

Enhanced Diffusion Welding of TD-NiCr Sheet

A unique vacuum hot press welding procedure can produce lap welds with parent metal strength in TD-NiCr sheet. Recrystallization and grain growth across the weld interface is achieved by using specially processed parent material

BY KENNETH H. HOLKO AND THOMAS J. MOORE

ABSTRACT. A method termed "enhanced diffusion welding" has been developed to produce solid state welds in TD-NiCr sheet with weld strengths of 100% of the parent metal strength. Previous work done without this method has resulted in weak welds (less than 60% parent metal strength). With the enhanced diffusion welding method, 1.6 mm (0.060 in.) TD-NiCr sheet was joined in a vacuum hot press in a lap configuration. Weldments were made that equalled the parent metal strength when tested in tensile-shear and creep-rupture shear at 1090C (2000F).

Both specially processed (unrecrystallized) and commercial TD-NiCr were welded successfully. However, the specially processed TD-NiCr was preferred over commercial TD-NiCr since the weld line could be eliminated as judged by light microscopy. Fracture took place in the parent material, away from the weld, when the welds in the specially processed material were tested. With the commercial TD-NiCr, fracture took place at the weld line indicating a plane of weakness.

The best preweld surface preparation procedure involved sanding through 600-grit paper and subsequent electropolishing. The electropolishing step was found to be necessary to prevent the formation of a continuous weld line with small, recrystallized grains and to allow complete grain growth across the weld line to occur.

A two-step weld cycle was found to work best in this study. It consisted of: (1) 207 MN/m² (30 ksi) and 705C (1300F) for 1 hour; (2) 15 MN/m² (2 ksi) and 1190C (2175F) for 2 hours. The first step provided the micro-alignment and intimate contact required for diffusion welding. The second step accelerated diffusion, weld formation and grain growth across the original weld interface.

Introduction

A dispersion strengthened nickel-base alloy, commercially designated TD-NiCr (Ni-20Cr-2ThO₂) is currently of interest because of its good high temperature strength and oxidation resistance. TD-NiCr derives its high temperature strength from mechanical working of the Ni-20Cr matrix which contains a fine dispersion of ThO₂ particles. TD-NiCr sheet is being considered for applications where metal temperatures may reach about 1200C (2200F) in an oxidizing environment. Examples of potential

applications include jet engine components and the heat shield panels of Space Shuttle vehicles¹ as shown in Fig. 1.

Joining dispersion-strengthened materials such as TD-NiCr by conventional fusion welding processes results in joint efficiencies (joint strength/parent metal strength X 100) of only about 40 to 50% at elevated temperatures.² Fusion welding TD-NiCr destroys the ThO₂ dispersion and the benefit of mechanical working is lost. The resulting strength of the fusion weldment is similar to that of thorium-free Ni-20Cr.

