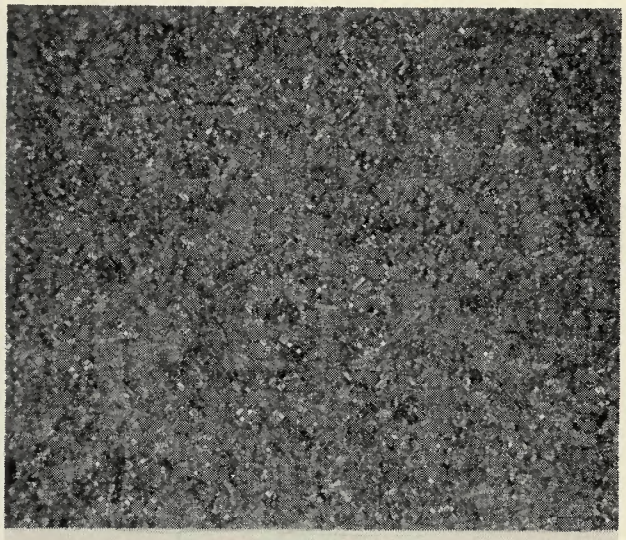
Type 316 and type 321 stainless steel are austenitic grades which contain chromium and nickel as the principal alloying elements. Both alloys cannot be hardened by heat treatment and because of this and other similarities only type 321 was evaluated for its behavior after gas quenching versus conventional water quenching.

The annealing temperature range for type 321 is approximately 1750 to 2050F; however, the furnace used in these studies was not capable of these

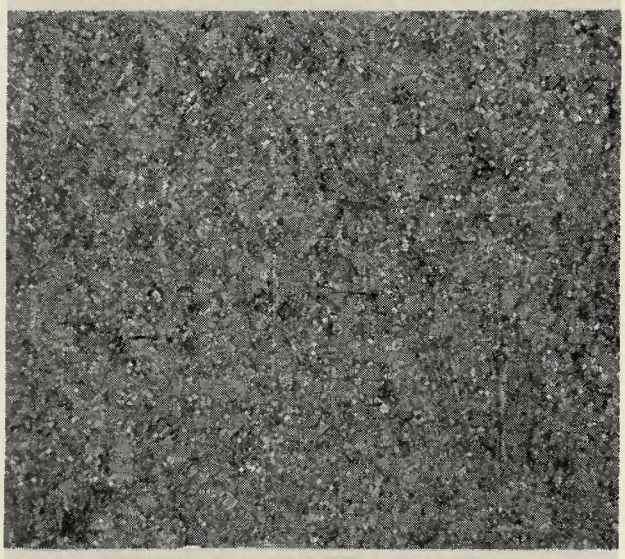
(a) Oil Quenched

(b) Nitrogen Gas

Fig. 4—Microstructures of 4130 steel austenitized at 1575F for 15 minutes, oil or nitrogen gas quenched and tempered at 1050F for 1 hour. Mag: 500X



