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Brazing of a Dispersion Strengthened Nickel Base Alloy Made by Mechanical Alloying

A hard-to-weld dispersion strengthened alloy can be brazed with strength of 80% of base metal strength; oxidation resistance is good.

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ABSTRACT. This paper discusses the joining of a dispersion strengthened alloy made by a new process called mechanical alloying. This process makes it possible for the first time to produce alloys with the long sought combination of dispersion strengthening and age hardening. After a brief review of the joining of dispersion strengthened materials and of the difficulties of arc welding them, the paper concentrates on the results of a brazing program. Thirteen brazing alloys, some commercial and some experimental, were examined. Brazing was carried out in dry hydrogen and in a vacuum. Evaluation of the brazed joints consisted of x-radiography, metallography, elevated temperature tensile testing, and high temperature oxidation resistance. Both single lap and Miller-Peaslee specimens were tested.

It is shown that brazed joints with 1900 F tensile strengths corresponding to approximately 80% of base metal strength can be made with commercial and experimental brazing alloys. Oxidation resistance similar to that of the sheet can also be achieved. Brazing must be done in vacuum because flow of the brazing alloy does not occur in dry hydrogen.

Introduction

Mechanical alloying is a new process which can produce homogeneous composite particles with an internal structure that is both uniform and intimately dispersed (Ref. 1). The key feature of the process is high energy ball milling of the constituent powders. This is done dry, without surfactants, with the result that the powders weld to each other. Over a period of time this welding and subsequent breaking of the particles causes them to be intimately mixed or kneaded into each other (Ref. 2). Figure 1 illustrates the process occurring with time. It can be seen that the powders become progressively more intimately mixed until after 40 h a homogeneous structure is formed. This fully processed powder is packed into steel cans and consolidated by hot working.

Although this method of alloying by mechanical means can be applied to a wide range of materials, one obviously important area is the production of nickel-base superalloys. This is because mechanical alloying makes it possible for the first time to produce alloys with the long sought combination of dispersion strengthening and age hardening. This combination was not achievable before be-

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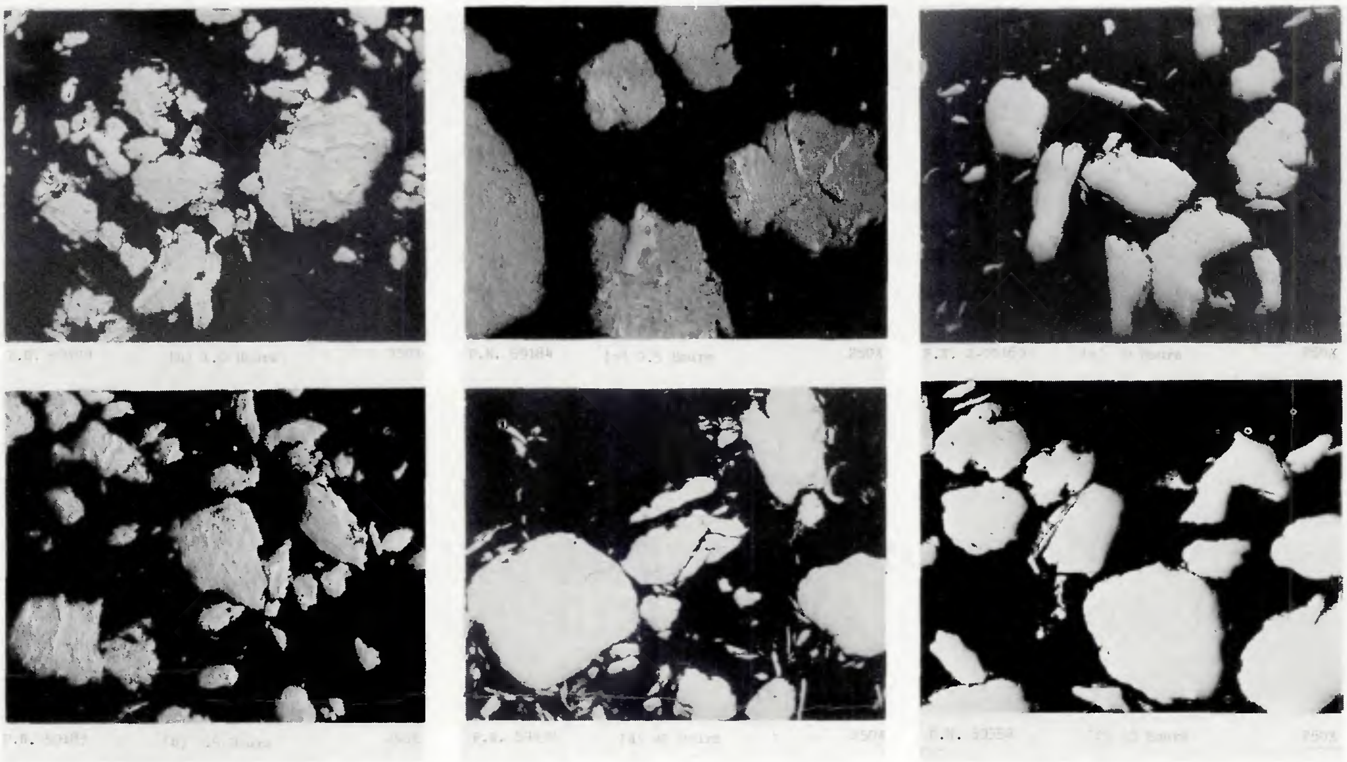