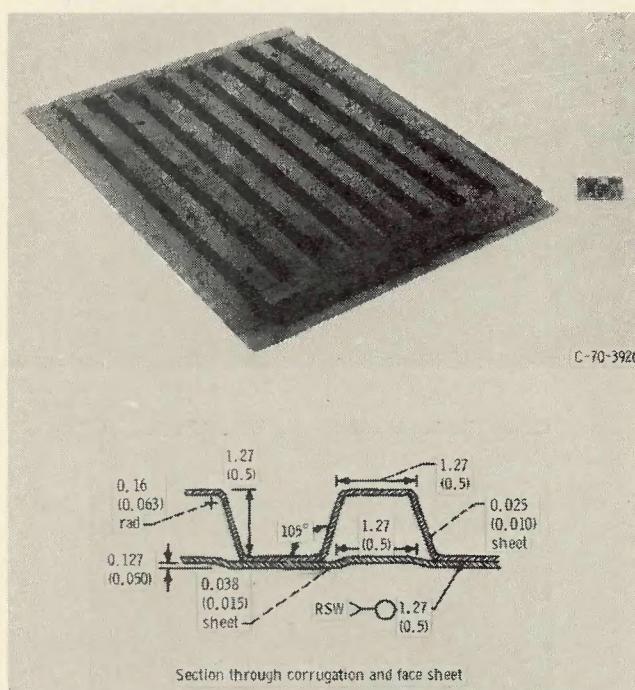


Fig. 1—Proposed TD-NiCr heat shield for space shuttle. Dimensions are in centimeters (in.). Face sheet, 0.038-centimeter (0.015-in.) thick TD-NiCr sheet. Rib structure, 0.025 centimeter (0.010-in.) thick TD-NiCr sheet spot welded to face plate. Surface finish, produced by grit blasting with Al₂O₃ and oxidizing for 1 hour at 1204C (2200F). Structure produced under USAF contract by McDonnell-Douglas Corp.

The authors are affiliated with the Lewis Research Center of the National Aeronautics and Space Administration, Cleveland, Ohio.

TD-NiCr has been joined by brazing.³ However, base metal strength may be reduced from the effects of diffusion between the braze alloy and TD-NiCr. The ThO₂ dispersion may be lost and/or porosity may develop in the TD-NiCr. Also, the braze alloys are weaker than TD-NiCr at elevated temperatures and joint efficiencies of 50% are typical.²

Solid-state welding is a promising approach to joining TD-NiCr since melting is avoided and a foreign material need not be introduced at the joint. However, two problem areas have been encountered with solid-state welding TD-NiCr sheet. The first is that a thin recrystallized band of small grains form at a continuous weld line between the two pieces of TD-NiCr being joined.³ At elevated temperatures, the continuous weld line (which acts as a grain boundary) and small grains are weak and the joint fails at low stresses. Typically, joint

efficiency is 0 to 60%. Secondly, unwelded areas occur sporadically at the weld line. The high creep strength of TD-NiCr at elevated temperatures prevents the intimate contact required at the faying surfaces to completely avoid unwelded areas. The problems will be illustrated herein.

The purpose of this report is to describe the techniques that were developed to eliminate the problems noted above. Both specially processed (SP) and commercial TD-NiCr sheet 1.6 mm (0.060 in.) thick were included in this study. The SP sheet had not received the final recrystallization heat treatment that is normally used in the production of commercial TD-NiCr. The effects of various surface preparations and vacuum hot press weld cycles on the properties of lap-weldments were evaluated. Evaluation of the most promising weldments was accomplished by tensile-shear and creep-rupture shear tests at 1090C

(2000F) and by metallographic analysis. A recommended two-step weld cycle for the TD-NiCr alloy is described.

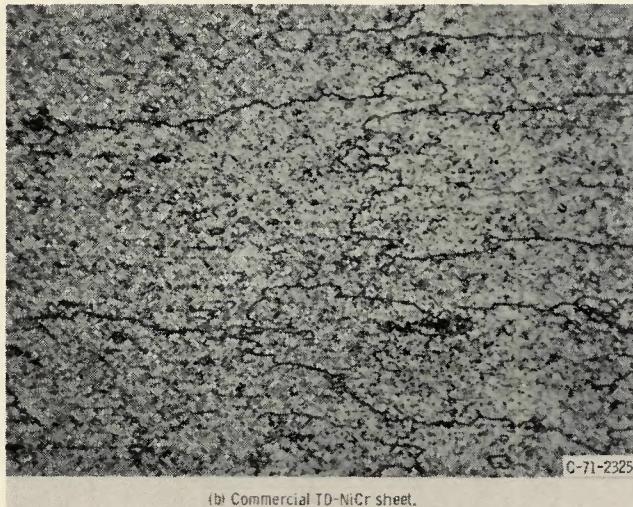
Materials and Procedure

Specially processed material. Specially processed (SP) TD-NiCr 1.6 mm (0.060 in.) thick was purchased from Fansteel, Inc. The nominal composition is Ni-20Cr-2ThO₂. The special processing consisted of leaving out the final recrystallization heat treatment that is normally given to commercial TD-NiCr after thermo-mechanical processing. So, the SP TD-NiCr is actually in the unrecrystallized condition. TD-NiCr in the SP condition has a grain size that is too fine to see as shown by the metallographic section in Fig. 2.

