Solid-State Welding of TD-Nickel Bar

Butt and lap joint welds are made in a ½ in. diameter bar and best joint efficiency at 2000° F is obtained with a 0.004 in. cobalt-alloy interlayer

BY T. J. MOORE AND K. H. HOLKO

ABSTRACT. Solid-state butt and lap joint welds were made in ½ in. (12.7 mm) diameter TD-nickel bar in a hot isostatic press at 2000° F (1093° C) and 20 ksi (138 MN/m^2) for 2 hr. Various interlayer materials were used in the butt joints.

Best butt joint efficiency (percent of base metal strength) at 2000° F (1093° C) was obtained with a 0.004 in. (0.10 mm) cobalt-alloy interlayer: stress-rupture joint efficiency was 15% for 100 hr life and tensile joint efficiency was 100%. Thus, stress-rupture testing was found to be a more severe criterion. In short-time shear tests of lap joints (without interlayers), base metal shear strength was matched in 2000° F (1093° C) tests.

Introduction

Higher operating temperatures in advanced jet engines have increased the demand for high-temperature materials. One of the most promising materials in the 1900-2200° F (1038°-1204° C) temperature range is TD-nickel (TD-Ni). This material is strengthened with a fine dispersion of tho i (about 2-vol-%) in a pure nickel matrix.

TD-Ni has good ductility and therefore good fabricability. Joining by fusion welding processes, however, is not suitable because melting destroys the thoria dispersion and strengthening effects produced by thermomechanical processing. Solid-state welding processes would appear to offer great promise because welds can be made without melting the parent material. (A discussion of solid-state welding mechanism is presented in Appendix A.)

The objective of this study was to weld TD-Ni using a solid-state welding process which produced only micro-deformation. A hot isostatic pressure (HIP) process was selected to produce the welds. (This process has been called gas pressure bonding, but HIP welding is used throughout this paper and is suggested as a more proper term in Appendix B.)

TD-Ni in bar form was selected as the base metal for several reasons. In bar form TD-Ni is considerably stronger than sheet material.[1] The bar has a wrought microstructure that is stable to about 2400° F (1316° C) and application of solid-state welding techniques to TD-Ni bar has not been extensively investigated. In cursory studies of solid-state butt welds in TD-Ni bar, Metcalfe[2] reported 30% joint efficiency* in 2000° F (1093° C) tensile tests. In this study HIP welds were made in $^1/_2$ in. (12.7 mm) diameter TD-Ni bar in a square butt configuration with and without interlayers. Joint quality and structural stability of both types of joints were evaluated using metallographic techniques.

*Joint efficiency = (weld strength/base metal strength) x 100.

Materials, Apparatus, and Procedure

Materials

Commercial $^1/_2$ in. (12.7 mm) diameter TD-Ni bar* was used to make both butt and lap joints. This material was in the wrought condition, and it contained elongated pencil-shaped grains parallel to the axis of the bar. The structure was very stable; it could not be recrystallized by heat treatment at 2500° F (1371° C) for 100 hr. Tensile and shear strengths of the $^1/_2$ in. (12.7 mm) diameter bar at room temperature and at 2000° F (1093° C) are shown in Table 1. Note that at 2000° F (1093° C) the shear strength is only one-seventh of the ultimate tensile strength which indicates the directionality of the properties of the bar material.

Table 1—Tensile and Shear Properties of $^1/_2$ in. (12.7 mm) Diameter TD-Nickel Bar

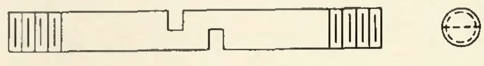
(a) *Tensile data*[a] [*Test atmosphere, air*]

Heat treatment	Test temperature °F	°C	Yield strength ksi	MN/m^2	Ultimate tensile strength ksi	MN/m^2	Elongation, %	Reduction in area, %
2000° F (1093° C) for 2 hr	RT	RT	85.1	586	95.3	656	22	82
None	2000	1093	—	—	22.9	158	[b]4	[b]5
2000° F (1093° C) for 2 hr	2000	1093	—	—	23.3	161	[b]4	[b]5

(b) *Shear data* [*No heat treatment; test atmosphere, air*]

Test temperature °F	°C	Shear strength[c] ksi	MN/m^2
RT	RT	56.0	386
2000	1093	3.3	23

[a] Room-temperature properties represent the average of five tests of butt welds where failure took place in the TD-Ni bar base metal (specimen 1 from Table 3, and specimens 2, 3, 4, and 5 from Table 4).
[b] Data taken from Rice.[1]
[c] Test specimen:



[d] RT—Room temperature.

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Table 2—Criteria for Selection of Materials

Interlayer material	Primary basis for selection
W, Mo, Cb, Ta	Good strength at 2000° F (1093° C) for the interlayer.
Ti, Cr, V, Pd, Fe	Increase in diffusion across the weld interfaces to promote grain growth and eliminate the original weld interface. Also selected to avoid ordered phase formation.
ThO_2	Extra thoria to compensate for possible losses.
TD-Ni sheet	Relatively good strength at 2000° F (1093° C) for the interlayer.
Hastelloy X	Strong Ni-base alloy in sheet form. Good tensile strength to 2000° F (1093° C). Good stress-rupture strength to 1800° F (982° C).
Cobalt-base alloy	Strongest cobalt-base alloy available in sheet form. At 2000° F[a] (1093° C) ultimate tensile strength —25 ksi (172 MN/m²). At 2000° F, (1093° C) stress for rupture in 100 hours —10 ksi (69 MN/m²).

[a] Strength given here is for tests of as-cast material (Ref. 3). This material may be somewhat weaker in sheet form.

Sheet materials of the following commercially-pure metals were used as interlayers in butt joints: 0.001 in. (25 μm) Cb; 0.0003 in. (7.5 μm) Ta; 0.001 in. (25 μm) Mo; 0.001 in. (25μm) W; 0.005 in. (0.13 mm) V; and 0.00025 in. (6 μm) Ti. For one joint, 0.020 in. (0.51 mm) TD-Ni sheet was used. A NASA-developed cobalt base alloy[3] was used in two thicknesses. Hastelloy X, a Ni-base alloy, was also used in two thicknesses. Chemical analyses (wt-%) of the cobalt alloy and Hastelloy X sheet materials were as follows:

*Purchased from E. I. duPont deNemours and Co.

	Hastelloy X[a]	*NASA cobalt-base alloy*
C	0.05	0.34,[b] 0.15[c]
Mn	0.84	—
Si	0.68	—
Cr	21.74	2.8
Ni	Bal.	—
Co	0.80	Bal.
Mo	8.66	—
W	0.14	25.0
Fe	17.71	—
Ti	—	0.9
Zr	—	0.4
Re	—	2.0

[a]0.018 in. (0.46 mm) material.
[b]0.013 in. (0.33 mm) material.
[c]0.004 in. (0.10 mm) material.

The criteria for selecting the above materials and other materials that were applied to the faying (mating) surfaces of the TD-Ni bar were as shown in Table 2.

These interlayer materials were selected with the assumption that strong metallic bonds could be achieved and that the weld interfaces would not be planes of weakness. It was recognized that some interlayer materials would have poor oxidation resistance and considerable differences in coefficient of thermal expansion in comparison to TD-Ni bar. Differential diffusion rates across weld interfaces during a 2000° F (1093° C) for 100 hr heat treatment were expected to produce varying degrees of Kirkendall voids. But the extent and pattern of void formation could not be predicted.

Commercially-pure nickel (Ni 200) bar, 1/2 in. (12.7 mm) diameter, was used in several runs as a substitute for TD-Ni base metal in diffusion studies.

Apparatus

The HIP welding runs were made in a cold-wall autoclave of the type sketched in Fig. 1. The autoclave used in this study can be operated at helium pressures up to 30.0 ksi (207 MN/m²) at temperatures up to 3000° F (1640° C). Equipment of this type has been used for the compaction of ceramics, cermets, and

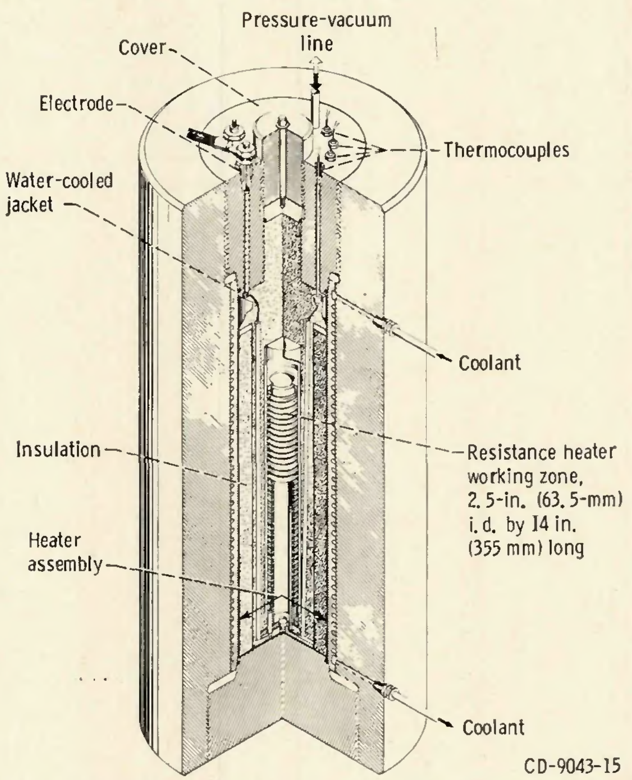


Fig. 1—Cold-wall autoclave used for hot-isostatic-pressure welding

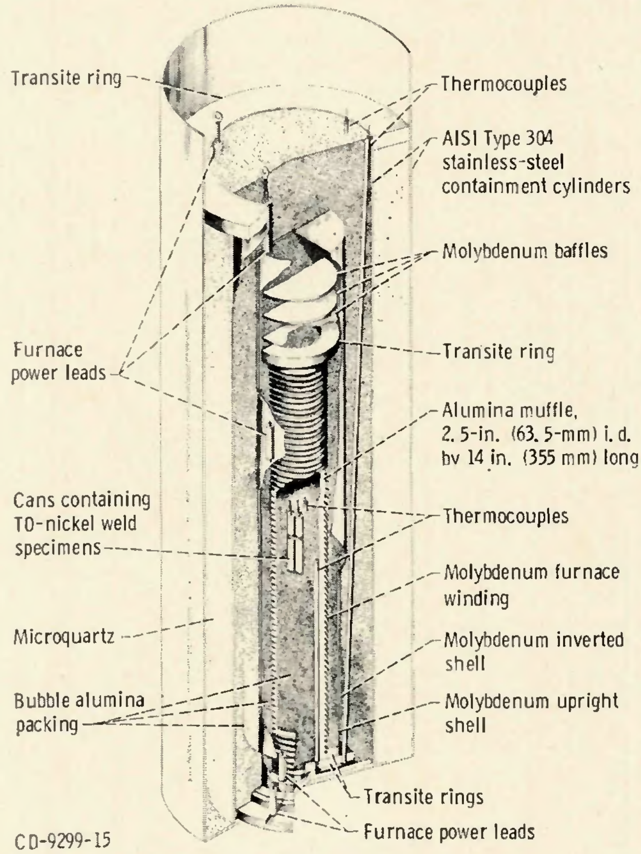


Fig. 2—Heater assembly with specimen location

metals, as well as for the HIP welding of structural members.[4]

The construction of the molybdenum-wound resistance heater assembly within the water-cooled jacket (Fig. 1) is shown in detail in Fig. 2. Microquartz is packed between the two stainless steel cylinders in the heater assembly. The test specimens were surrounded with bubble alumina, and all open areas were packed with bubble alumina to minimize the quantity of helium gas required.