(a) Oil Quenched

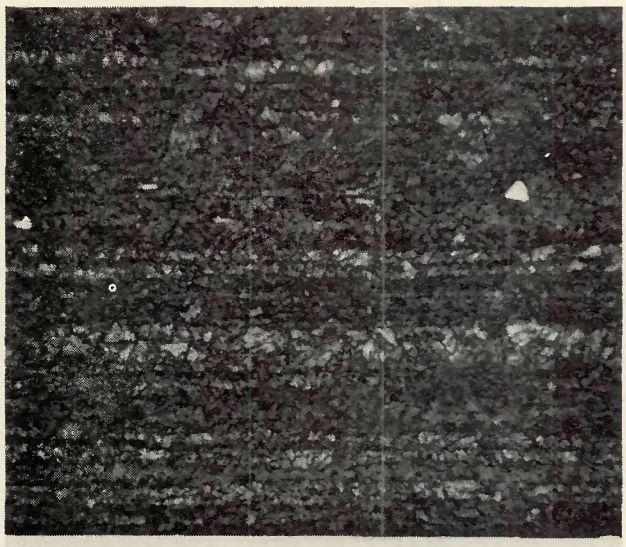


(b) Nitrogen Gas Quenched

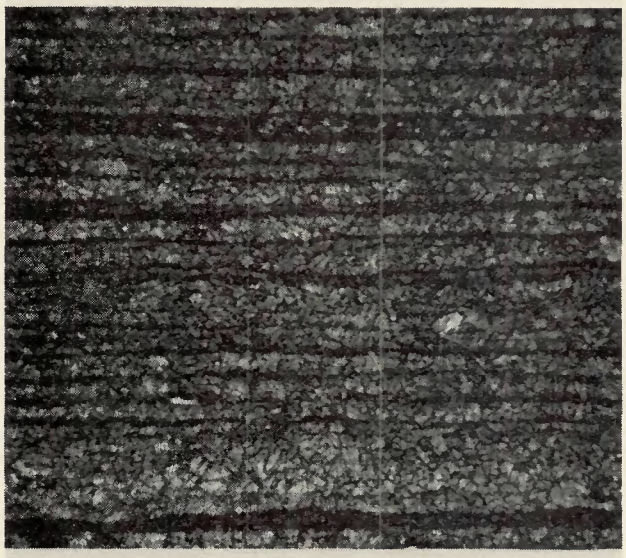
Fig. 5—Microstructures of 4340 steel austenitized at 1500F for 30 minutes, oil or nitrogen gas quenched and tempered at 900F for 1 hour. Mag: 500X

Table 3—Mechanical Properties of Type 321 Stainless Steel Vacuum Heat Treated and Gas Quenched

Thickness	Quenching medium	Quenching time from 1500 to 120F sec	Ultimate tensile strength ksi	Yield strength ksi	Elongation %
0.125	Water	—	88.8	38.7	48.0
0.125	Argon	40	87.4	39.6	48.5
0.125	Nitrogen	35	90.5	39.9	48.0
0.125	Helium	24	91.7	40.1	51.0



(a) Air Cooled



(b) Nitrogen Gas Quenched

Fig. 6—Microstructures of 18Ni250 maraging steel annealed at 1500F for 30 minutes, air-cooled or nitrogen gas quenched and reheated to 900F for 3 hours. Mag: 500X

temperatures. Because of this, the samples were heat treated by heating only to a temperature of 1500F and holding for 30 minutes. Following heating, the specimens were gas quenched in argon, nitrogen or helium. Control specimens were heated for the same time at the same temperature and quenched in water. The mechanical properties of these specimens are shown in Table 3. As can be seen, the properties for the gas quenched specimens are equal to those for the water quenched one.

The microstructures of these samples are shown in Fig. 7. The structures for all the samples are essentially the same.

Brazing Studies

Several of the samples were brazed into various configurations to determine, among other things, the quality of the joints that could be obtained. Several of these are shown in Fig. 8.

Although several different filler metals were evaluated in the program, only three were used in this part of the program: AMS 4777 was used to braze the low alloy and corrosion resistant steels. Braze alloy No. 719 was used for the 2219 aluminum and braze alloy No. 718 was used for the 6061 aluminum. These are described separately below.

Low Alloy and Corrosion Resistant Steels

AMS 4777 brazing alloy was used to braze the low alloy and corrosion resistant steels. This brazing alloy is self-fluxing and has been found to perform well in vacuum. The alloy is nickel base and contains about 7% chromium, 3% iron, 4.5% silicon, 3% boron and 0.06% maximum carbon. For brazing, this alloy was mixed with a lacquer binder to form a slurry which could be applied around joints. Brazing of all the low alloy and corrosion resistant alloy steels was done at 1925F for 15 minutes. A vacuum of about 5×10^{-5} torr was achieved during brazing. The samples were quenched directly from the brazing temperature.

The corrosion resistant steels were put in an annealed condition by the brazing process and therefore were evaluated in this condition. The low alloy steels were heat treated following brazing as follows:

- 4130 steel—austenitize at 1575F for 15 minutes, nitrogen quench, temper at 1050F for 60 minutes
- 4340 steel—austenitize at 1525F for 30 minutes, nitrogen quench, temper at 900F for 60 minutes
- 18Ni250 steel—anneal at 1500F for 30 minutes, nitrogen quench, heat to 900F for 3 hours

Figures 9 through 12 are photomicrographs of metallographic cross sections through representative brazed joints made using the steel materials.