SP TD-NiCr was selected for use in this study for two reasons. First, since the microstructure is unrecrystallized, it was felt that the weld line could be eliminated by recrystallization and grain growth after welding. Secondly, we have found that SP TD-NiCr has good ductility (13% elongation in 25 mm (1 in.) at 705C (1300F). We planned to use this ductility to our advantage by developing the intimate contact required for diffusion welding at this temperature. At temperatures higher than about 815C (1500F), the SP microstructure begins to recrystallize and becomes similar to the commercial TD-NiCr. The ductility of TD-NiCr after recrystallization decreases with increasing temperature, and typically is only about 2% elongation at 1090C (2000F). This low



(a) Specially processed TD-NiCr sheet.



(b) Commercial TD-NiCr sheet.

Fig. 2—Parent metal microstructures of specially processed and commercial TD-NiCr. Material was 1.6-millimeters (0.060-in.) thick and sectioned parallel to rolling direction. Etchant, 100 milliliters H₂O, 2 grams CrO₃ and 10 milliliters H₂SO₄ (electrolytic, 3 V dc). Mag: 500X

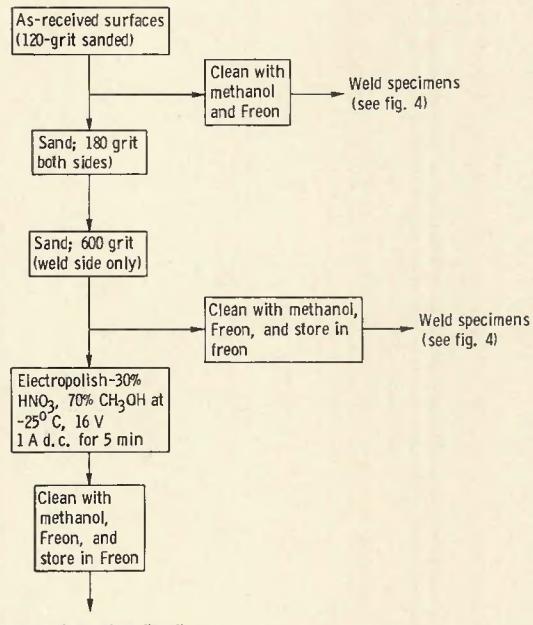


Fig. 3—Flow diagram for various weld specimen surface preparations

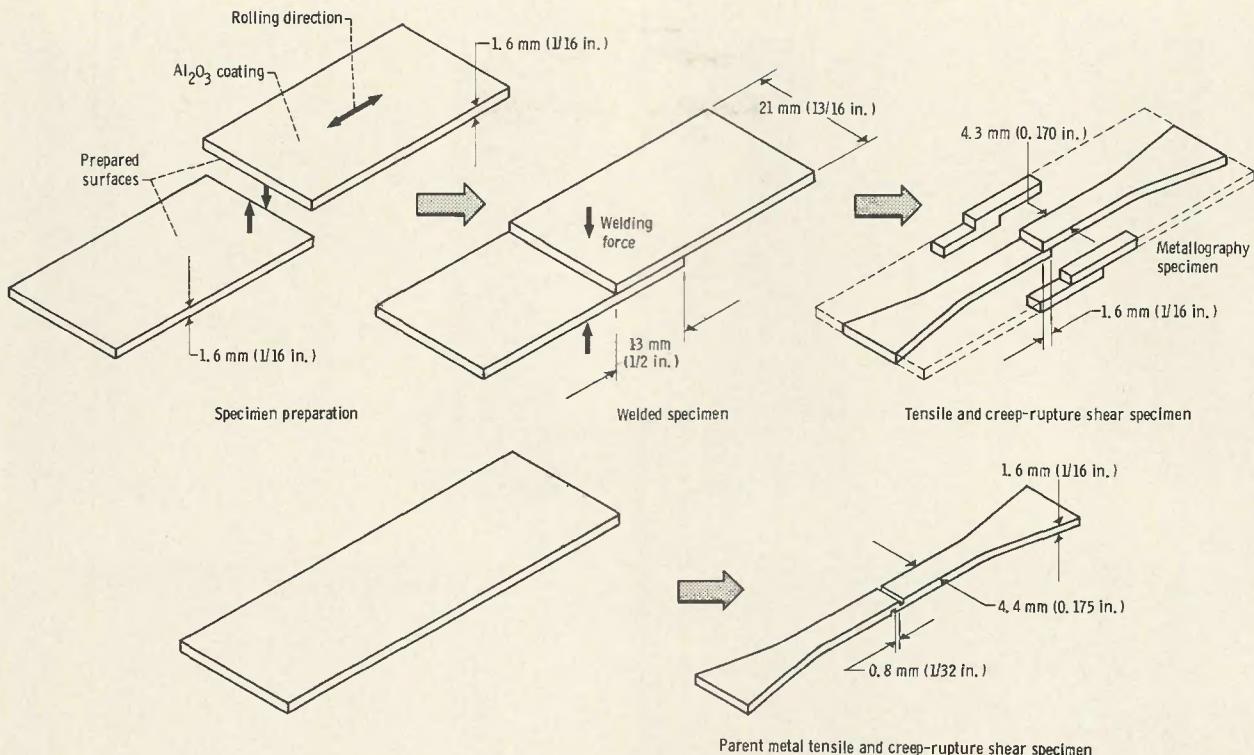


Fig. 4—Weld specimen configuration and location of tensile-shear, creep-rupture shear and metallography specimens from weldment and parentmetal.

ductility coupled with high temperature creep-resistance makes it difficult to develop intimate contact without cracking at the higher temperatures.

Commercial material. Commercial TD-NiCr 1.6 mm (0.060 in.) thick was purchased from Fansteel, Inc. It was included in this study for a comparison with SP TD-NiCr. Commercial TD-NiCr is made from the SP material by recrystallization at 1180°C (2150°F) for 2 hours. This heat treatment is termed "stress relieving" by the manufacturer. The commercial material is more ductile at room temperature than the SP material and is thus