Fig. 1 — Structures of composite powders processed for various times in a 10-S attritor operated at 132 rpm. Ball charge; 175 kg of +0.6 cm nickel pellets; 10 kg of powders. Reduced 56%

cause the methods that were available to make dispersion strengthened materials with a sufficiently fine dispersion of a stable oxide were not suited to the incorporation of gamma prime (γ') forming elements such as aluminum, titanium and columbium. The reasons were either that the γ' formers became oxidized or, as in the case of the selective reduction process, the oxides of aluminum and titanium were too stable to be readily reduced to the metals (Ref. 1).

The alloy described in this report, IN-853, is a nickel-base alloy containing chromium, aluminum and titanium and dispersion strengthened with yttria. Its composition and tensile properties are shown in Table 1. Of particular interest is its 1000 h strength which is plotted against temperature in Fig. 2. The curves for NIMONIC* alloy 80A and TDNi are included for comparison and to show how the IN-853 combines the intermediate strength of NIMONIC alloy 80A (precipitation hardened by gamma prime) with the high temperature strength of TDNi (dispersion strengthened with thoria). The fine structure of IN-853 is shown in Fig. 3.

The difficulties of welding dispersion strengthened alloys result primarily from the need to maintain a well distributed dispersoid phase in order to retain the high temperature strength. When dispersion strengthened materials are arc welded, for example, the dispersoid frequently agglomerates to form pockets of slag which cause porosity and cracking in the weld. This seems to happen with all fusion processes although the extent of the agglomeration varies (Refs. 3 to 13).

In our studies of the joining of mechanically alloyed materials we made gas tungsten-arc (GTA), flash butt, resistance spot and inertia welds in the IN-853 alloy. The GTA, flash butt and resistance spot welds all showed agglomeration but the spot welds in 1/16 in. sheet exhibited approximately 90% joint efficiency in tensile tests at 1400 F and 1900 F. In stress rupture tests, the spot welds sustained moderate loads for extended periods of time but their rupture strengths did not approach those of the unwelded sheet.

Sound welds were also made by the inertia welding process and they performed well in short-time tensile tests at room temperature and 1400 F. The room temperature strengths, for example, corresponded to 100% joint efficiency. However in stress rupture tests at 1400 F and 1900 F all the specimens failed in relatively short times at low loads.

This work with the use of conventional welding processes to join mechanically alloyed materials was part of a wider program which also included brazing and diffusion bonding. As the program progressed it became apparent that processes such as brazing and diffusion bonding which do not melt the base metal would probably be the more suitable joining pro-

Table 1 — Nominal Composition and Tensile Properties of IN-853

Nominal Composition

C	Al	Ti	Cr	Zr	B	Y_2O_3	Ni
.05	1.5	2.5	20.0	.07	.007	1.3	Bal.

Tensile Properties of Bar

Temp., F	0.2% Y.S. ksi	U.T.S. ksi	Elong. %	R.A. %
R.T.	129	175	9	14
1400	81	84	25	33
1900	21	23	9	19

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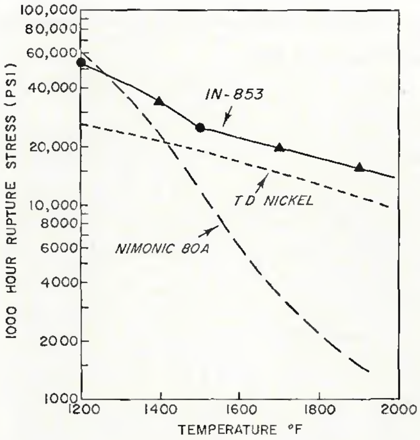

Fig. 2 — Stress for 100 h life in IN-853 as a function of temperature. The values for TDNi and Nimonic 80A are included for comparison (from Ref. 1)

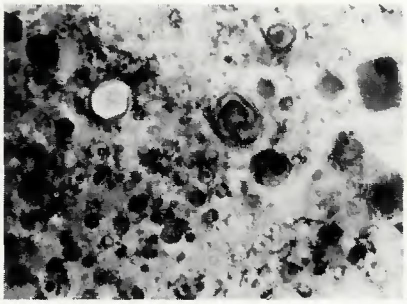

Fig. 3 — Transmission electron micrograph showing structure of IN-853 (X99,000, reduced 43%)

cesses and, for that reason, emphasis was placed on them. The results of the brazing program are described here.

Experimental Procedure

Base Materials

Brazing was done on an experimental batch of IN-853 sheet usually produced by rolling of as extruded bar. Commercial quantities of sheet are not yet available. The sheet was always brazed in the heat treated, coarse grained condition (Fig. 4) in which the material exhibits its highest elevated temperature strengths (Refs. 1, 14).