Pt–6 wt-% Rh/Pt–30 wt-% Rh thermocouples were used to monitor the temperature in this program. An inverted molybdenum shell placed over the open upright molybdenum shell, and molybdenum baffles, effectively minimized thermal gradients in the furnace—Fig. 2. The inverted shell and baffles also served to prevent overheating of the autoclave cover.

Welding Procedure

General Procedure. A schematic sketch presented in Fig. 3 shows the setup for HIP butt welding two $^1/_2$ in. (12.7 mm) diameter x $1^1/_2$ in. (38.1 mm) long specimens to produce a 3 in. (76.2 mm) long weldment. AISI Type 304 stainless steel wire was wrapped around the TD-Ni specimens to minimize offset at the weld joint in the slightly oversize Type 304 stainless steel cans. After the TD-Ni specimens were placed in the can, the lid with the evacuation tube was gas tungsten-arc welded to the can in an argon-filled welding chamber. An electron beam welding machine was used for evacuation and tube closure. Prior to making the electron beam closure weld, the can was heated to 600° F (316° C) and held at 2×10^{-5} torr (2.7×10^{-3} N/m^2) for ½ hr in the electron beam welding chamber.

After a helium leak test, the sealed cans were placed in the cold-wall autoclave (as shown in Fig. 2) and exposed to the HIP welding cycle shown in Fig. 4. Time for the entire run was about 12 hr, during which the peak temperature and pressure of 2000° F (1093° C) and 20 ksi (138 MN/m^2) helium were maintained for 2 hr. Typical appearance of the cans after exposure to the HIP welding cycle is shown in Fig. 5. The TD-Ni

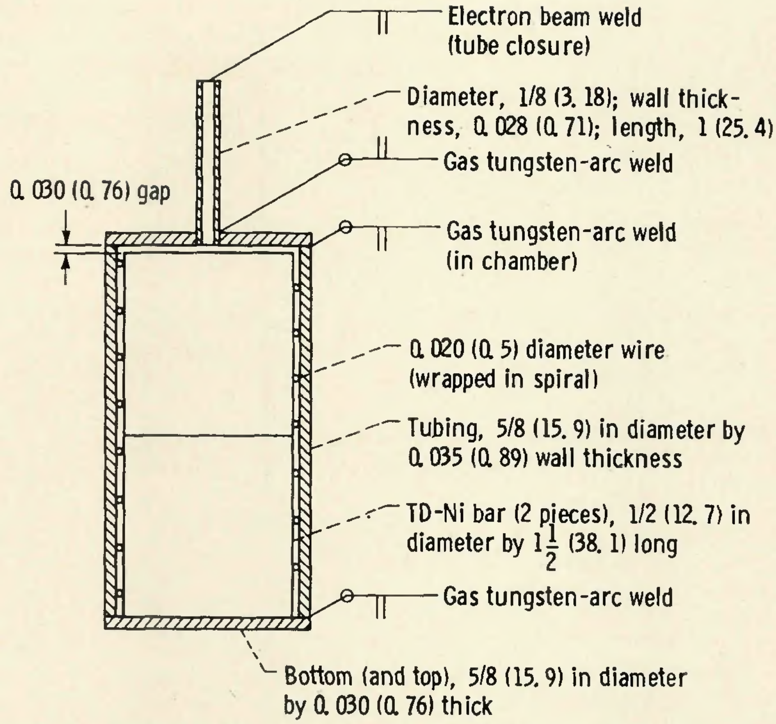


Fig. 3—Schematic of ½ in. (12.7 mm) diameter TD-nickel bar specimens in AISI Type 304 stainless-steel cans. Specimens are wrapped with Type 304 stainless-steel wire to minimize misalignment of the specimens at the butt joint. (Dimensions are in inches (mm))

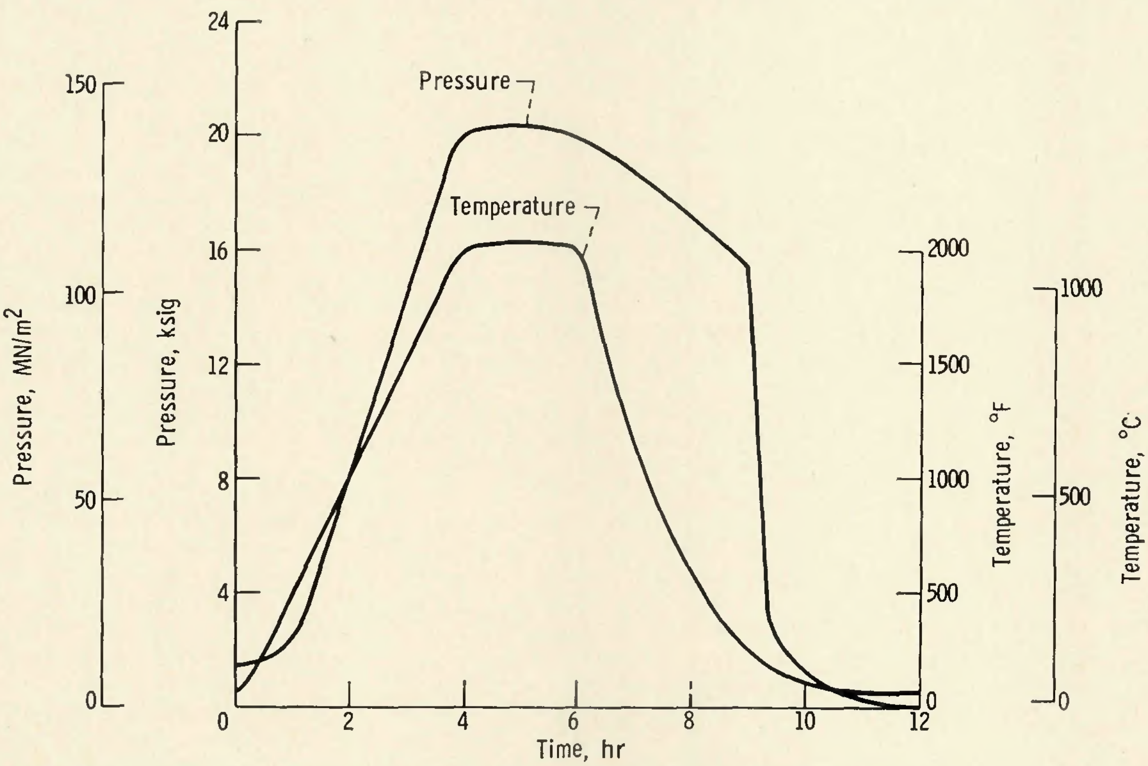


Fig. 4—Typical pressure and temperature profiles for hot-isostatic-pressure (HIP welding run)

Fig. 5—AISI Type 304 stainless-steel can (approx. 1 cm diameter) containing TD-nickel weldments after exposure to hot isostatic-pressure (HIP) welding cycle. (The imprint of the wire on the can and the flattening of the evacuation tube attest to the fact that HIP welding of the specimens was achieved)

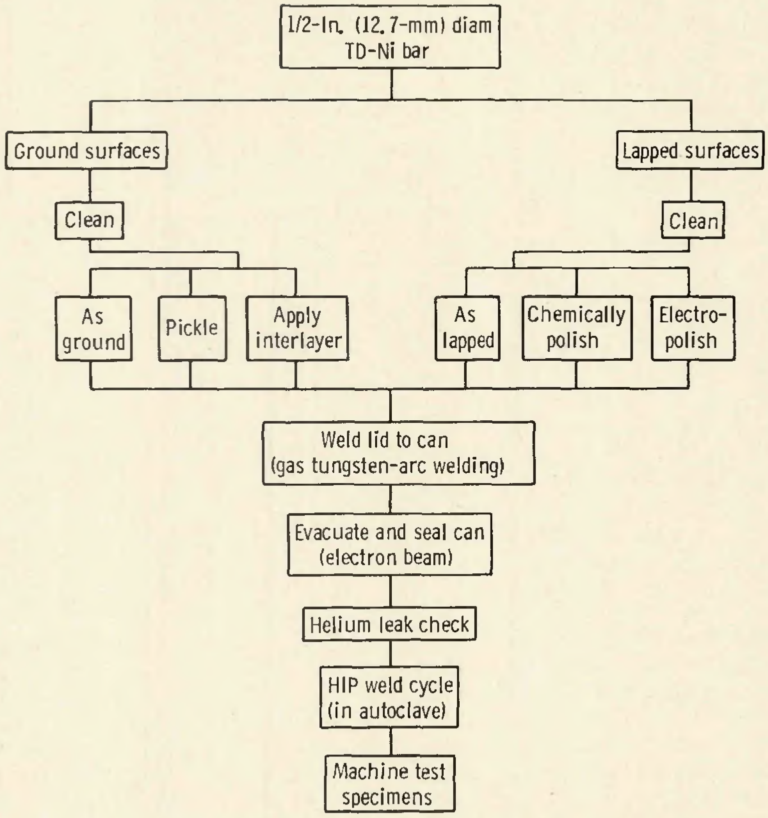


Fig. 6—Flow diagram for hot-isostatic-pressure (HIP) welding TD-nickel bar

specimens inside the can were welded to each other and to the can. Note that the imprint of the wire that was wrapped around the TD-Ni specimens shows through the can. Also note that the evacuation tube was flattened.

Surface Finish for Butt Joints. Several faying surface preparation procedures were used in a cursory study of surface effects on microstructure and properties of HIP welds. The flow diagram in Fig. 6 shows that both as-ground, 16 rms (40.7×10^{-6} cm rms), and as-lapped, 4 rms (10.1×10^{-6} cm rms), surface finishes were used. The TD-Ni specimens were ultrasonically cleaned in freon, then in a detergent, followed by a distilled water rinse. And the stainless steel cans were cleaned with a light acid etch, and rinsed in distilled water. In further faying surface preparation, (see Fig. 6), some groups of TD-Ni specimens were treated as follows:

1. Pickle: Immerse specimens in a 125° F (52° C) solution (300 ml HNO_3, 50 ml HF, 1000 ml H_2O) for 10 min. Rinse in distilled water.
2. Chemically polish: Immerse specimens in a 170° F (77° C) solution (42.5 ml H_3PO_4, 42.5 ml CH_3CO_2H, 15 ml HNO_3, 3 ml H_2SO_4, 15 ml H_2O) for 10 min. Rinse in a solution of 5% ammonium hydroxide in distilled water.
3. Electropolish: Polish specimen in a 30 ml HNO_3, 70 ml CH_3OH solution at room temperature for 2½ min at $7^1/_2$ v.