more formable. The nominal composition is Ni-20Cr-2ThO₂. A metallographic section parallel to the principal rolling direction is shown in Fig. 2. Notice the large grain size and small dark Cr₂O₃ particles in the microstructure. The magnification in Fig. 2 is not high enough to observe the ThO₂ dispersion which is responsible for the good high temperature strength of TD-NiCr.

Welding Procedure

Equipment. A vacuum hot press was used to diffusion weld lap joints in both commercial and SP TD-NiCr. The weld specimens were radiantly heated by a tantalum resistance heater. Molybdenum rams were used to transmit welding force from a 220 KN (25 ton) hydraulic press to the weld specimens. A pressure of

2×10^{-5} torr was maintained in the vacuum chamber during welding.

Specimen Preparation. The as-received TD-NiCr sheet (both types) had 120-grit sanded surfaces with the surface scratches parallel to the principal rolling direction. The surfaces were prepared for welding in three different ways as shown in Fig. 3. Some weld specimens (as shown in Fig. 4) were prepared by just cleaning the as-received 120-grit sanded surfaces with methanol and Freon (trichloro-trifluoroethane) prior to welding. Other specimens were prepared by sanding both sides with 180-grit paper, sanding the side to be welded with 600-grit paper, cleaning with methanol and Freon, and storing in Freon to minimize oxidation. Finally, some specimens were sanded through 600-grit paper as described above, then electropolished, cleaned with methanol and Freon, and stored in Freon. The specimen surfaces in contact with the molybdenum rams were

coated with Al₂O₃, as shown in Fig. 4, to prevent sticking.

Weld Cycles After overlapping the specimens approximately 13 mm (1/2 in.), a vacuum of 2×10^{-5} torr was attained in the weld chamber. The specimens were heated to the welding temperature, welding force was applied, and diffusion welding was achieved. No measurable deformation was recorded after welding.