Fig. 4 — Longitudinal cross section of extruded IN-853 (From Ref. 1:X100, reduced 9%)

Brazing Alloys

Thirteen brazing alloys were evaluated. They can be grouped as follows:

1. Commercial nickel-base high temperature alloys (A through E in Table 2)
2. An alloy of the platinum group (F in Table 2)
3. Brazing alloys developed for TDNi and TDNiCr (Ref. 15) (G and H in Table 2)
4. Alloys of our own design selected on basis of their melting temperature (I through L in Table 2)
5. Cu-10%Ni (M in Table 2) used because of a report of its successful use to braze nickel alloys (Ref. 16).

Alloys A through E and Alloys G and H are commercially available in powder form while the 60Pd-40Ni (Alloy F) and the Cu-10%Ni (Alloy M) were purchased as wire. We made the other four alloys (Alloys I through L) by melting in the electron beam furnace and crushing to fine chips.

Brazing Techniques

A few initial brazing tests were conducted in a tube furnace through which dry hydrogen from a cracked ammonia generator and palladium diffuser flowed continuously. Most of the brazing, however, was done in a vacuum unit consisting of a two in. diam quartz tube, a vacuum pumping station, and a high frequency induction coil. A Pt-10%Rh thermocouple spot welded to the specimen and passed through a vacuum seal was used to record temperature.

The single lap joints were prepared in the manner recommended as a standard by the American Welding Society (Ref. 17). Joints were made in 1/8 in. thick IN-853 sheet with overlaps of 1/16, 1/8, 1/4, 1/2 and 3/4 in. In these specimens, a clearance of 0.002 in. was established by using stainless steel shims outside the test area and then tack welding the specimen edges (Fig. 5). The shims were subsequently machined away during preparation of tensile specimens.

Brazing alloys in the form of wire were placed in the position shown in Fig. 5 and held in place by a few drops of brazing cement. Alloys in the form of powder or fine chips were first mixed with brazing cement to form a slurry which was then applied to the specimen.

Miller-Peaslee specimens (Ref. 18) were also prepared in the recommended manner using contact clearance and with the brazing alloy placed along the entire length of the joint on one side of the specimen.

All pieces of IN-853 to be brazed were first etched at 120 F in a solution of HF, HNO_3 and water, an etchant recommended for nickel-base alloys in the Brazing Manual (Ref. 19).

Brazed joints were made by heating the specimen from 1000 F to the brazing temperature at a rate of approximately 200 F/min, holding at temperature for 5 min and then cooling freely in vacuum. Brazing vacuums ranged from 10^{-2} to 10^{-4} mm Hg while cooling vacuums were of the order of 10^{-4} to 10^{-5} mm Hg.

Evaluation of Brazed Joints

In early flow tests, samples of the brazing alloy were placed on the surface of a piece of IN-853 and the sample heat treated to the brazing temperature to see if flow occurred. Alloys showing most promise were used to make single lap joints. All joints were X-rayed to examine for flow of the brazing alloy. They were then tensile tested at elevated temperature (1900 F) and examined metallographically for extent of flow, degree of erosion, and the general appearance of the joints. When the four most suitable brazing alloys had been selected, each alloy was used to make single lap shear joints with a range of overlap distances and these were tensile tested at 1900 F. At this stage, the Miller-Peaslee specimens were also prepared and tested. Finally, joints made with the two strongest brazing alloys were oxidation tested in air and in air +5% H_2O at 1830 F (1000 C) and their resistance to oxidation determined through metallographic examination.

Table 2 — Compositions and Brazing Temperatures of the Brazing Alloys that were Evaluated

Alloy ident.	Composition, %	Brazing temp., F
A	Ni-19Cr-10Si	2125-2175
B	Ni-16.5Cr-4Si-4Fe-3.8B	1950-2200
C	Ni-7Cr-4Si-3Fe-3B	1850-2150
D	Ni-4.5Si-2.9B	1850-2150
E	Ni-3.5Si-1.0B	1900-2150
F	60Pd-40Ni	2260-2300
G	Ni-16.1Cr-16.3Mo-5.9Fe-4W-4Si	2375
H	Ni-16Cr-25.5Mo-.17Fe-4W-4.3Si	2375
I	79Co-21Cb	2260
J	76.5Ni-23.5Cb	2320
K	48.4Ni-51.6Cb	2140
L	68Co-32Ta	2330
M	Cu-10Ni	2200