Interlayers for Butt Joints. For some other butt joints, a metal interlayer in the form of thin sheet material was cleaned in acetone and placed between as-ground faying surfaces. For still other butt joints as-ground faying surfaces were clad with various pure metals using vapor plating or electron beam evaporation techniques. Thoria powder was used as an interlayer for one butt joint with as-ground faying surface.

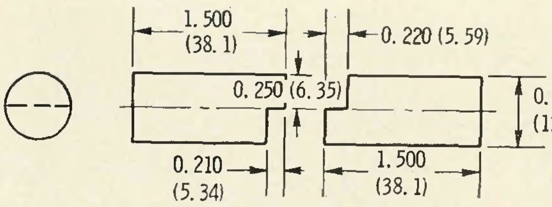
The balance of the procedures outlined in Fig. 6 was followed after the TD-Ni welding specimens were placed in the can.

Lap Joints. In addition to the butt joints, some work was done in the HIP welding of lap joints using similar procedures except that the lap joint had as-milled 32 rms (81.3×10^{-6} cm rms) faying surfaces. Design of the lap joint that was subsequently tested in shear is shown in Fig. 7.

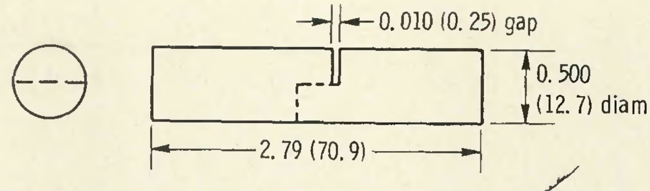
Testing Procedure

Tensile and stress-rupture testing were conducted on the welded specimens in the as-welded and in the heat-treated conditions. Heat treatment involved exposure at 2000° F (1093° C) for 100 hr in argon. Some specimens were heat treated while inside (and still welded to) the stainless steel can. Others were heat treated after the can had been removed by machining. Tensile tests were conducted using a button-head specimen, 2 in. long (50.8 mm) with a 0.160 in. (4.06 mm) diameter × 1 in. (25.4 mm) gage length. The 3 in. (76.2 mm) long stress-rupture specimens had threaded ends and a 0.250 in. (6.3 mm) diameter × 1 in. (25.4 mm) gage length.

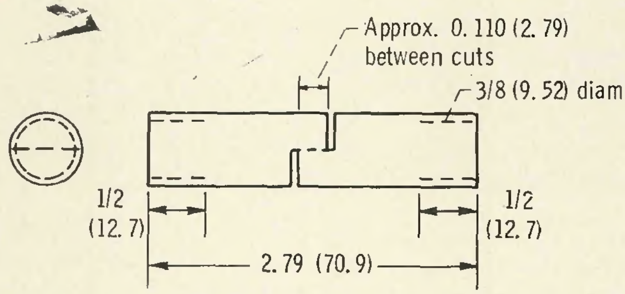
Design of the lap joint specimen was previously shown in Fig. 7. The lap joint was designed to be tested in shear upon the application of a tensile load. Although pure shear was not achieved, there was only slight joint rotation because of the low shear strength and low shear ductility of the TD-Ni bar. All of the above test specimens were machined from the central portion of the $^1/_2$ in. (12.7 mm) diameter TD-Ni bar with the joints at mid-length. Unwelded base metal specimens were machined to obtain comparative test data.



(a) Mating pieces for lap joints.



(b) Setup for hot-isostatic-pressure welding.



(c) Machined for shear test of lap joint. Area in shear = 0.110 x 0.375 = 0.041 sq in. (26.6 sq mm).

Fig. 7—Design of lap joint shear test specimen. (Dimensions are in inches (mm))

Tensile tests were run in air and in vacuum (5×10^{-5} torr, 6.6×10^{-3} N/m^2). Vacuum testing was used primarily to prevent oxidation of reactive and refractory metal interlayers. One tensile test was run in argon in order to prevent oxidation of a tungsten interlayer. The short-time tensile and shear specimens, tested at 2000° F (1093° C), were held at temperature for 5 min prior to the application of load. For all tensile and shear tests, a cross-head speed of 0.05 in (1.3 mm) per minute was maintained. Stress-rupture testing was conducted at 2000° F (1093° C) in helium for two reasons. First of all, the helium atmosphere eliminated possible stress-oxidation effects for joints with interlayers. Secondly, fracture path and location can more readily be studied if the fracture surfaces are not oxidized.

Results and Discussion

Effect of Surface Finish on Butt Joints

Microstructure. The microstructures of HIP butt welds in the TD-Ni bar with the various surface finishes studied are shown in Fig. 8. No unwelded regions were found during examination of unetched specimens at up to X500 magnification. Thus, these HIP welds were judged to be sound. Varying amounts of recrystallization can be observed in Fig. 8. Two factors are believed to influence the extent of recrystallization that occurs during HIP welding.

1. Surface flatness.
2. Amount of residual cold work produced in surface preparation.

Thus, joints with ground surfaces had the most recrystallization—Fig. 8(a). Microhardness in the recrystallized region of this joint was 238 DPH compared to 254 DPH in the wrought structure. Lapped surfaces produced a fine-grained recrystallization zone—Fig. 8(b), and lapped-plus-chemically-polished surfaces gave only localized areas of recrystallization—Fig. 8(c). Once formed, these microstructures appear to be extremely stable as evaluated by light microscopy. Heat treatment at 2000° F (1093° C) for 100 hr showed no apparent effect.

However, electron microscopy (Fig. 9(a)) and electron microprobe X-ray raster micrograph examination (Fig. 9(b)) of the joint with as-ground surfaces showed thoria depletion in the recrystallized region. Some recrystallized grains appeared to have a normal dispersion; others appeared to be thoria-free (Fig. 9(a)). These changes in thoria distribution were confirmed by electron microprobe line scan and spectral scan. It would appear, therefore, that recrystallization promotes thoria movement resulting in areas of depletion and agglomeration. As-welded joints showed lesser thoria depletion and agglomeration than heat-treated joints.

Loss of the uniform thoria dispersion and loss of the original texture are believed to weaken the TD-Ni material, especially at elevated temperatures. In addition, since elevated temperature fracture occurs by an intergranular mechanism,[5] reduced tensile strength would be expected for tests normal to the butt welds.

Properties. The results of mechanical tests of HIP weldments with various surface finishes studied are shown in Table 3. In the room temperature tensile test of a specimen with ground surfaces, failure took place in the base metal away from the joint. This showed that at room temperature the HIP weld is stronger than the base metal. This confirms reports from the literature[2, 6] that solid-state welds in

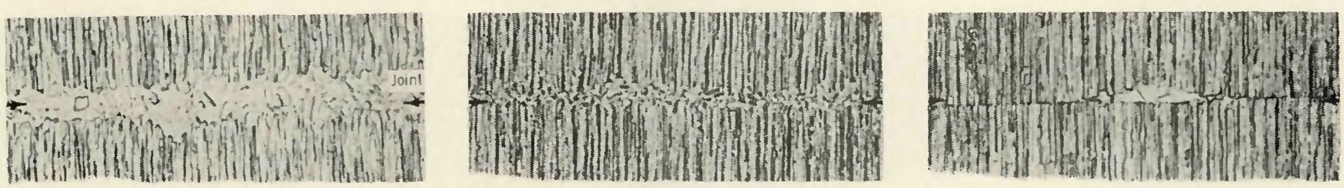


Fig. 8—Effect of surface finish on microstructure of hot-isostatic-pressure weldments in ½ in. (12.7 mm) diameter TD-nickel bar. Etchant: 92 milliliters HC1, 3 milliliters HNO_3, 5 milliliters H_2SO_4 (swab). Left (a)—ground surfaces, heat treated for 100 hr at 2000° F (1093° C); center (b)—lapped surfaces, as-welded; right (c)—lapped plus chemically polished surfaces, as-welded. Etched. X500 (reduced 28% on reproduction)

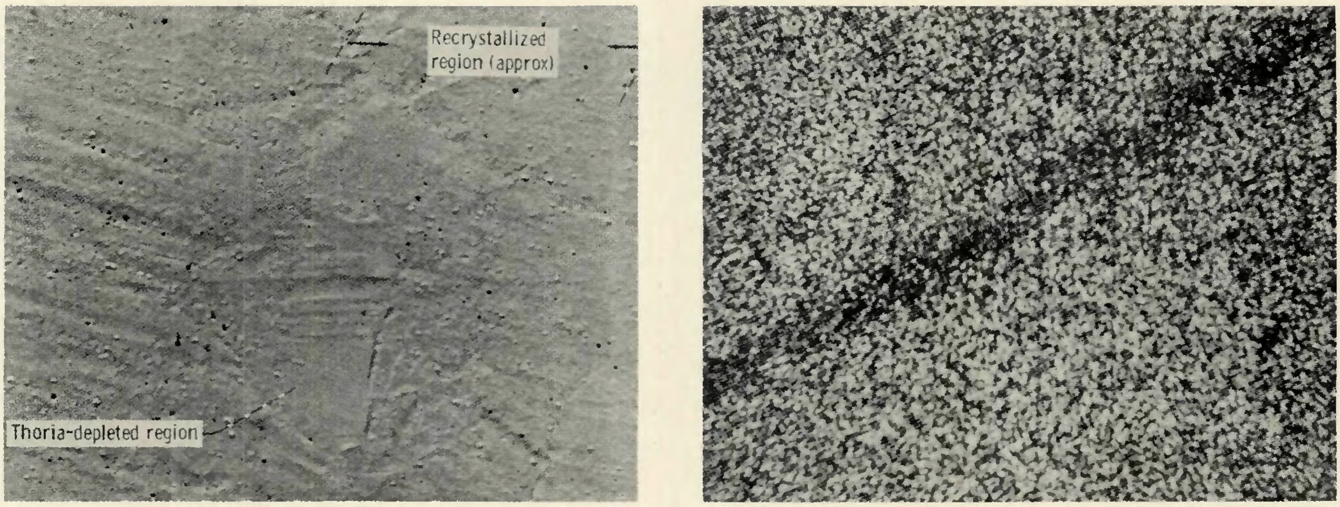


Fig. 9—Regions of thoria depletion and normal distribution in recrystallized region of heat-treated TD-nickel bar hot-isostatic-pressure (HIP) butt weld shown in Fig. 8(a). Etchant: electrolytic using 2 volts and solution of 30 milliliters H_2O, 30 milliliters H_2SO_4, and 40 milliliters H_3PO_4. Left (a)—electron photomicrograph of replica taken from recrystallized region of HIP butt weld in TD-nickel bar with wrought base metal structure on either side. Regions of thoria depletion and normal distribution as compared to base metal, are shown. Etched; X9500. Right (b)—electron microprobe X-ray raster micrograph of thorium at recrystallized region (dark band) shown in Figs. 8(a) and 9(a). Low resultant thorium concentration shown is due to presence of thoria-depleted and normal distribution regions. Unetched; X2000

TD-Ni are strong at room temperature.

Tensile testing at 2000° F (1093° C) gave quite a different story. Joint efficiency varied from 40 to 54% using comparative base metal strength data from Table 1. All weldments failed at the joint with a square-edge fracture. For all 2000° F (1093° C) tensile fractures, there was no measurable percent elongation or reduction of area. The fracture path for the as-ground specimen (no. 1) tested in air at 2000° F (1093° C) occurred partly through the recrystallized grain boundaries and partly at the junction between recrystallized and wrought base metal—Fig. 10. A large portion of the fracture took place among the original weld interface.