A limited amount of experimentation was done with the weld cycle variables of temperature, time and pressure. From the evaluation of welds made with various cycles, the two-part weld cycle shown in Table 1 is recommended and was used for most of the work described in this report. This cycle consists of a low temperature, high pressure portion (705°C or 1300°F) and 210 MN/m² (30 ksi) for 1 hr followed by a high temperature, low pressure portion (1190°C or 2175°F) and 15 MN/m² (2 ksi) for 2 hr.

Table 1—Typical Weld Cycles

	Temperature °C	Temperature °F	Pressure MN/m ²	Pressure ksi	Time hr	Vacuum, torr
Recommended cycle:						
Step 1	705	1300	210	30	1.0	2×10^{-5}
Step 2	1190	2175	15	2	2.0	
Representative early cycle (not recom- mended)	1040	1900	40	6	1.0	2×10^{-5}

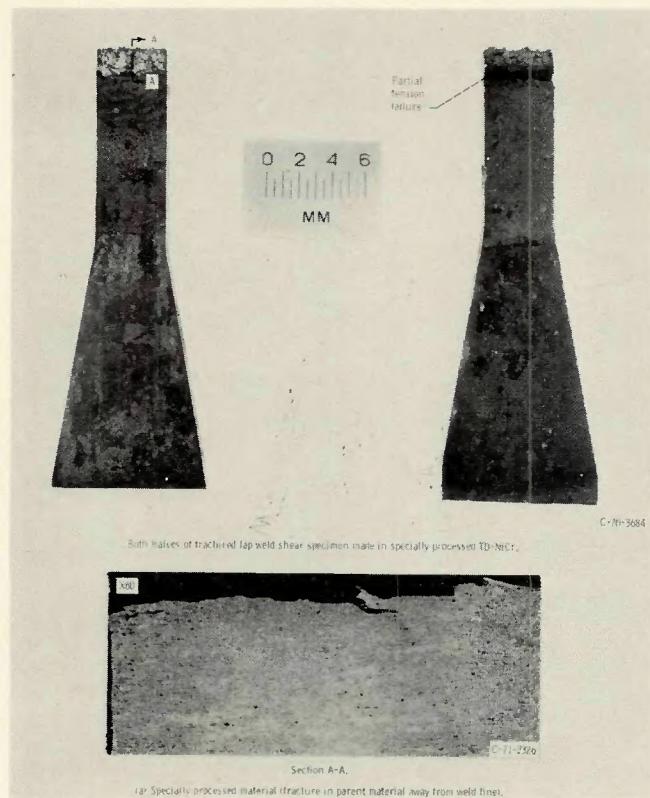


Fig. 5—Typical weld tensile-shear specimen fractures of 1.6-millimeter (0.060-in.) TD-NiCr. Planar and cross-sectional views. Etchant, 100 milliliters H_2O , 2 grams CrO_3 and 10 milliliters H_2SO_4 (electrolytic, 3 V dc)

Time did not permit a thorough investigation of each of the weld cycle variables. But various combinations of temperature, time and pressure were tried and are tabulated in the appendix. A cycle that is representative of many of these single-step combinations is shown in Table 1 for comparison with the two-step recommended cycle used for most of the study. Variable weld strengths resulted from most of the single-step combinations tried. For example, of two creep-rupture shear specimens machined

from the same weld specimen in early studies, one failed on loading while the other had 100% joint efficiency.

Weld Evaluation

The weldments were evaluated both metallographically and by tensile and creep-rupture shear tests. The locations of the test samples with respect to the weldments are shown in Fig. 4.

The same specimen configuration was machined from the weldments for tensile-shear and creep-rupture shear testing as shown in Fig. 4. This test specimen configuration had a test sec-

tion with a 1.6 mm (0.060 in.) overlap and a 4.3 mm (0.170 in.) width. All specimens were heat treated at 1260°C (2300°F) for 1 hour in vacuum prior to testing. Specimens were tensile shear tested in air at 1090°C (2000°F) at a crosshead speed of 1.3 mm/min (0.05 in./min). Creep-rupture shear tests were conducted at 1090°C (2000°F) with dead weight loading of approximately 70 N (16 lb).