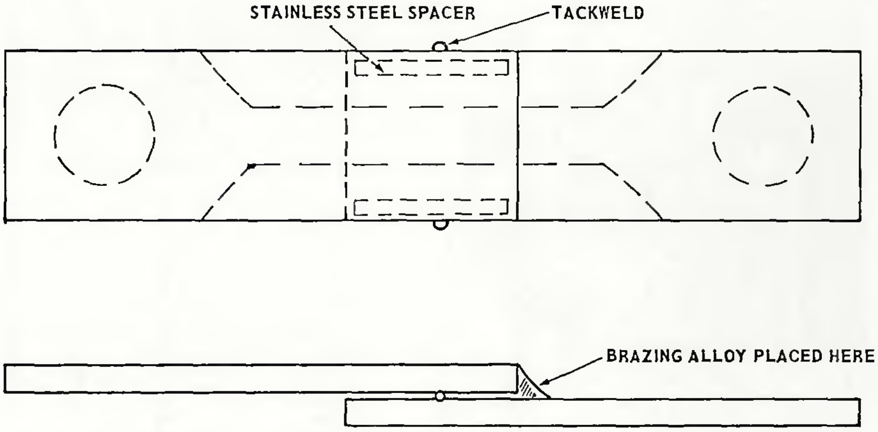

Fig. 5 — Single lap shear test specimen

Results

Flowability Tests in Hydrogen and Vacuum

The first five nickel-base alloys listed in Table 2 were evaluated at 2150 F in dry hydrogen. On IN-853 no flow occurred with any of the alloys even though flow occurred on a piece of INCONEL* alloy 600 which was included as a control. Running hydrogen through the furnace for several days to make sure the tube was free from contamination before the sample was put into the hot zone did not alter this situation. However when the IN-853 pieces were nickel plated, flow occurred with all five brazing alloys.

In the good vacuum ($<10^{-4}$ mm Hg) of the experimental vacuum brazing unit that we built, flow occurred readily with all thirteen alloys. Under these conditions in which good flow could be obtained reproducibly, it was possible to make and evaluate actual joints.

Joints Made in Vacuum

For the joints to be made in vacuum, eight of the original thirteen brazing alloys were selected for the reasons outlined below. Those selected were Alloys A, C, F, H, I, J, L, and M in Table 2.

1. Alloy A is a commonly used alloy which in one study (Ref. 15) had been selected over others as the most suitable brazing alloy for TDNi for service up to 1900 F. Also since it does not contain boron, difficulties caused by erosion were expected to be minimized.
2. Of the other four commercial nickel-base alloys (B, C, D, and E) we picked Alloy C because it contained chromium for corrosion resistance and had a relatively low boron level.
3. Alloy H was preferred over the similar Alloy G because of indications of greater stability in elevated temperature exposures (Ref. 15).
4. Alloy J was preferred over Alloy K because although both are from the same alloy system, Alloy J has the higher melting temperature and therefore was expected to be stronger at elevated temperatures.

The eight alloys selected were used to make lap joints in sheet approximately 1/16 in. thick using a 3/8 in. overlap. Brazing was done on the high end of the brazing range and all eight alloys flowed completely down the 3/8 in. overlap. Tensile samples machined from these pieces were tensile tested at 1900 F. All specimens broke at similar loads through tensile failure in the sheet.

In view of the sheet failures the relative merits of the brazing alloys were evaluated principally on the basis of metallographic appearance of the joints. This assessment of degree of erosion and general appearance is given in Table 3. The boron-containing nickel alloy (Alloy C), the 60Pd-40Ni (Alloy F), and the 76Ni-25Cb (Alloy J) all produced substantial erosion and consequently were rejected (Fig. 6 shows an example). The appearance of the joints made with the 68Co-32Ta alloy (Alloy L) was also considered unsuitable. Joints made with the remaining four alloys were considered acceptable (Fig. 7 through 10). These four alloys (A, H, I, and M) were selected for further evaluation and used to make single lap joints in 1/8 in. IN-853 sheet. Each alloy was used to make joints with overlap distances of 1/16, 1/8, 1/4, 1/2 and 3/4 in.

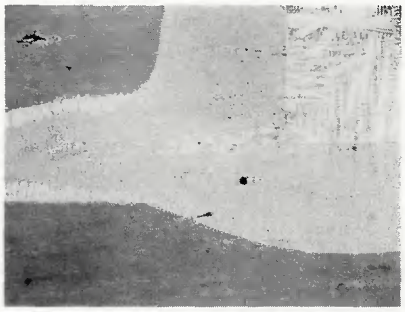

Fig. 6 — An example of excessive erosion at braze fillet (X50, reduced 61%)

Strength of Single Lap Shear Joints

The results of tensile tests of the single lap shear specimens are listed

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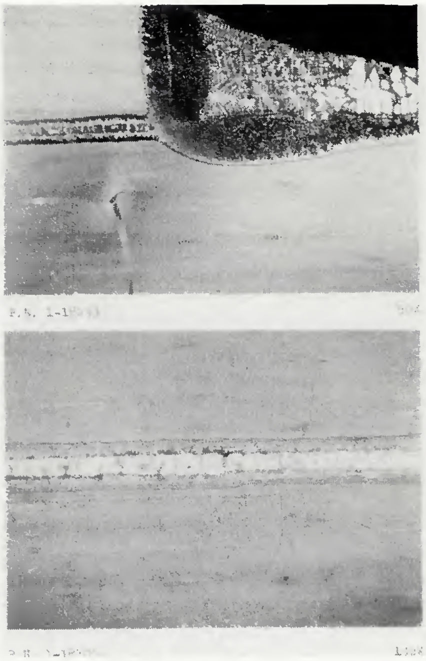