Figure 9 is of a brazed joint between 4130 steel sheet material. As can be seen, the braze contains only a small amount of porosity and there is a minimum of alloying with the base metal. The shear strength of brazes

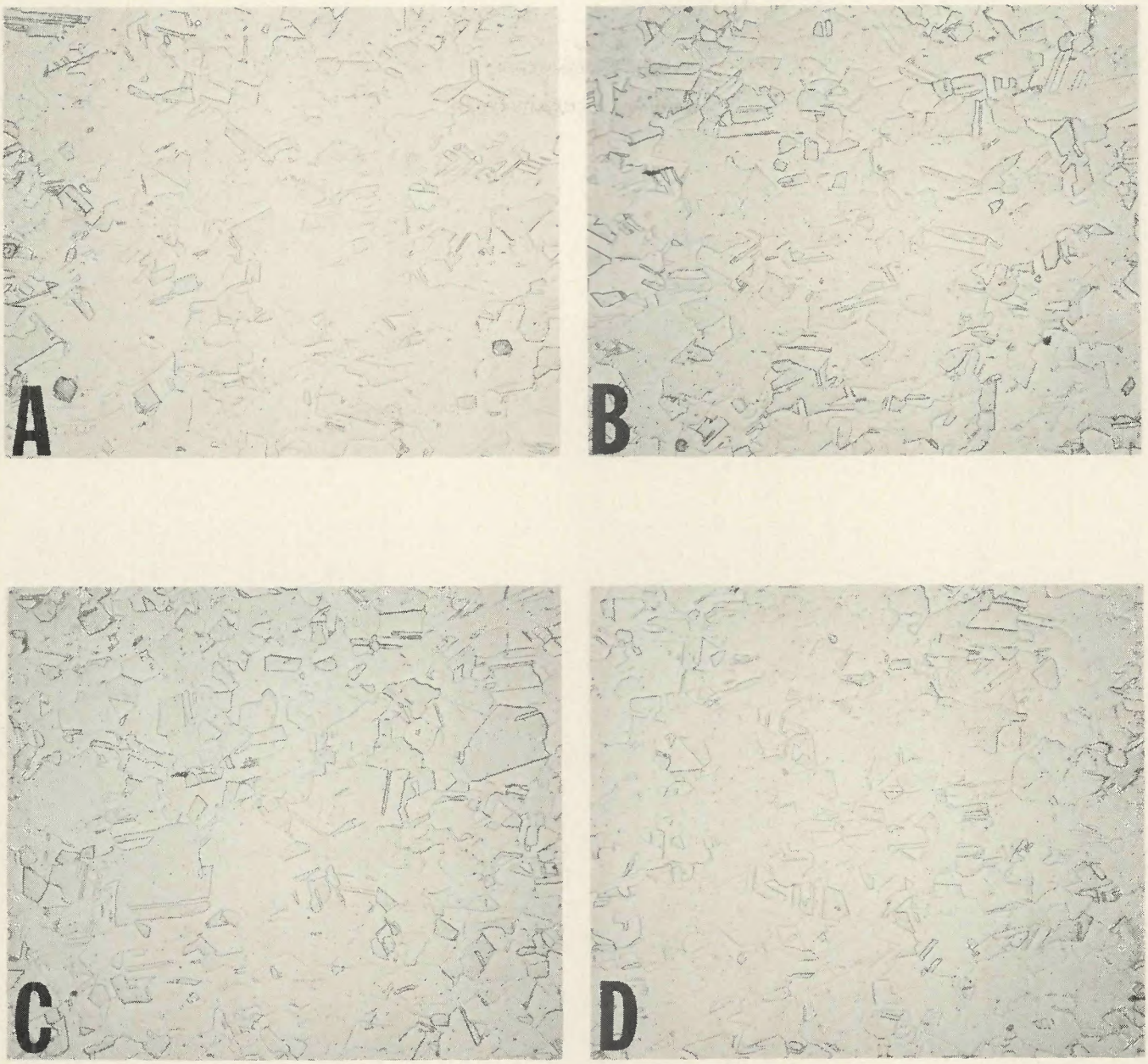


Fig. 7—Microstructures of Type 321 stainless steel heated to 1500F for 30 minutes and water or gas quenched. (a) water quenched; (b) argon gas quenched; (c) nitrogen gas quenched; and (d) helium gas quenched. Mag: 500X

made in this material as measured from shear-lap specimens was found to be good.