To compare weld strengths to parent metal strength, parent metal shear specimens were machined (see Fig. 4)

Table 2—TD-NiCr Parent Metal Tensile-Shear and Creep-Rupture Shear Strengths at 1090C (2000F)

Specimen	Type of test	Material condition	Shear stress at failure		Time, hr
			MN/m ²	ksi	
C-1	Tensile-shear	Commercial plus heat treated ^a	45.2	6.56	—
C-3	Tensile-shear	Commercial plus heat treated ^a	70.3	10.20	—
X-1	Tensile-shear	Specially processed plus heat treated ^a	72.4	10.50	—
X-2	Tensile-shear	Specially processed plus heat treated ^a	72.4	10.50	—
	Tensile-shear		65.1 av	9.5 av	
	Tensile-shear				
C-2	Creep-rupture shear	Commercial plus heat treated ^a	19.3	2.80	261.0
U-2	Creep-rupture shear	Specially processed plus heat treated ^a	27.2	3.95	2.2
U-5	Creep-rupture shear		25.2	3.70	27.2
U-6	Creep-rupture shear		21.8	3.16	66.6

^a Heat treated at 1260C (2300F)/1 hr/vacuum

Table 3—Tensile-Shear and Creep-Rupture Shear Strengths of TD-NiCr Welds at 1090C (2000F)

Specimen	Type of test	Material condition	Shear stress at failure		Time, hr	Location of fracture
			MN/m ²	ksi		
HP-84	Tensile-shear	Specially processed plus heat treated ^a	56.0	8.14	—	Parent
HP-85	Tensile-shear		44.8	6.50	—	Parent
HP-89	Tensile-shear		52.4	7.60	—	Parent and weld
HP-90	Tensile-shear		52.4	7.60	—	Parent and weld
			51.4 av	7.31 av		
HP-86	Tensile-shear	Commercial plus heat treated ^a	27.7	4.02	—	Weld
HP-87	Tensile-shear		51.6	7.50	—	Weld
			39.7 av	5.76 av		
HP-81	Creep-rupture	Specially processed plus heat treated ^a	20.1	2.92	108.4	Parent
HP-83	Creep-rupture		20.5	2.97	335.4+	Parent
HP-88	Creep-rupture		24.1	3.50	23.5	Parent
HP-91	Creep-rupture		27.4	3.97	13.9	Parent
HP-93	Creep-rupture	Commercial plus heat treated ^a	20.7	3.03	405.4+	Parent
HP-96	Creep-rupture		23.4	3.40	379.9+	Parent
HP-97	Creep-rupture		27.9	4.05	17.3	Parent
HP-78	Creep-rupture		20.7	3.01	43.0	Parent
HP-80	Creep-rupture	Commercial plus heat treated ^a	23.4	3.40	287.2+	Weld

^a Heat treated at 1260C (2300F)/1 hr/vacuum after welding

and tested. Testing conditions were the same as for the weld specimens. There was one difference between the specimen configurations, however. As shown in Fig. 4, there is some bending moment on the shear area inherent in the weld specimen design, since it is a straight overlap. There is less bending moment with the parent metal specimen since the shear area lies on the axis of loading. This difference in specimen configuration could result in higher indicated shear strength for the parent material.

Metallographic sections were cut from the edges of selected weldments. They were polished and etched electrolytically with a solution of 100 ml H₂O and 2 g CrO₃ and 10 ml H₂SO₄. Some replica electron microscopy work also was included.

Results and Discussion

The best diffusion welds in TD-NiCr were produced by carefully selecting the metallurgical condition of the starting material, weld specimen surface preparation and weld cycle variables. The welds obtained by the selected combination of the above variables were as strong as the parent material and metallurgically undistinguishable from the parent material. The proper metallurgical condition was the SP or unrecrystallized form of TD-NiCr as shown in Fig. 2. The recommended surface preparation is the sanding and electropolishing procedure shown in Fig. 3 and the recommended weld cycle is the two-step cycle shown in Table 1.