Fig. 7 — Appearance of joint made with alloy A (Ni-Cr-Si); both reduced 61%

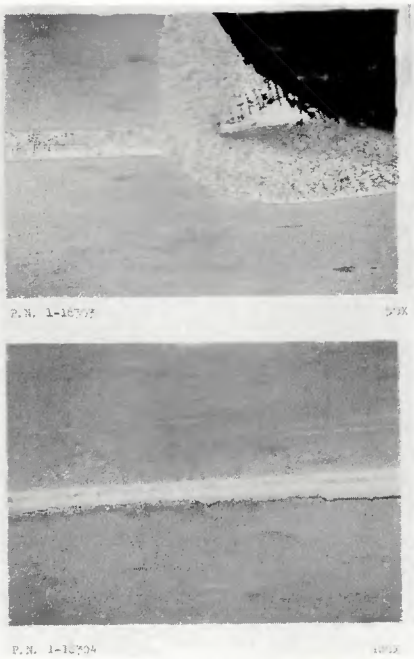

Fig. 8 — Appearance of joint made with alloy H (Ni-Cr-Mo-Fe-W-Si); both reduced 63%

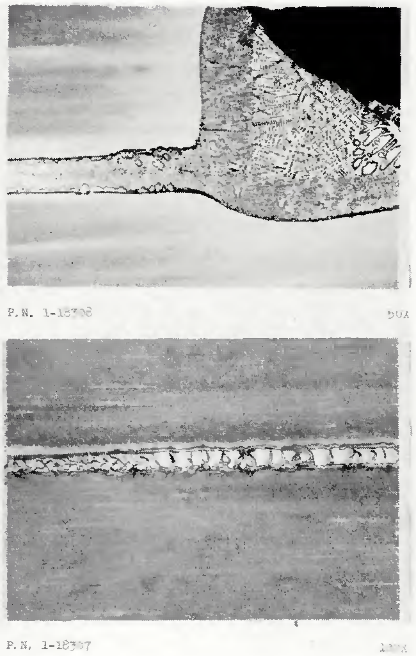

Fig. 9 — Appearance of joint made with alloy I (Co-Cb); both reduced 63%

Table 3 — Assessment of Brazed Joints on the Basis of Appearance[a]

Brazing alloy	Degree of erosion	Bond appearance
A (Ni-Cr-Si)	Little	Uniform, sound
C (Ni-Cr-Si-Fe-B)	Severe	Uniform, sound
F (Pd-Ni)	Severe	Non-uniform, porous
H (Ni-Cr-Mo-W-Si)	Medium	Non-uniform, sound
I (Co-Cb)	Medium	Uniform, sound
J (Ni-Cb)	Severe	Non-uniform, cracks
L (Co-Ta)	Little	Attacked sheet
M (Cu-Ni)	Little	Uniform, sound

(a) Flow was completely down 3/8 in. overlap in each case

in Table 4. These results are also plotted in Fig. 11 to show average shear strength versus overlap distance and in Fig. 12 to show how average tensile strength varies with overlap distance. Solid points indicate that the specimen broke by tensile fracture in the sheet while open points show that failure was by shear of the joint. Of the four alloys tested, the 79Co-21Cb alloy (Alloy I) was the strongest although its margin over the Ni-Cr-Mo-W-Si (Alloy H) was very slight. The Ni-Cr-Si (Alloy A) and the Cu-10Ni (Alloy M) followed in order of decreasing strength.

Some of the single lap shear specimens failed by fibrous tearing of the sheet (Fig. 13) probably as a result of the bending stresses that are set up when specimens of this kind are pulled in tension. To circumvent this problem joints were made using the Miller-Peaslee specimen.

In the Miller-Peaslee tests only the Co-Cb (Alloy I) and Ni-Cr-Mo-W-Si (Alloy H) were examined and again they appeared to have very similar strengths (Table 5). In this case the Alloy H showed the slight superiority (Figs. 14 and 15).

Oxidation Testing of Brazed Joints

Oxidation tests were run for 100 h at 1830 F (1000 C) in air and in air +5% H_2O on brazed joints made with the two strongest alloys — Alloys H and I. The nickel alloy braze (Alloy H) exhibited oxidation resistance similar to that of the IN-853 while the Co-Cb alloy showed greater attack (Fig. 16 and 17).

The latter behavior would probably be expected for a simple binary alloy which does not contain chromium. It means that this alloy would most likely be useful only where the fabricated part is subsequently coated with an oxidation resistant coating.

Fig. 10 — Appearance of joint made with alloy M (Cu-10Ni); both reduced 61%

Discussion

The results of this work show that IN-853, a nickel-base alloy made by mechanical alloying, can be brazed in vacuum with commercial nickel-base brazing alloys and with a Co-Cb alloy