Figure 10 is of a brazed joint between 4340 steel sheet material. The brazes made in this material were similar to those in the 4130 steel material in that they contained little porosity and had only a small amount of alloying with the base metal. The flow of the brazing alloy was observed to be excellent.

Figure 11 shows a brazed joint between sheets of the 18Ni250 maraging steel. The brazing alloy showed good flow and filleting but in general the brazes in this material contained more porosity than in the 4130 or 4340 steels. The shear strength of the braze was found to be comparable to that of the brazes in the 4340 material.

Figure 12 contains photomicrographs of a brazed joint in type 316 stainless steel. Again, the flow and filleting of the brazing alloy was good and the amount of porosity was very small. Some alloying of the brazing alloy with the base metal was observed but in general the brazes were considered to be of acceptable quality.

Brazing of Aluminum Alloys

There is no commercial brazing alloy available for use with 2014 aluminum because of the low temperature at which this material begins to melt. Therefore, no brazing attempts were made on 2014 aluminum.

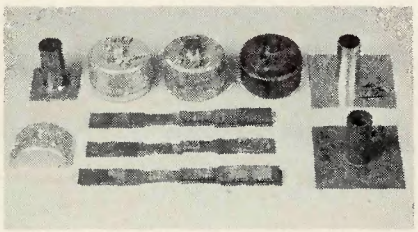
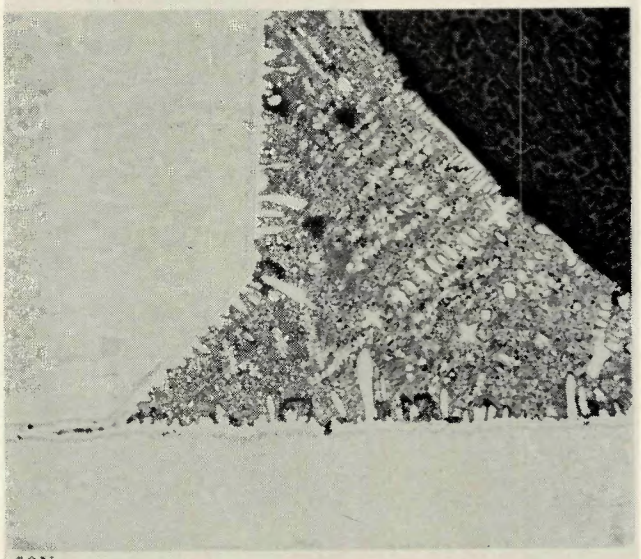


Fig. 8—Samples joined by vacuum brazing plus vacuum heat treated and gas quenched

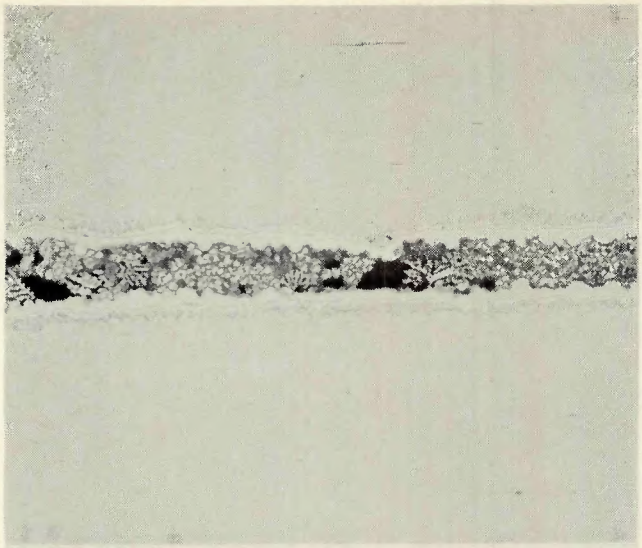
Brazing alloy No. 719 was used to braze the 2219 aluminum alloys. It is an aluminum-base alloy containing about 10% silicon, 10% zinc, 4% copper, 0.8% iron, and minor additions of magnesium, manganese and chromium.