Lap Joint Properties

Mechanical property data are for both SP and commercial TD-NiCr. All weld specimens were prepared by 600-grit sanding plus electropolishing

(see Fig. 3). Only the recommended weld cycle (see Table 1) was used for these test specimens. Just prior to testing, all specimens (parent and weldment) were heat treated at 1260C (2300F) for 1 hour in vacuum (1×10^{-5} torr). All specimens were tested in air at 1090C (2000F). Only shear-type testing was done and the specimen configurations are shown in Fig. 4.

Tensile-Shear Tests The 1090C (2000F) tensile-shear strength data for the parent materials used in this study are shown in Table 2. The material that was specially processed plus heat treated at 1260C (2300F) for 1 hour exhibited strengths equivalent to that of the heat treated commercial TD-NiCr. The average parent metal tensile-shear strength is 65 MN/m² (9.5 ksi) at 1090C (2000F).

The welds made in SP TD-NiCr had an average tensile-shear strength of about 50 MN/m² (7.5 ksi) (see Table 3). This represents a joint efficiency of 79%. However, the SP TD-NiCr weldments failed mostly in the parent material, away from the original weld interface, as shown in Fig. 5. Close inspection of the planar view of

the failed specimen in Fig. 5 shows a tension-type fracture away from the weld in addition to the shear fracture. This is due to the bending moment on the joint that exists when the shear load is applied because of the weld specimen configuration. This bending moment is much less when testing the parent metal configuration (Fig. 4). Because of the bending moment and location of the fracture, it is believed that the joint efficiency is actually 100%. So, parent metal strength has been attained.

Welds made in commercial TD-NiCr had an average shear strength of about 40 MN/m² (5.8 ksi) (as shown in Table 3). This represents a joint efficiency of 62%. These commercial TD-NiCr weldments failed at the original weld interface with a smooth fracture surface as shown in Fig. 5. This is considered a bad fracture mode for diffusion welds since it is indicative of a plane of weakness.

Creep-Rupture Shear Tests Parent TD-NiCr creep-rupture shear data are shown in Table 2 and shear stress values as a function of time-to-rupture are presented in Fig. 6. The solid line drawn in Fig. 6 is for the parent

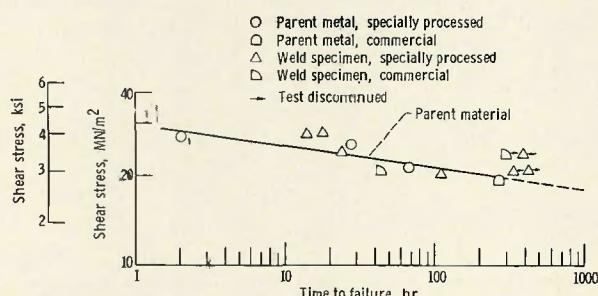


Fig. 6—Shear stress as function of time to rupture for parent and diffusion welded lap joints in 1.6 millimeter (0.060-in.) TD-NiCr sheet at 1090C (2000F) in air. All samples annealed at 1260C (2300F) for 1 hour prior to test

material and shows that a 100-hour life at 21 MN/m² (3.1 ksi) is expected.

Creep-rupture shear data for welds in SP and commercial TD-NiCr are shown in Table 3 and plotted along with parent metal data in Fig. 6. Comparison of parent and weld

strengths in Fig. 6 shows that parent metal strength has been obtained for welds in both SP and commercial TD-NiCr.

For all four welds in the SP TD-NiCr, fracture always occurred in the parent material away from the weld. So again the welds in SP TD-NiCr

have a better fracture mode. The bending moment did not significantly lower the recorded weld strength during creep-rupture testing because lower loads were used than in short-time tensile shear testing. Also, the creep resistance of the TD-NiCr may have lessened the effect of the bending moment. For two welds in commercial material, fracture occurred once through the parent material and once at the original weld interface.

Microstructural Evaluation

Specimens prepared from both specially processed and commercial material were examined by light microscopy and electron replica microscopy. The recommended weld cycle was used in all cases. The electropolished surface preparation was evaluated for both materials. And the 120-grit and 600-grit sanded surface preparations were evaluated for the SP material.