One of the 2000° F (1093° C) tensile tests (no. 4, Table 3) was run in vacuum and in three cases the HIP weldments were heat treated prior to tensile testing. But these variations had little effect on 2000° F (1093° C) tensile strength.

One 2000° F (1093° C) stress-rupture test (in helium) of a specimen with ground surfaces gave very poor stress-rupture strength. Joint failure occurred in 0.2 hr at a stress of 3 ksi (21 MN/m^2). In comparison, unwelded base metal can sustain a stress of 11 ksi (76 MN/m^2) for 100 hr at 2000° F (1093° C).[7]

Discussion. Various faying surface finishes on TD-Nickel bar were found to produce varying degrees of recrystallization at HIP butt welded joints. This recrystallization was accompanied by loss of the thoria dispersion, and, in no case, was it possible to retain the original microstructure at the joint. At room temperature the HIP weld was stronger than the base metal. But at 2000° F (1093° C) the HIP weld was weak in both tensile and stress-rupture tests.

Effects of Interlayers on Butt Joints

Microstructure. The microstructures of the two most promising butt joints containing interlayers are shown in Fig. 11 in the heat treated condition. In the as-welded condition, no voids were observed. However, during the 2000° F (1093° C) /100 hr test treatment, Kirkendall voids developed sporatically in the TD-Ni bar near the cobalt-alloy interleaf—Fig. 11(a). For the joint with the Hastelloy X interlayer, a few Kirkendall voids formed just inside the interlayer. Small voids of this type are shown in Fig. 11(b). Microprobe and hardness data, shown in Fig. 11, indicate the extent of diffusion that has taken place across the weld interfaces.

In the cobalt alloy/TD-Ni joint (Fig. 11(a)), nickel has diffused into the cobalt alloy from the TD-Ni faster than the interlayer elements have diffused into the TD-Ni. This results in porosity formation in the TD-Ni. Apparently the stable, elongated TD-Ni bar grain boundaries offer high diffusion rate paths during heat treatment. Similar cobalt alloy/Ni 200 joints did not exhibit porosity after the same heat treatment. For both the cobalt alloy/TD-Ni and the cobalt alloy/Ni 200 joints, the interlayer elements were restrained from taking advantage of the grain boundary diffusion if they were tied up as carbides and other compounds.

A small amount (0.3–0.5 wt-%) of Th was present in the cobalt-alloy interleaf. That was indicated by using the spectral scan technique on a cobalt alloy/Ni 200 weldment and comparing it to the cobalt alloy/TD-Ni weldment. Also, ThO_2 depletion has occurred 50–100 μ into the TD-Ni from the weld interface—Fig. 11(a). The Th-depleted region will not be as strong as the base metal.

Heat treatment moves the hardness gradient well inside the interlayer and away from the weld interface as seen in Fig. 11(a). This movement of the hardness gradient by virtue of diffusion increases joint strength as described by Kharchenko.[8]

In the Hastelloy X/TD-Ni joint (Fig. 11(b)), the interlayer elements diffuse into the TD-Ni slightly faster than Ni diffuses into the interlayer. The molybdenum in the Hastelloy X interlayer formed carbides and a compound that was rich in Si. It is felt that the formation of these compounds prevented Mo diffusion out of the interleaf rather than a decreased diffusion rate of Mo.

A joint in a TD-Ni bar with a 0.001 in. (25 μm) Mo interlayer is shown in Fig. 12(a) along with microprobe analysis and hardness determinations. A similar HIP weld with Ni 200 base metal and a Mo interlayer is presented in Fig. 12(b) in order to illustrate relative diffusion effects in the different base metals. Kirkendall voids are evident in the TD-Ni base metal (Fig. 12(a)), because Ni has diffused into

Table 3—Tensile and Stress-Rupture Data for HIP Butt Weldments With Various Surface Finishes

(a) *Room-temperature tensile test:*

Specimen	Surface preparation	Ultimate tensile strength, ksi	Ultimate tensile strength, MN/m²	Elongation, %	Reduction in area, %	Fracture location
1	As-ground	95.0	655	18	63	Base metal

(b) *2000° F (1093° C) tensile tests:*

Specimen	Surface preparation	Postweld heat treatment	Test atmosphere	Ultimate tensile strength[a], ksi	Ultimate tensile strength[a], MN/m²	Joint[b] efficiency, %
1	Ground	None	Air	12.1	83	52
2	Lapped	2000° F (1093° C) for 100 hr[c]	Air	11.1	76	48
3	Lapped and electropolished	2000° F (1093° C) for 100 hr[c]	Air	9.3	64	40
4	Ground and pickled	None	Vacuum	10.3	71	45
5	Lapped and chemically polished	2000° F (1093° C) for 100 hr[c]	Air	12.5	86	54

(c) *2000° F (1093° C) stress-rupture test:*

Specimen	Surface preparation	Postwelding heat treatment	Test atmosphere	Stress, ksi	Stress, MN/m²	Life, hr[a]
1	Ground	Outgassed at 2200° F (1204° C) for 1/2 hr in vacuum (with can removed)	Helium	3	21	0.2

[a] Square-edge fracture at the joint with <1 percent elongation.

[b] Joint efficiency = $\frac{\text{Weld joint strength}}{\text{Base metal strength}} \times 100$.

[c] Heat treated in the can in an argon atmosphere.

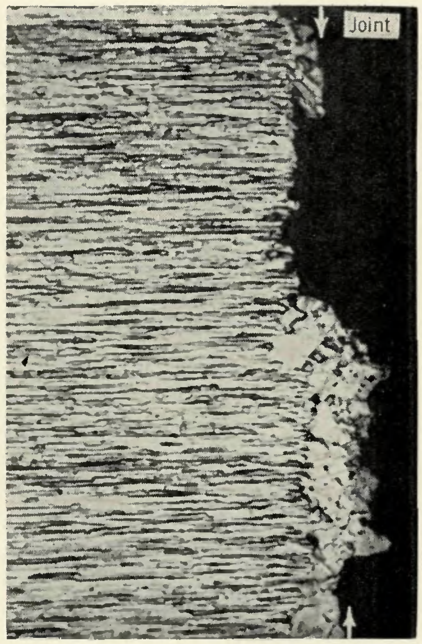


Fig. 10—Fracture of hot-isostatic-pressure butt weldment (as ground surfaces) in 2000° F (1093° C) tensile test (specimen 1, Table 4). Etchant: 92 milliliters HCl, 3 milliliters HNO_3, 5 milliliters H_2SO_4 (swab). X500

the Mo interlayer faster than the Mo diffused into the TD-Ni. However, most of the Mo has diffused out of the original interlayer, producing an alloy of about 97.5% Ni, 2.5% Mo.

The Mo diffusion into the base metal is believed to have taken place primarily along the TD-Ni grain boundaries. So, by comparing the Mo/TD-Ni joint (Fig. 12(a)) to the Hastelloy X/TD-Ni joint (Fig. 11(b)), it is verified that Mo compound formation is the reason for the immobility of the Mo in the Hastelloy X/TD-Ni joint and not the relatively large size of the Mo atom. In addition, radically different diffusion effects are also noted in microprobe analysis and hardness of the Mo-interlayer joint in TD-Ni compared to that found for a Mo-interlayer joint in Ni 200 base metal. For the Mo-Ni 200 joint, the Mo was essentially unable to move into the Ni 200 because only a minimum number of grain boundaries are available as diffusion paths—Fig. 12(b). On the other hand, nearly all the Mo diffused along the many grain boundaries of the TD-Ni—Fig. 12(a).

The decrease in the hardness gradient (Fig. 12(b)) and the diffusion of the Mo into TD-Ni was accompanied by a strengthening effect at 2000° F (1093° C) as will be shown.

Properties. The results of room temperature tensile tests on HIP-welded butt joints with several interlayers are shown in Table 4. The joints with cobalt alloy and Hastelloy X interlayers were stronger than the base metal since fracture took place away from the joints. Elongation was 20 to 26% for the cobalt alloy and Hastelloy X joints. Brittle fracture took place at the joint with the 0.001 in. (25 μm) W interlayer; joint efficiency was 85%.

Table 5 shows the 2000° F (1093° C) tensile test results for joints with interlayers between ground faying surfaces. Note that square-edge fracture at the joint with <1% elongation was characteristic of all tests. All of the weldments listed in Table 5 were free of voids as determined by light microscopy in the as-welded condition. Other interlayers tried, that produced unsound joints (not listed in Table 5), were 0.001 in. (25 μm) Cb and 0.005 in. (0.12 mm) vanadium joints which cracked during HIP welding and an electron beam evaporated Cr joint which had entrapped oxides at the weld line.

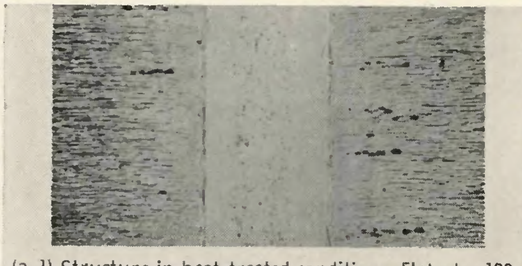
After postweld heat treatment (2000° F (1093° C) for 100 hr) Kirkendall voids, in varying degrees, were found in all joints listed in Table 5 except for the ThO_2-coated joints (specimens 24, 31, 32, and 36).

A graphic presentation showing tensile strength data for the stronger joints with interlayers and the base metal is shown in Fig. 13. Heat treatment produced a strengthening effect for joints with cobalt-alloy, Hastelloy X, and Mo interlayers even though some Kirkendall voids were inherently produced. The exception for this strengthening effect was one specimen with a 0.004 inch (0.10 mm) cobalt-alloy interlayer. This specimen (no. 11 from Table 5), which was heat treated after first removing the can had lower strength than the as-welded specimen (no. 10). Other specimens (nos. 12 and 13) with the 0.004 in. (0.10 mm) cobalt-alloy interlayer that were heat treated in the Type 304 stainless steel can were much stronger than the as-welded specimen (no. 10) whether subsequent tensile testing was conducted in air (94% joint efficiency) or in vacuum (99% joint efficiency).