100X Electrolytic Chromic Acid Etch

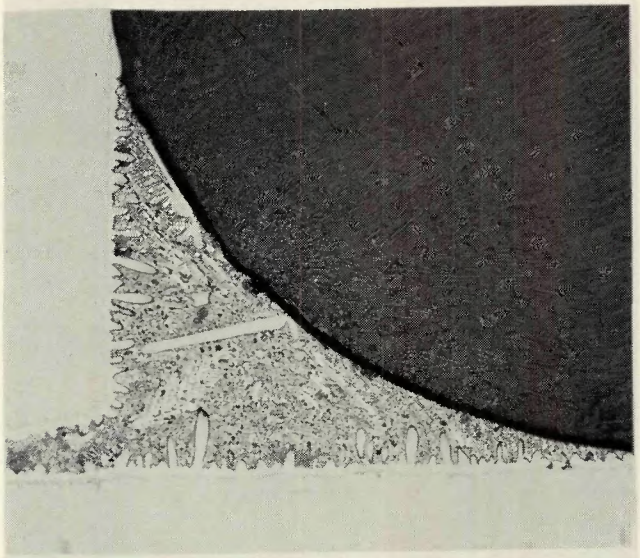


50X Electrolytic Chromic Acid Etch

Fig. 9—Brazed joint 4130 steel to 4130 steel using AMS 4777 brazing alloy

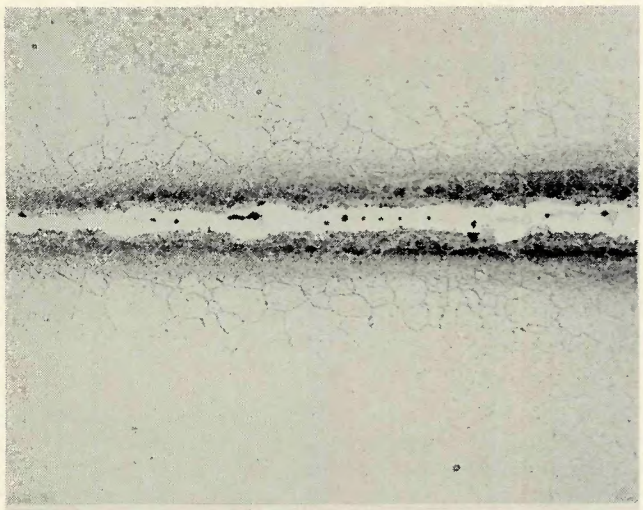


100X Electrolytic Chromic Acid Etch



50X Electrolytic Chromic Acid Etch