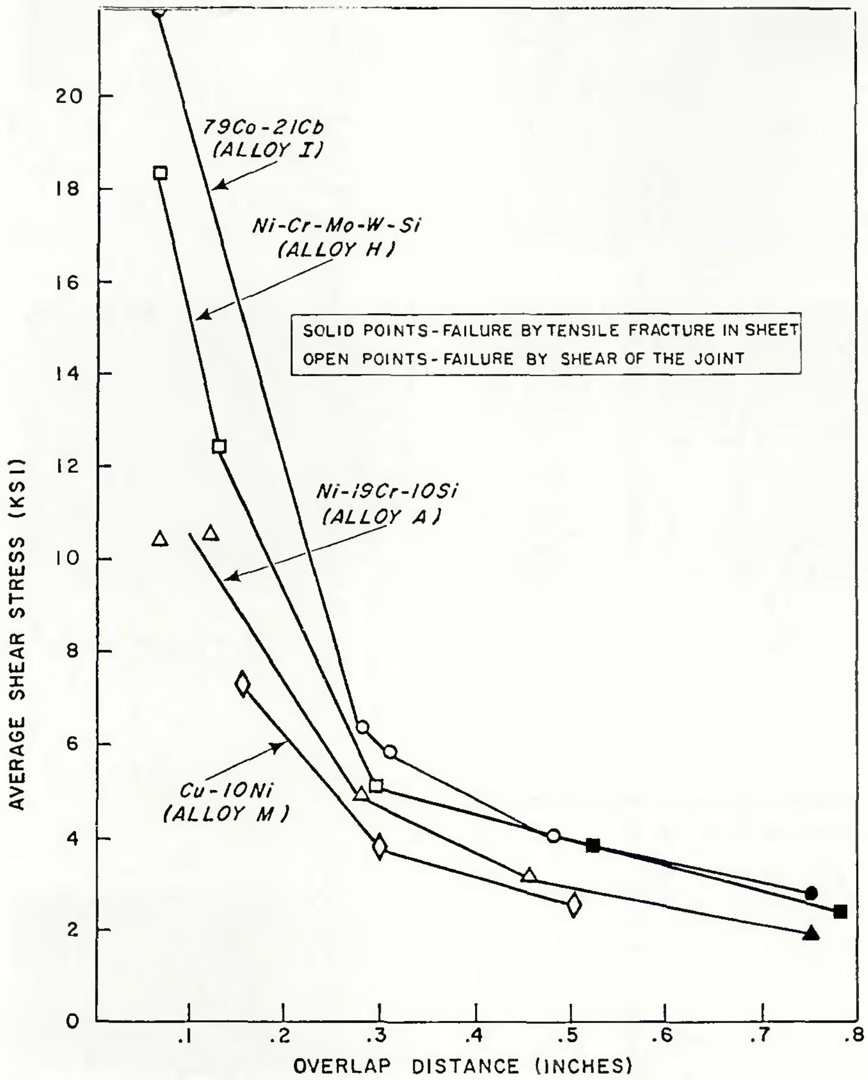

Fig. 11 — Average shear stress of brazed joints at 1900 F as a function of overlap distance (single lap shear specimens)

Table 4 — Strengths of Single Lap Shear Joints at 1900 F

Brazing alloy	Overlap distance in.	Shear stress, psi	Tensile stress, psi	Mode of failure
Ni-Cr-Si (A)	.07	10,400	5,570	Shear in Joint
	.12	10,600	9,500	Shear in Joint
	.28	4,900	10,200	Shear in Joint
	.46	3,100	10,800	Shear in Joint
	.75	1,960	11,300	Tensile Failure in Sheet
Ni-Cr-Mo-W-Si (H)	.07	18,300	9,840	Shear in Joint
	.13	12,300	11,800	Shear in Joint
	.29	5,100	11,200	Fibrous Tear in Sheet
	.52	3,950	15,600	Tensile Failure in Sheet
	.78	2,450	14,000	Tensile Failure in Sheet
Co-Cb	.07	21,900	11,700	Fibrous Tear in Sheet
	.28	6,300	13,200	Shear in Joint
	.31	5,900	13,700	Fibrous Tear in Sheet
	.48	4,000	14,700	Shear in Joint
	.75	2,700	15,500	Tensile Failure in Sheet
Cu-10Ni (M)	.16	7,300	8,800	Shear in Joint
	.30	3,800	8,700	Shear in Joint
	.50	2,600	10,000	Shear in Joint

which to our knowledge has not previously been used for brazing. The 60Pd-40Ni brazing alloy and the nickel alloys containing boron were not suitable because they produced substantial erosion of the sheet. Although brazing was readily accomplished in vacuum it was possible in hydrogen only if the IN-853 pieces were coated, for example, with nickel. Without the plating, flow of the brazing alloy did not occur. This is an apparent paradox because oxide equilibrium curves show that, at the temperature of interest, vacuums of the order of 10^{-27} mm Hg are needed to reduce Al_2O_3 whereas in dry hydrogen a comparatively practical dew point of approximately -175 F should do the job. Other workers have commented on this situation (Ref. 20) without being able to fully explain it. The literature does however appear unanimous that the nickel-base high temperature alloys are best brazed in vacuum. This is particularly so since there appears to be a trend away from plating surfaces in order to insure braze flow.

The elevated temperature strengths of brazements made with the commercial Ni-Cr-Mo-W-Si (Alloy H) and the experimental Co-Cb (Alloy I) were very similar. At the higher overlaps, tensile strengths were approximately 15 ksi. This value is approximately 80% of the 1900 F tensile strength of the particular batch of sheet being tested. In fact for the Alloy H single lap joints, tensile failure occurred in the sheet at overlaps of 1/2 and 3/4 in. (4X thickness and 6X thickness respectively) while for Alloy I, sheet failure occurred with the 3/4 in. overlap only. The 3/4 in. overlap joint made with Alloy A also failed in the sheet although in this case at a somewhat lower strength. Relatively few tests were made with the Cu-10%Ni alloy because of concern that the high copper content would make it unsuitable for high temperature service.