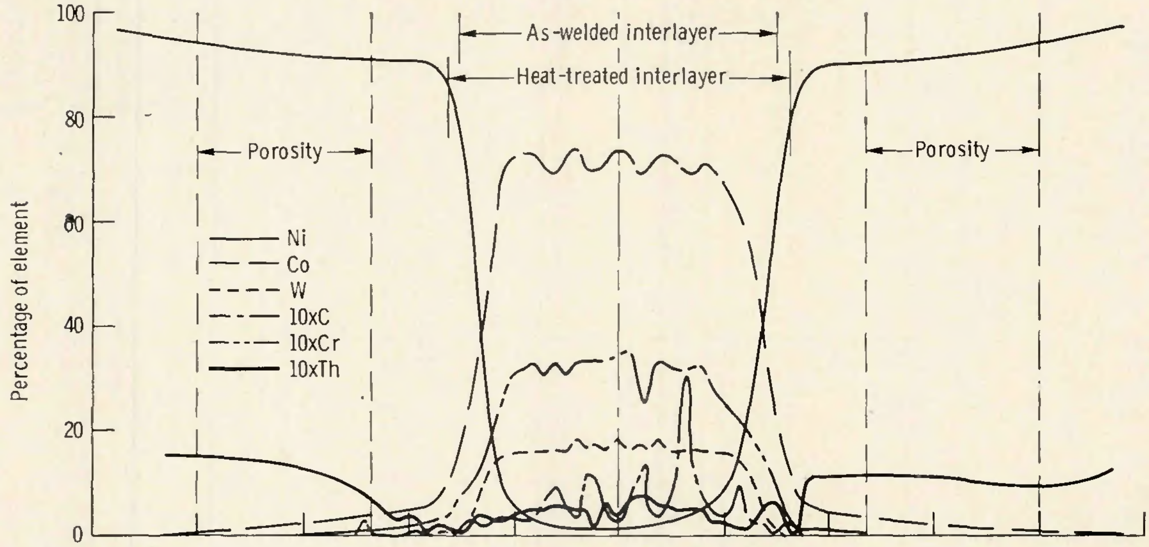
Examination of the strength data (Table 5) for tests of joints with 0.018 in. (0.46 mm) Hastelloy X interlayers indicated that test atmosphere had a pronounced effect on 2000° F (1093° C) tensile strength—Fig. 13. Specimen no. 15, heat treated with the can removed and tested in air, gave 62% joint efficiency; specimen no. 16, tested in vacuum, gave 87% joint efficiency. Similar effects can be noted for joints with the 0.002 in. (51 μm) Hastelloy X interlayer. These data, therefore, suggest that a stress-oxidation effect may reduce 2000° F (1093° C) tensile strength of joints with Hastelloy X interlayers.

For joints with a 0.001 in. (25 μm) Mo interlayer heat treatment in the can increased joint efficiency to 61% compared to 48% as-welded.

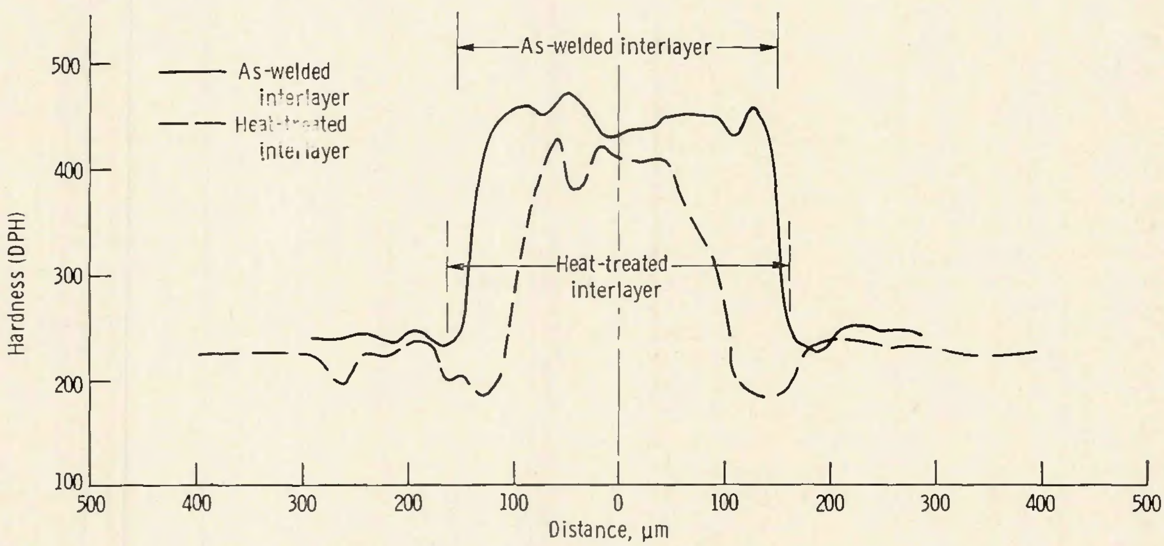
Figure 14 shows the mode of fracture for 2000° F (1093° C) tensile tests of butt joints with cobalt-alloy, Hastelloy X, and Mo interlayers. For the cobalt-alloy (Fig. 14(a)), and Hastelloy X (Fig. 14(b)) joint fracture took place at the interfaces between the interlayer and the parent material. Fracture for the joint with the Mo interlayer took place partly through the interlayer and partly at



(a-1) Structure in heat-treated condition. Etched; x100.



(a-2) Chemistry in heat-treated condition.



(a-3) Hardness in as-welded and heat-treated conditions.

(a) 0.013-Inch (0.33-mm) cobalt-alloy interlayer.

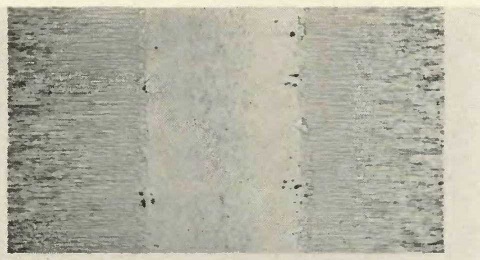
Fig. 11—Microstructure, microprobe chemistry traverses, and microhardness traverses of hot-isostatic-pressure (HIP) butt welds in ½ in. (12.7 mm) TD-nickel with cobalt alloy and Hastelloy X interlayers. Etchant, 92 milliliters HCl, 3 milliliters HNO_3, 5 milliliters H_2SO_4 (swab). Continued on next page

the interlayer-base metal interface. Actually the composition of the "Mo" interlayer after the 2000° F (1093° C) for 100-hr heat treatment was about 97.5% Ni–2.5% Mo (Fig. 12(a)).

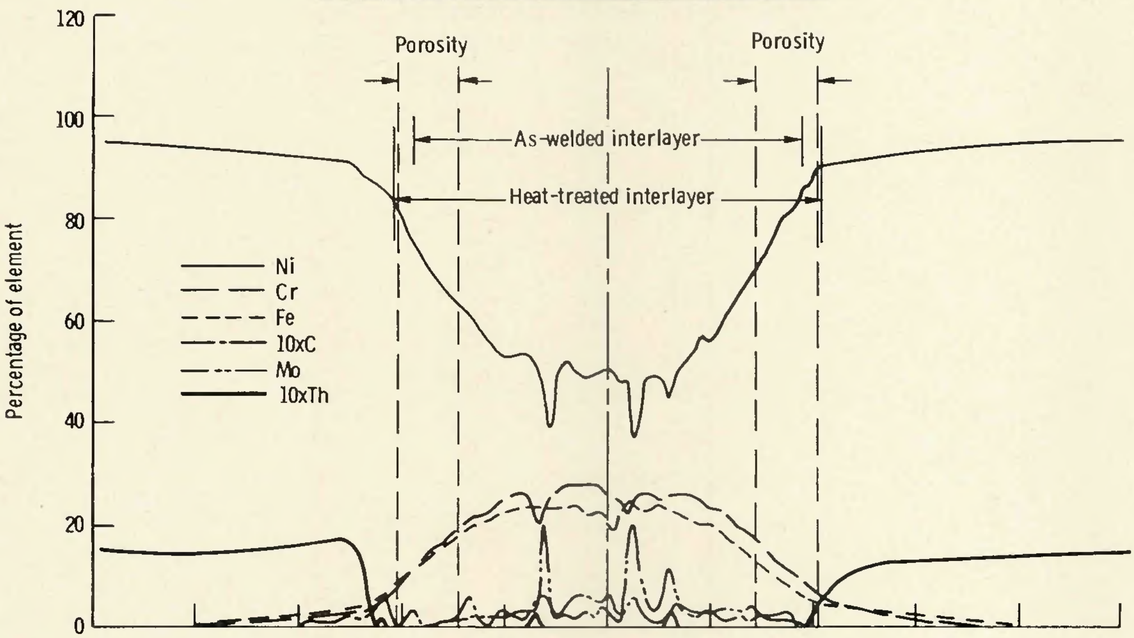
Stress-rupture tests were conducted at 2000° F (1093° C) in helium on the joints with cobalt-alloy and Hastelloy X interlayers that were highly efficient in 2000° F (1093° C) tensile tests. These stress-rupture data are shown in Table 6. All weldments were heat treated prior to stress-rupture testing—Table 6.

From the stress-rupture plot of these data shown as Fig. 15, it is evident that the weld joints are much weaker than the base metal. For instance, the stress for rupture in 100 hr is about 11 ksi (75.8 MN/m^2) for the base metal[7] and 1.7 ksi (11.7 MN/m^2) for the strongest joints (0.004 in. (0.10 mm) cobalt-alloy interlayers). This is only 15% joint efficiency where stress-rupture joint efficiency is defined as the ratio of weld joint to base metal strength for 100 hr life at 2000° F (1093° C). All the weldments exhibited a characteristic square-edge fracture at the joint with <1% elongation.

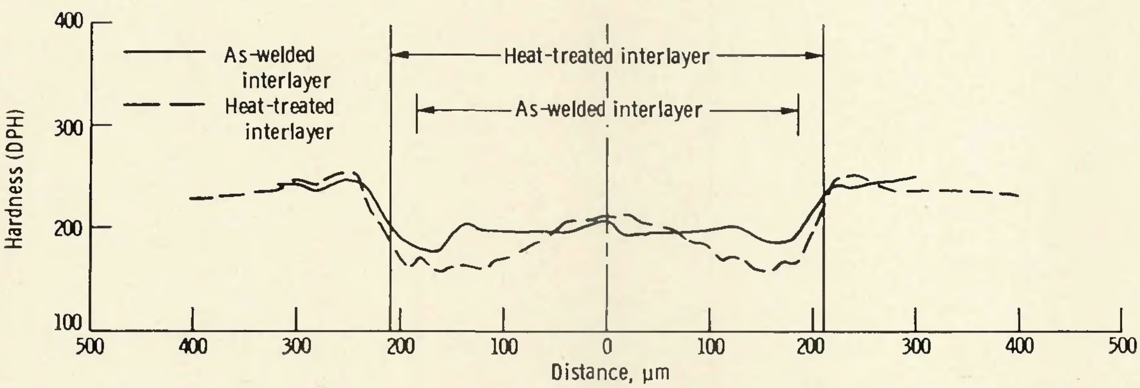
In regard to the mode of stress-rupture fracture, the joints with cobalt-alloy interlayers failed at the cobalt alloy-base metal interface. In no case did failure occur within the interlayer material. Loss of the uniform ThO_2 dispersion and loss of texture are probable factors in weakening the TD-Ni bar very near the weld interface. Shear stress produced by differ-



(b-1) Structure in heat-treated condition. Etched. x100.



(b-2) Chemistry in heat-treated condition.

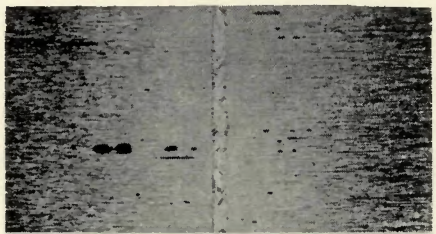


(b-3) Hardness in as-welded and heat-treated conditions.