Fig. 10—Brazed joint 4340 steel to 4340 steel using AMS 4777 brazing alloy



100X Glyceregia Etch

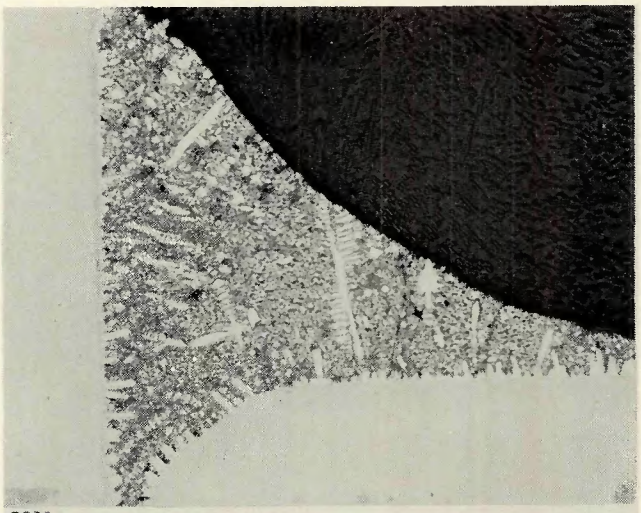


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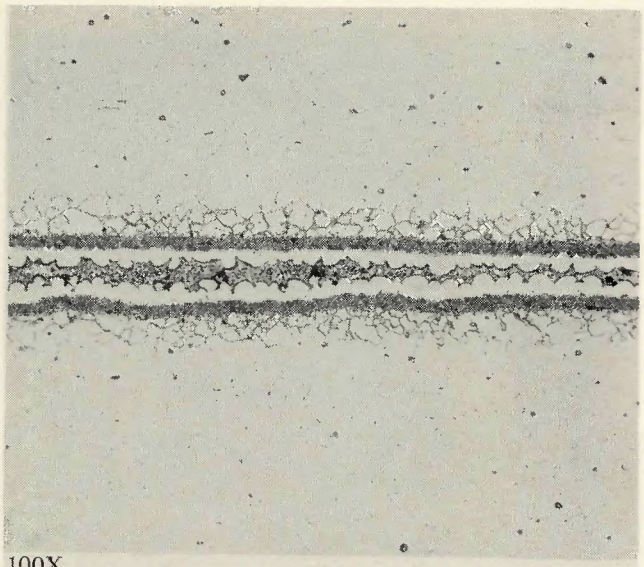
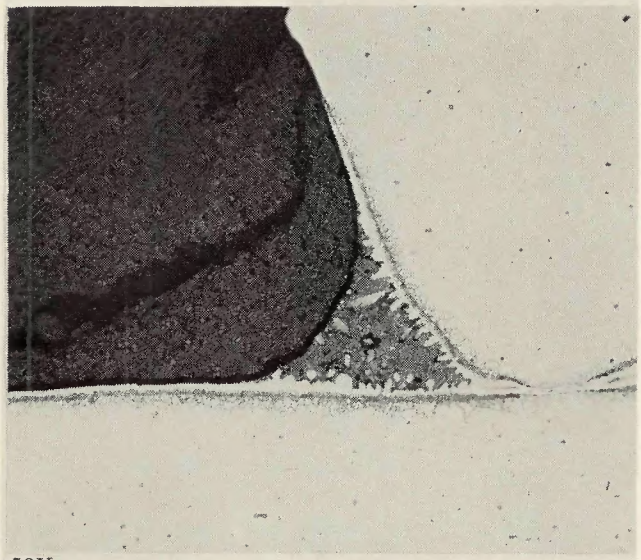
Fig. 11—Brazed joint 18Ni250 steel to 18Ni250 steel using AMS 4777 brazing alloy