Some idea of the significance of the tensile results can be obtained by comparing them with values measured on 0.050 in. thick TDNi sheet which was brazed with Alloys G and H in Table 2. In tensile tests at 2000 F, the overlaps necessary to cause base metal failure in TDNi were 4t for Alloy G and 4.5t for Alloy H (Ref. 15). Thus the results obtained on IN-853 in this work show some agreement with previous data for TDNi.

With regard to oxidation resistance the 100 h tests at 1000 C (1830 F) suggest that the oxidation resistance of Alloy H is at least the equivalent of the IN-853 sheet. The Co-Cb alloy, on the other hand, appears deficient in this respect and would probably need to be coated. There is reason to think that it would be particularly suited for this, because alloys of this kind have been suggested as substrates for use prior to aluminizing (Ref. 21). The recommendation was based on their ability to form stable oxidation resistant aluminides.

Conclusions

1. Brazed joints with useful strengths have been made in IN-853 sheet with two commercial nickel-base alloys, a Cu-10%Ni alloy, and an experimental 79Co-21Cb alloy.

2. The strongest joints were made with a Ni-Cr-Mo-W-Si and the 79Co-21Cb brazing alloys. With 4t and 6t overlaps, tensile strengths at 1900 F corresponded to approximately 80% of the sheet strength.

3. The oxidation resistance of the Ni-Cr-Mo-W-Si alloy is similar to that of the IN-853 sheet. The Co-Cb has poorer resistance and would probably need to be coated.

4. Nickel brazing alloys containing boron and a 60Pd-40Ni alloy caused erosion of the IN-853 sheet and therefore do not appear to be suitable.

5. Brazing must be done in vacuum ($<10^{-4}$ mm Hg). Flow of the brazing alloy does not occur in dry hydrogen.

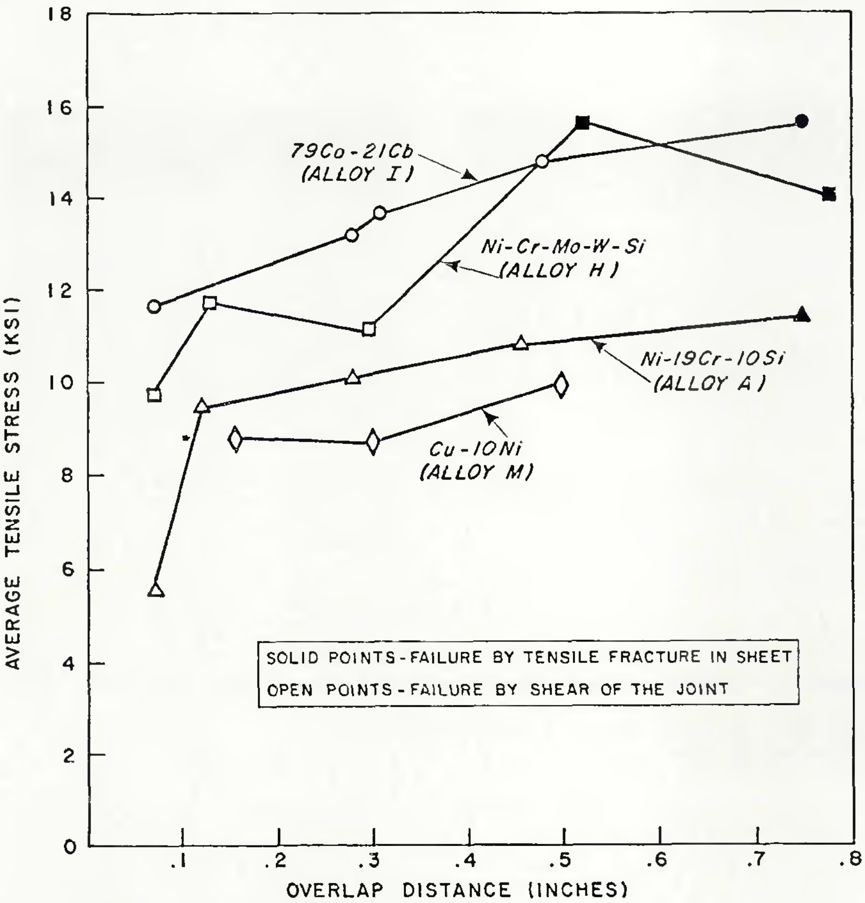

Fig. 12 — Average tensile stress of brazed joints at 1900 F as a function of overlap distance (single lap shear specimens)

Fig. 13 — Failure of a brazed joint by tearing through the sheet (X50, reduced 54%)