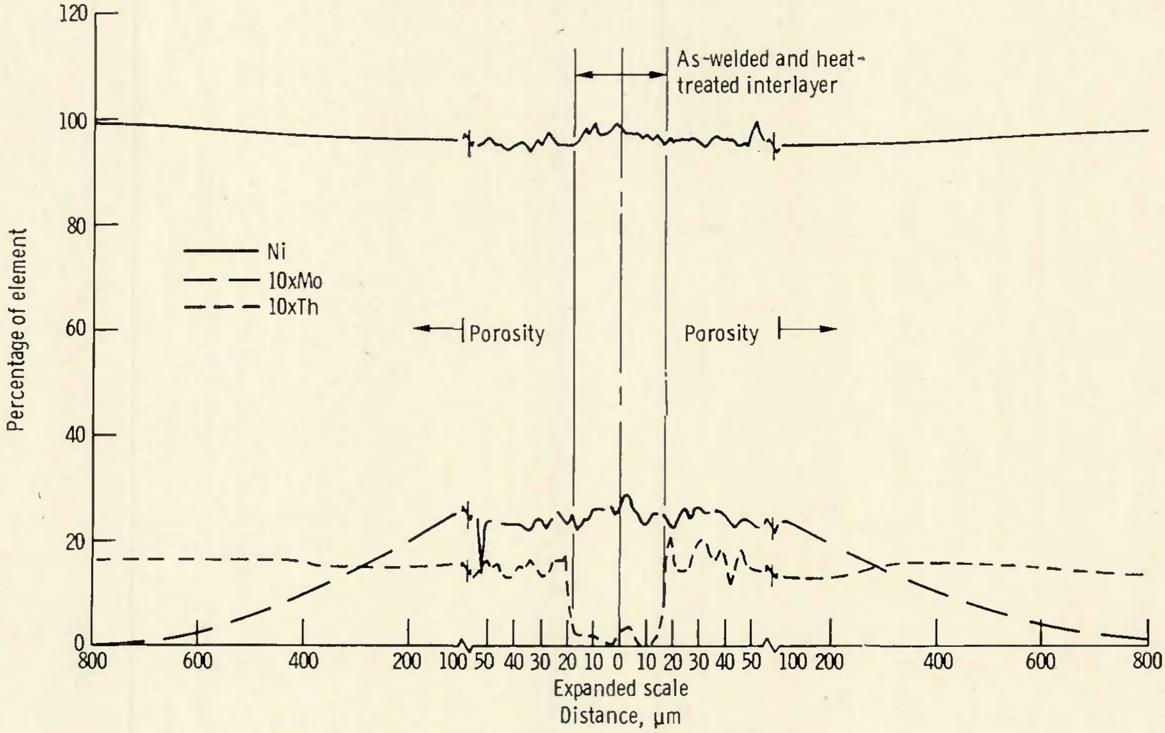
(b) 0.018-Inch (0.43-mm) Hastelloy X interlayer.

Fig. 11—(Continued)

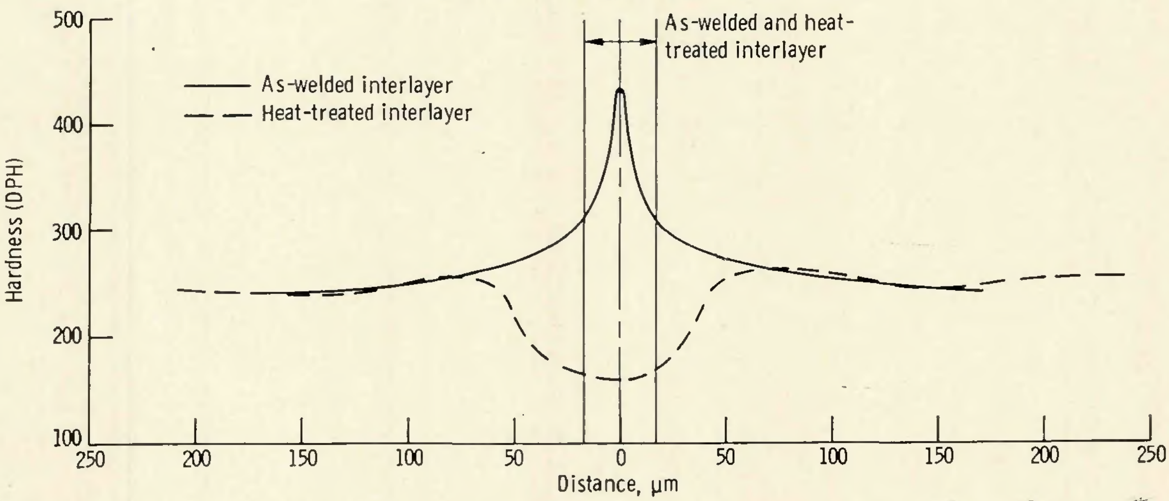
ential thermal expansion is also believed to contribute to a weakening effect at the weld interface. Data published by Freche et al.[3] indicate that the cobalt-alloy interlayer can at 2000° F (1093° C) support a stress of 10 ksi (689 MN/m²) for about 100 hours in the as-cast condition. If the cobalt alloy in sheet form has similar strength



(a-1) Structure in heat-treated condition. Etched; x100.



(a-2) Chemistry in heat-treated conditions.



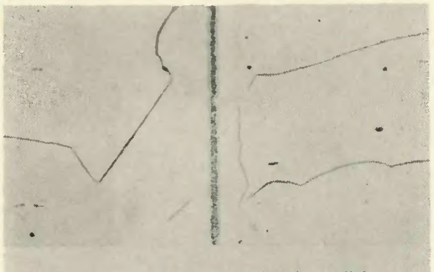
(a-3) Hardness in as-welded and heat-treated conditions.

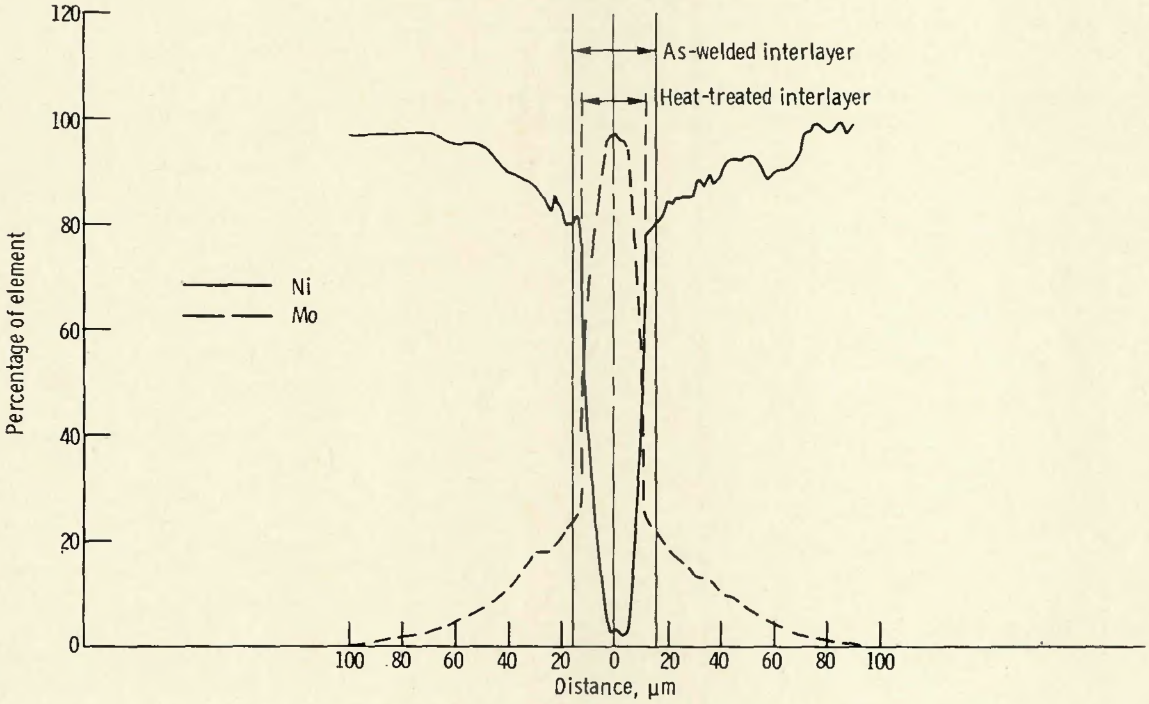
(a) 0.001-Inch (25-µm) molybdenum interlayer and TD-Ni parent material. Etchant, 92 milliliters HCl, 3 milliliters HNO_3, 5 milliliters H_2SO_4 (swab).

Fig. 12—Microstructure, microprobe chemistry traverses, and microhardness traverses of hot-isostatic-pressure (HIP) butt welds in ½ in. (12.7 mm) TD-nickel and nickel 200 bars with 0.001 in. (25 µm) molybdenum interlayers. Continued on next page

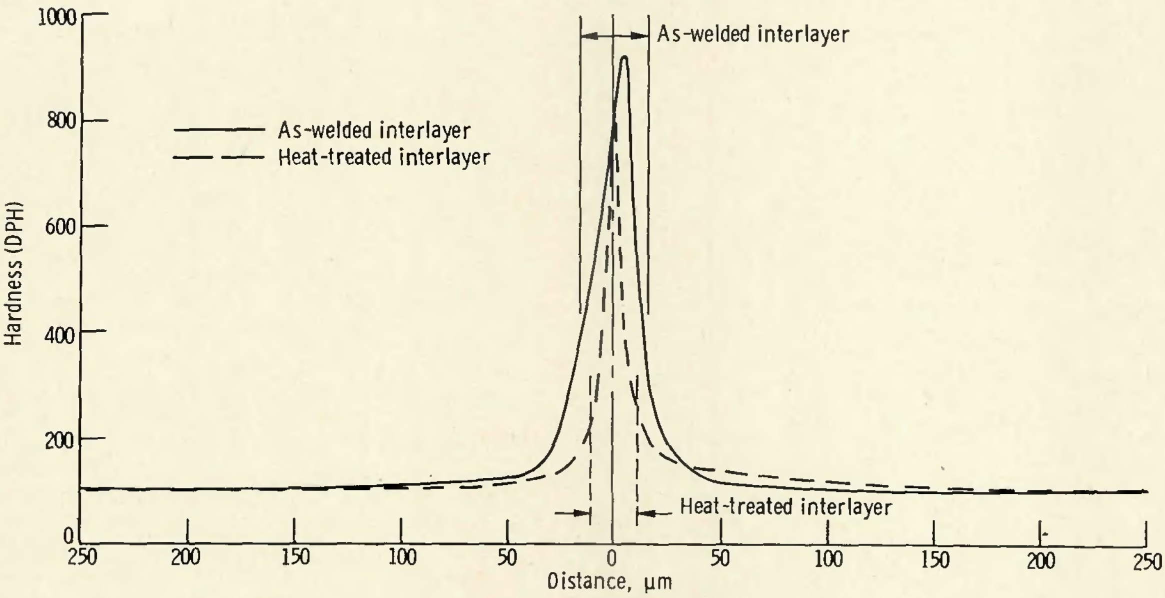
it would nearly match the TD-Ni bar in stress-rupture strength. The weak link, however, is the weld interface.