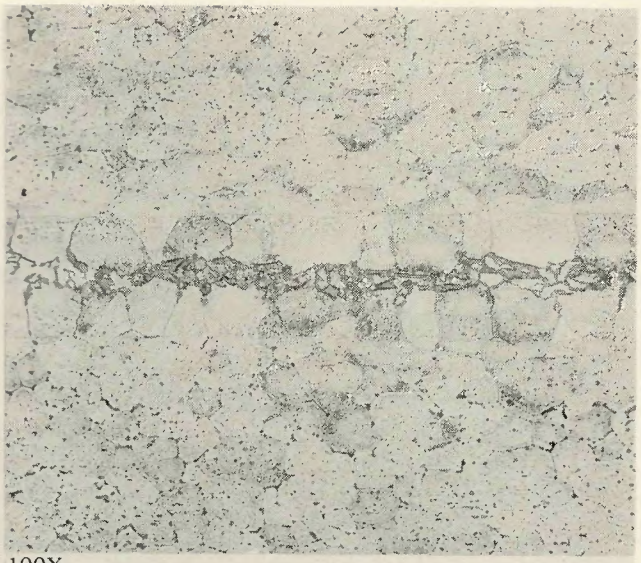


100X Electrolytic Chromic Acid Etch

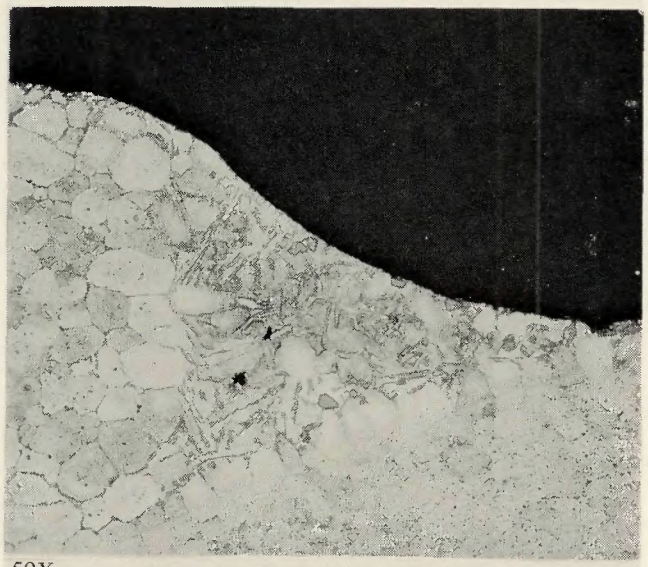


50X Electrolytic Chromic Acid Etch

Fig. 12—Brazed joint 316 stainless steel to 316 stainless steel using AMS 4777 brazing alloy



100X 1/2 Percent Hydrofluoric Acid Etch



50X 1/2 Percent Hydrofluoric Acid Etch

Fig. 13—2219 aluminum container brazed using No. 719 brazing alloy

Brazing was done by wrapping the work pieces in aluminum foil and placing small chips of magnesium in the foil. (Magnesium vaporizes and encourages flow and filleting by the brazing alloy.) The brazing was done at 1060F at a vacuum of about 5×10^{-5} torr.

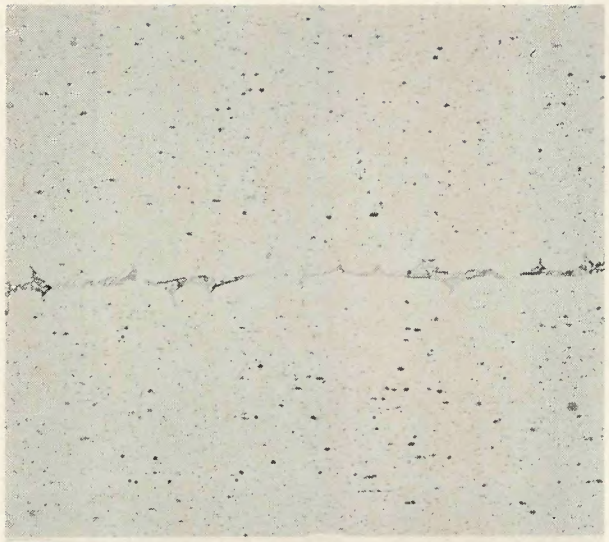
Since the brazing temperature of 1060F is above the initial melting temperature of 2219 aluminum, which is 1010F, incipient melting of the grain boundaries of the base metal would be expected. This did occur as shown by the photomicrographs in Fig. 13. Even though sound welds were achieved, the incipient melting undoubtedly would lower the mechanical properties of the material and therefore it does not appear that vacuum brazing of this alloy can be done using this brazing alloy.

Brazing alloy No. 718 was used to braze the 6061 aluminum alloy. This brazing alloy has an aluminum base and contains about 12% silicon, 0.8% iron, 0.3% copper and minor amounts of zinc, magnesium and manganese. Brazing was done at 1100F in a vacuum of about 5×10^{-5} torr. This temperature is below the initial melting temperature of 6061. After brazing, the samples were vacuum heat treated at 985F for 20 minutes, nitrogen gas quenched and artificially aged at 320F for 18 hours.

Figure 14 contains photomicrographs of a typical brazed joint in 6061 made with the No. 718 brazing alloy. The braze was of good quality and free of porosity. Shear-lap specimens revealed brazes in this material to have good strength.

Summary

All of the materials studied were found to have comparable properties and microstructures when conventionally heat treated and quenched and when heat treated in vacuum and quenched by gas. In fact, in some materials, improved properties were attained by the gas quenching process. Brazed joints of good quality were achieved by the process in all the materials studied except the 2014 aluminum, for which no suitable brazing alloy with low enough melting



100X 1/2 Percent Hydrofluoric Acid Etch

50X 1/2 Percent Hydrofluoric Acid Etch

Fig. 14—6061 aluminum container brazed using No. 718 brazing alloy

point is available, the 2219 aluminum in which incipient melting of grain boundaries occurred, and the 18Ni250 steel in which brazed joints with good flow and filleting were achieved but in which quite a bit of porosity was present. The results indicate that the VBGQ process holds promise as a reliable method of joining certain materials.

References

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