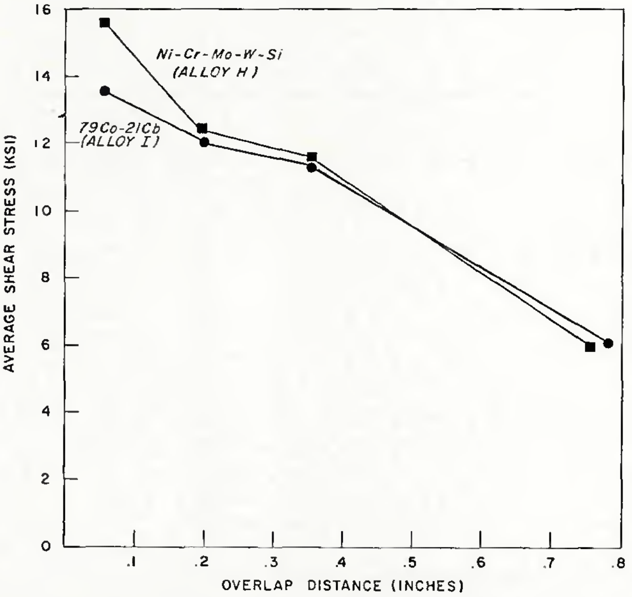

Fig. 14 — Average shear stress of brazed joints at 1900 F as a function of overlap distance (Miller-Peaslee specimens)

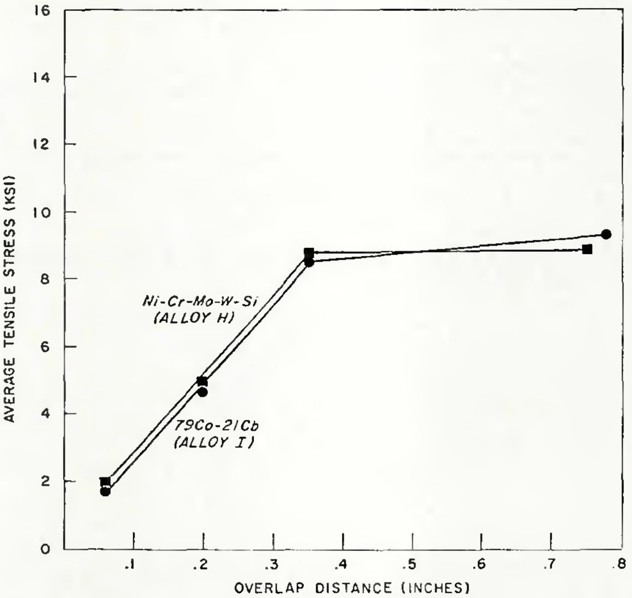

Fig. 15 — Average tensile stress of brazed joints at 1900 F as a function of overlap distance (Miller-Peaslee specimens)

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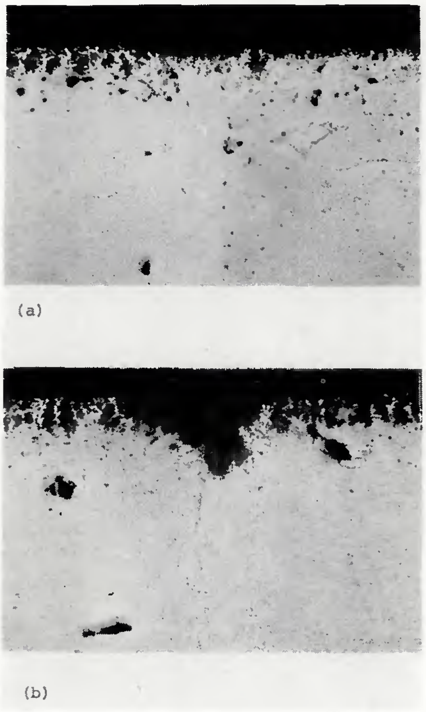

Fig. 16 — Oxidation resistance of brazed joints made with (a) alloy H and (b) alloy I exposed for 100 hours at 1830 F (1000 C) in air

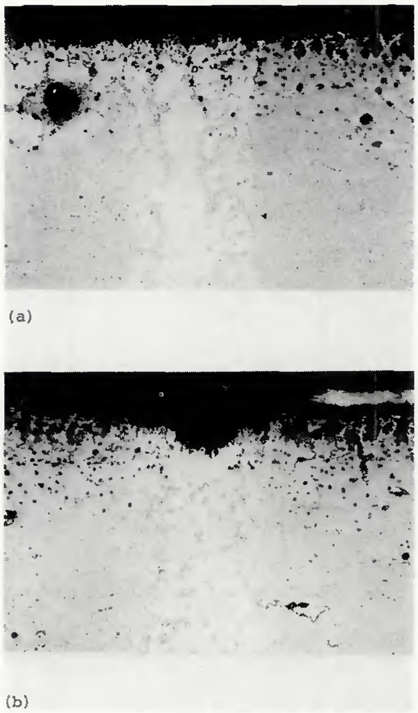

Fig. 17 — Oxidation resistance of brazed joints made with (a) alloy H and (b) alloy I exposed for 100 h at 1830 F (1000 C) in air + 5% H_2O

Table 5 — Strengths of Brazed Joints at 1900 F (Miller-Peaslee Specimens)

Brazing alloy	Overlap distance in.	Shear stress psi	Tensile stress psi
Ni-Cr-Mo-W-Si	.06	15,700	2,000
(H)	.19	12,300	4,912
	.36	11,600	8,784
	.76	6,000	8,928
Co-Cb	.06	13,600	1,744
(I)	.19	12,100	4,752
	.36	11,400	8,688
	.78	6,100	9,360