With the Hastelloy X interlayer, the weld interface was also a plane of weakness—Fig. 14(b). But since the Hastelloy X material is weak in stress-rupture above about 1800° F (982° C), fracture in the 2000° F (1093°


(b-1) Structure in heat-treated condition. Etched; x100.



(b-2) Chemistry in heat-treated condition (no thorium).



(b-3) Hardness in as-welded and heat-treated conditions.

Fig. 12—(Continued)

Table 4—Room-Temperature Tensile Data for HIP Butt Weldments with Interlayers[a]

Specimen no.	Interlayer	Yield strength ksi	Yield strength MN/m²	Ultimate tensile strength ksi	Ultimate tensile strength MN/m²	Elongation, %	Reduction in area, %	Fracture location
1	0.001 in. (25 μm) tungsten	—	—	81.4	560	0	0	At joint
2	0.013 in. (0.33 mm) cobalt alloy	80.8	556	94.9	654	26	81	Base metal
3	0.018 in.(0.46 mm) Hastelloy X	85.7	590	95.1	655	20	81	
4	0.004 in. (0.10 mm) cobalt-alloy	90.6	625	95.7	660	21	82	
5	0.002 in. (51 μm) Hastelloy X	83.2	574	95.9	660	24	81	

[a] Welds made with as-ground surfaces; no postweld heat treatment.

Table 5—2000° F (1093° C) Tensile Data for HIP Butt Weldments with Interlayers

Specimen no.	Interlayer	Post-weld heat treatment[a]	Test atmosphere	Ultimate tensile strength[b] ksi	Ultimate tensile strength[b] MN/m²	Joint efficiency,[c] %
6	0.013 in. (0.33 mm) cobalt alloy	None	Air	13.0	90	56
7		HT1	Air	14.6	101	63
8		HT2	Vacuum	23.8	164	100
9		HT2	Vacuum	17.0	117	74
10	0.004 in. (0.10 mm) cobalt alloy	None	Air	15.3	105	66
11		HT1	Air	14.4	99	62
12		HT2	Vacuum	22.8	157	99
13		HT2	Air	21.8	150	94
14	0.018 in. (0.46 mm) Hastelloy X	None	Air	13.2	91	57
15		HT1	Air	14.4	99	62
16		HT1	Vacuum	20.1	139	87
17		HT2	Vacuum	20.1	139	87
18		HT2	Vacuum	19.0	131	82
19	0.002 in. (51 μm) Hastelloy X	None	Air	13.7	94	59
20		HT1	Air	14.7	101	64
21		HT1	Vacuum	18.6	128	80
22		HT2	Air	14.1	97	61
23		HT2	Vacuum	19.1	132	83
24	0.020 in. (0.51 mm) TD-Ni sheet	None	Vacuum	10.6	73	46
25	0.001 in. (25 μm) molybdenum	None	Vacuum	11.2	77	48
26	0.001 in. (25 μm) molybdenum	HT2	Vacuum	14.2	98	61
27	0.001 in. (25 μm) tungsten	None	Argon	10.2	70	44
28	0.001 in. (25 μm) tungsten	HT2	Vacuum	6.4	44	28
29	0.00025 in. (6 μm) titanium	HT2		12.9	89	56
30	0.0003 in. (7.5 μm) tantalum	None		10.7	74	46
31	Titanium vapor plate	HT2		10.2	70	44
32	Palladium vapor plate			11.8	81	51
33	Molybdenum electron beam (EB) evaporated			7.4	50	32
34	Tungsten EB evaporated			10.3	72	45
35	Iron EB evaporated			6.4	44	28
36	ThO_2 sintered on faying surfaces		Air	12.6	87	54

[a] HT1: Can removed, then heat treated at 2000° F (1093° C) for 100 hr in argon.
HT2: Heat treated in the can at 2000° F (1093° C) for 100 hr in argon.
[b] All square-edge fractures at the joints with <1 % elongation.
[c] Joint efficiency = $\frac{\text{Weld joint strength}}{\text{Base metal strength}} \times 100$.

C) stress-rupture tests took place partly at the weld interface and partly through the Hastelloy X interlayer material.

Discussion. HIP butt welded joints in TD-Ni bar with cobalt alloy and Hastelloy X interlayers were stronger than the base metal at room temperature. Use of these interlayers also produced butt joints with up to 100% joint efficiency in 2000° F (1093° C) tensile tests. Unfortunately, the 2000° F (1093° C) stress-rupture strength of all joints with interlayers was poor. Therefore, it was shown that stress-rupture testing is a much more severe criterion than 2000° F (1093° C) tensile testing. Room temperature tensile tests are of little or no value in evaluating the suitability of weldments for long-term elevated temperature service.

Lap Joints

Microstructure. Figure 16 shows the structure obtained in the lap joints. This photomicrograph was taken at mid-length of the joint as indicated on the sketch. Except for the small unwelded region the joint is difficult to find because it is oriented parallel to the long pencil-shaped grains. Heat treatment at 2000° F (1093° C) for 100 hr did not change the appearance of the microstructures at X500 magnification.

Shear Tests. Short-time shear test data are shown in Table 7 for duplicate tests of weldments at room temperature and at 2000° F (1093° C). A summary of the shear test data is shown in Fig. 17 with comparative data for the base metal taken from Table 1. At room temperature joint efficiency was 96% and at 2000° F (1093° C) the joint efficiency was 94%. Note that the base metal is relatively weak in shear. At 2000° F (1093° C) the tensile strength is 23.1 ksi (159 MN/m²) compared to a shear strength of 3.3 ksi (22.8 MN/m²) (Table 1). As noted in Table 7, one weldment failed at the joint and one failed in the base metal away from the joint at both room temperature and at 2000° F (1093° C).

These studies have shown that the shear strength of lap joints is about equivalent to that of the parent material at both room temperature and 2000° F (1093° C).

Concluding Remarks

The results of this solid-state welding study have indicated that an interruption in the microstructure of TD-nickel bar at a butt weld produces a weakening effect. Butt joints in which the long pencil-shaped grain pattern is disturbed are about half as strong as the base metal in 2000° F (1093° C) tensile tests. Interlayers can be used to increase 2000° F (1093° C) tensile strength. But stress-rupture properties of joints with interlayers are very poor. Foreign materials of any kind may be undesirable where the stress-rupture strength of solid-state welded or brazed joints is important. Thus, we believe that welded butt joints that match the strength of TD-Ni bar at elevated temperatures probably cannot be made unless the original bar microstructure is somehow preserved or restored at the joint. The usage of TD-Ni material may not be seriously impaired by the fact that butt joints are weak because:

1. Butt joints are not used extensively in industrial applications.
2. It may be possible to increase the material thickness at the joint.
3. The joint may be located at a low-stress region of the structure.

It is suggested that high temperature weakening effects near the weld line in butt joints with and without interlayers include loss of the original TD-Ni texture, loss of the thoria dispersion, and the presence of a large percentage of grain boundary area normal to the testing direction. Shear stresses that are developed due to differential thermal expansion of interlayers could also contribute to a weakening effect at the butt joints.

Based on the fact that in short-time shear tests lap joints are about as strong as the base metal, lap joints show considerable promise. Similar lap joints tested in stress-rupture must be run in order to confirm the desirability of using lap joints. Scarf joints may merit study also because a scarf butt joint would offer more joint area than a square butt joint. The scarf butt joint would also be at some angle to the applied axial load rather than normal to the load.

Summary of Results

Solid-state welds were made in $^1/_2$ in. (12.7 mm) diameter TD-nickel (TD-Ni) bar in both butt joints (with and without interlayers) and lap joints. Welding was accomplished in a 12 hr cycle using the hot isostatic pressure (HIP) welding process with peak parameters of 2000° F (1093° C) and 20.0 ksi (138 MN/m^2) helium for 2 hr. Tensile and shear joint efficiencies (defined as percent of base metal strength) were determined at room temperature and at 2000° F (1093° C). Stress-rupture tests were run at 2000° F (1093° C) on butt joints. Metallographic techniques also were used in the evaluation. The results are as follows:

1. For the joint with interlayers, stress-rupture tests at 2000° F (1093° C) were a much more severe criterion of joint strength than 2000° F (1093° C) tensile tests. The strongest joints in stress-rupture (with the 0.004 in. (0.10 mm) cobalt-alloy interlayer) for 100 hr life, had 15% joint efficiency.
2. Short-time shear tests of lap joints indicate that the HIP welds are about as strong as the base metal at room temperature and at 2000° F (1093° C).
3. All 2000° F (1093° C) tensile and stress-rupture specimens obtained from butt joints weldments failed with

Table 6—2000° F (1093° C) Stress-Rupture Data for HIP Butt Weldments with Interlayers (Stress-Rupture Atmosphere—Helium)

Interlayer	Specimen	Postweld heat treatment[a]	Stress, ksi	Stress, MN/m^2	Life,[b] hr
0.013 in. (0.33 mm) cobalt alloy	1	HT1	6	41	0.1
	2	HT1	2	14	1.0
	3	HT1	1.5	10	4.0
0.004 in. (0.10 mm) cobalt alloy	4	HT3+HT1	2.5	17	25.6
	5	HT1	3	21	2.2
	6	HT1	2	14	41
	7	HT1	1.5	10	191
0.018 in. (0.46 mm) Hastelloy X	8	HT1+HT3	3	21	1.0
	9	HT1	2	14	10
0.002in. (51 μm) Hastelloy X	10	HT1	3.5	24	2.4
	11	HT1+HT3	3	21	.2
	12	—	2	14	3.8

[a] HT1: Can removed, then specimen heat treated at 2000° F (1093° C) for 100 hr in argon. HT3: outgassed at 2200° F (1204° C) for $^1/_2$ hr in vacuum with can removed
[b] All square-edge fractures at joints, with <1% elongation.

Table 7—Short-Time Shear Test Data[a] for HIP Lap Weldments

Specimen	Test temperature[c] °F	Test temperature[c] °C	Shear strength ksi	Shear strength MN/m^2	Joint efficiency,[b] %	Fracture location
1	RT	RT	53.8	371	96	At joint
2	RT	RT	54.1	373	97	Base material
3	2000	1093	3.2	22	97	Base material at joint
4	2000	1093	3.0	21	91	Base material at joint

[a] Test specimen: see sketch shown in Table 1.
[b] Joint efficiency $= \frac{\text{Strength of weld joint}}{\text{Strength of base metal}}$
[c] RT = Room Temperature.

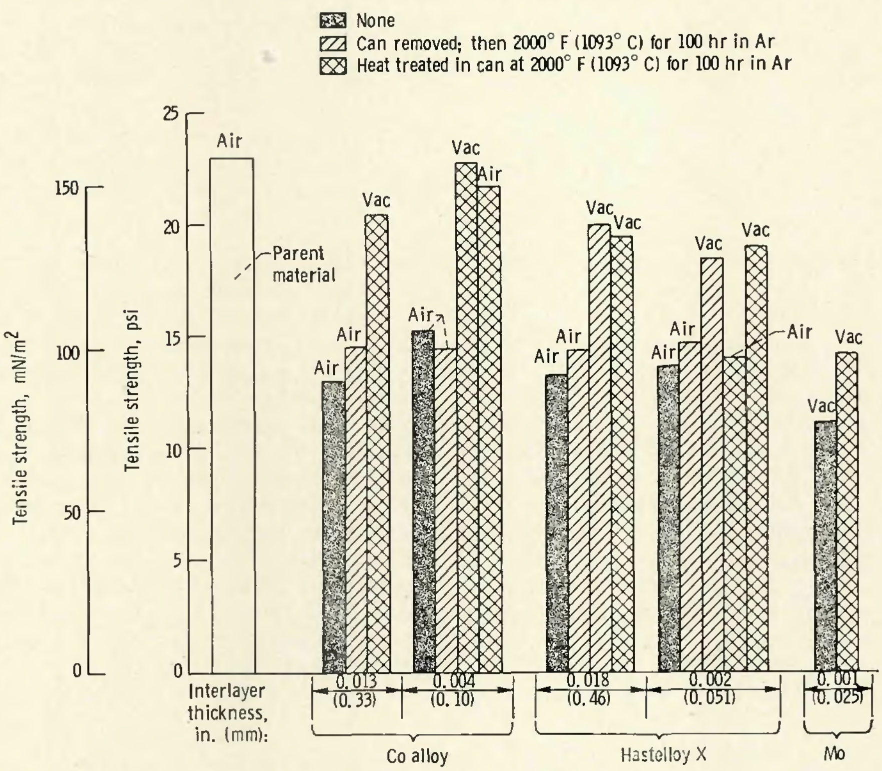


Fig. 13—Ultimate tensile strengths, at 2000° F (1093° C), of ½ in. (12.7 mm) diameter TD-Ni bar base metal and hot-isostatic-pressure (HIP) butt weldments with cobalt-alloy, Hastelloy X, and molybdenum interlayers. Testing was done both in air and in vacuum

Fig. 14—Fractures in 2000° F (1093° C) tensile tests of hot-isostatic-pressure butt welds with various interlayers. Etchants: (a) and (b)—swab with 92 milliliters HCl, 3 milliliters HNO_3, and 5 milliliters H_2SO_4, then electrolitically etch with 1 part H_2SO_4, 2 parts oxalic acid saturated solute; (c) swab with 92 milliliters HCl, 3 milliliters HNO_3, and 5 milliliters H_2SO_4. Left (a)—interlayer, 0.004 inch (0.10 mm) of cobalt alloy (specimen 11, Table 5); center (b)—interlayer, 0.002 in. (51 μm) of Hastelloy X (specimen 19, Table 5); right (c)—interlayer, 0.001 in. (25 μm) of molybdenum (specimen 26, Table 5) Etched; X500

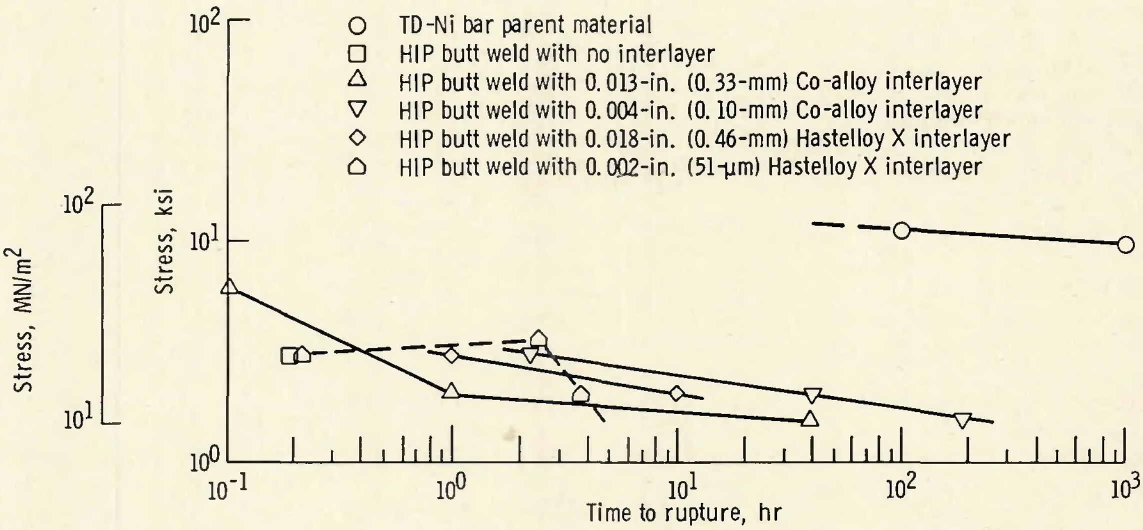


Fig. 15—Stress as function of time to rupture at 2000° F (1093° C) for hot-isostatic-pressure (HIP) butt weldments in ½ in. (12.7 mm) diameter TD-nickel bar with and without various interlayers

a characteristic square-edge appearance and <1% elongation.

4. In 2000° F (1093° C) tensile tests, butt joints with cobalt alloy and Hastelloy X interlayers had up to 100% and 87% joint efficiency, respectively. This represented the best results of the 15 interlayer materials evaluated. Without interlayers, maximum joint efficiency was about 55%.

5. Diffusion effects produced by postweld heat treatment (2000° F (1093° C) for 100 hr) tended to increase 2000° F (1093° C) tensile strength for joints with cobalt alloy, Hastelloy X, and Mo interlayers.

6. Butt joints with cobalt alloy, Hastelloy X, or with no interlayer have greater room temperature strength than the base metal.

Conclusions

1. For hot isostatic pressure (HIP) butt welds made in TD-nickel (TD-Ni) bar using interlayers, stress-rupture testing at 2000° F (1093° C) is a much more severe criterion of performance than 2000° F (1093° C) tensile tests. Interlayer materials can effectively increase joint efficiency (percent of base metal strength) in high temperature tensile tests. But the 2000° F (1093° C) stress-rupture strength is extremely poor.

2. Base metal properties can be achieved in 2000° F (1093° C) short-time shear tests of HIP-welded lap joints which have the joint in the plane of the axis of the TD-Ni bar.

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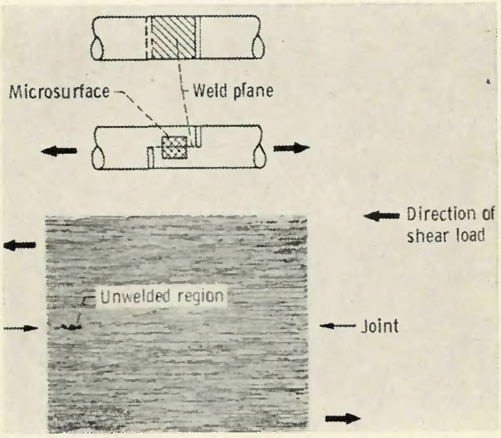

Fig. 16—Hot-isostatic-pressure-welded lap joint in ½ in. (12.7 mm) diameter TD-nickel bar. Original faying surfaces were milled and joint was heat treated at 2000° F (1093° C) for 100 hr after welding. Etchant: 92 milliliters HCl, 3 milliliters HNO_3, 5 milliliters H_2SO_4 (swab). X500 (reduced 10% on reproduction)

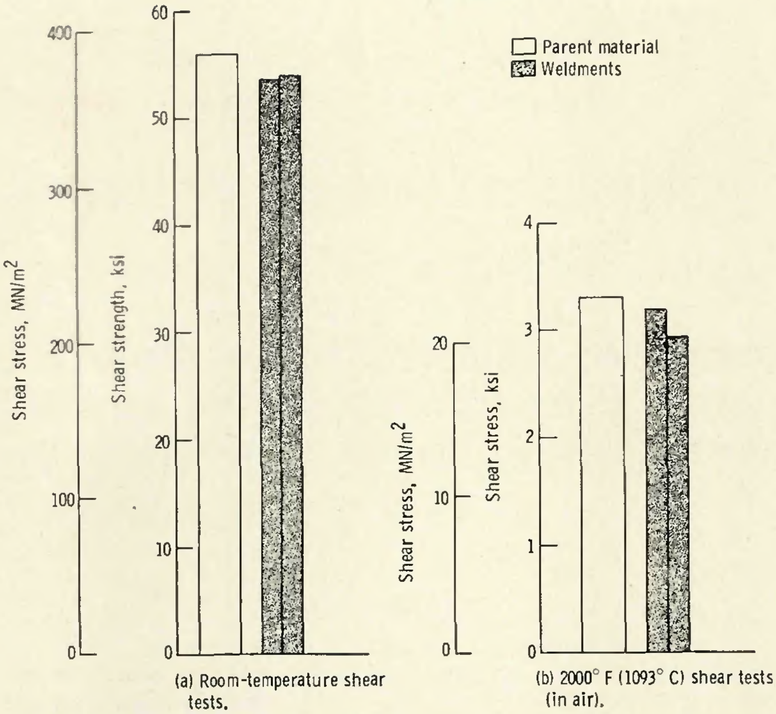

Fig. 17—Shear test results at room temperature and 2000° F (1093° C) of hot-isostatic-pressure-welded lap joints in ½ in. (12.7 mm) diameter TD-nickel bar in the as-welded condition. Base metal (i.e., parent material) specimens were shear tested using the same specimen design for purposes of comparison

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Appendix

A. Solid-State Welding Mechanism

In solid-state welding, the metallic bond may be achieved by bringing two clean surfaces into intimate contact.[9–13] Nikiforov[14] points out that, in diffusion welding, deformation caused by creep plays the leading part in making contact over the greater part of the weld interface. Thus, although diffusion across the weld interface may improve joint strength, it is secondary to joint formation.

In any solid-state welding process, however, it is likely that there will be at least some diffusion across the weld interface. Taylor's studies[15] with low-energy electron diffraction of the epitaxy of copper films on tungsten showed that the copper penetrated the tungsten to a depth of about five atomic layers.

In the HIP welding process used in this program, solid-state welding occurs when the combination of heat and external helium pressure cause the evacuated stainless steel cans to collapse and pressurize TD-Ni parts. The component of force normal to the faying surfaces promotes the intimate contact that is necessary to achieve welding. During the 2 hr hold time at high temperature and pressure, creep produced by diffusion mechanisms on either side of the weld interface is believed to play the major role in achieving the necessary contact over the entire faying surfaces. The pressure on the parts to be welded is equal in all directions. Thus, there is no macro-deformation of the TD-Ni bar. A side effect that occurs during the HIP welding process is that the stainless steel can is welded to the TD-Ni bar.

B. Terminology

The hot isostatic pressure (HIP) welding process used in this study to butt weld bar stock has been referred to as gas pressure bonding by its Battelle inventors.[4] Bonding processes in AMERICAN WELDING SOCIETY (AWS) terminology refer to the Adhesive Bonding of Metals, Chapter 46, *Welding Handbook*[16] and not to joining processes that involve development of the metallic bond between members. Since AWS has defined a process entitled "pressure gas welding" that involves heating with a gas flame,[17] it could cause confusion if the HIP welding process were called gas pressure welding. Therefore, HIP welding is believed to be a more suitable term.