Effect of Surface Preparation

The recommended surface preparation consists of sanding through 600-grit paper and then electropolishing as shown in Fig. 3. With this surface preparation and the recommended weld cycle (Table I), the best welds were made as shown by the metallographic sections in Fig. 7. Diffusion welding the SP TD-NiCr with the recommended surface preparation and weld cycle described above eliminated the weld line as shown in Fig. 7. Inspection of welds made in this manner showed only a few isolated areas where the weld line was observable. The mode of fracture in mechanically tested welds at 1090°C (2000°F) was through the parent material away from the weld line. This was expected since the weld line was eliminated.

Figure 7b shows a lap weld in commercial TD-NiCr. Observation of this weld and others like it reveal a relatively straight, continuous weld line

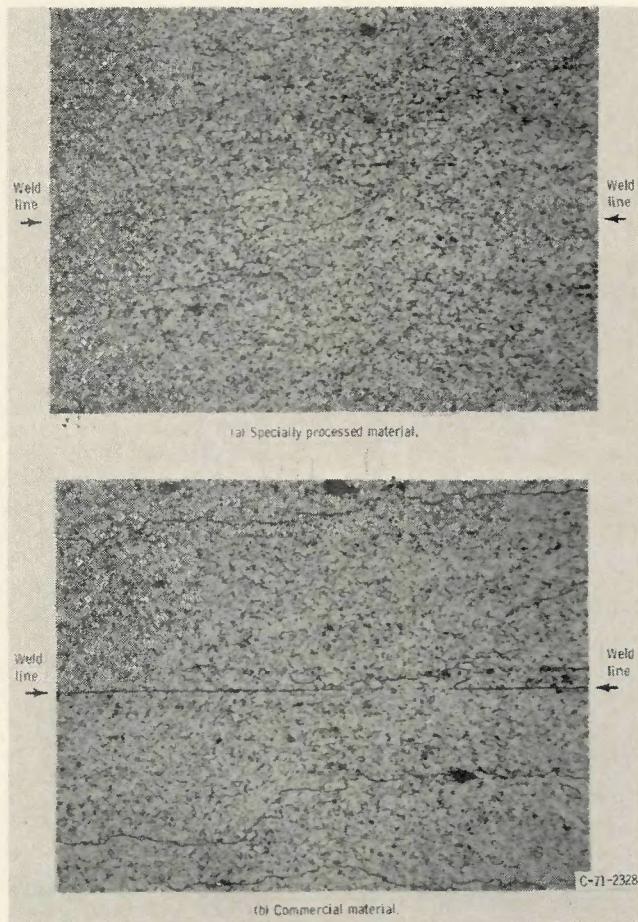


Fig. 7—Effect of material difference on weld quality using recommended surface preparation and weld cycle. Sections were taken parallel to rolling direction. Etchant, 100 milliliters H₂O, 2 grams CrO₃ and 10 milliliters H₂SO₄ (electrolytic, 3 V dc). Mag: 500X

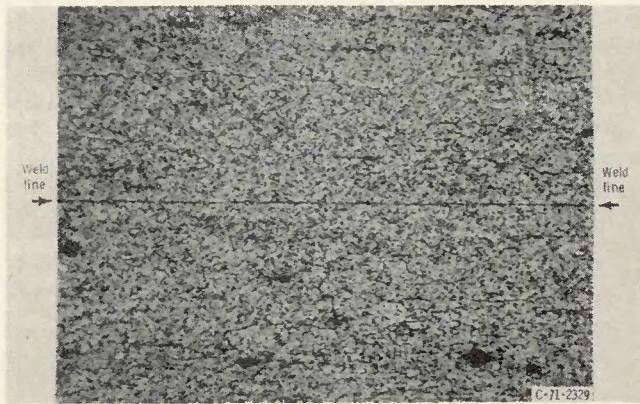


Fig. 8—Effect of 600-grit surface preparation (without electro-polishing) on weld quality of weld made in specially processed material with recommended weld cycle. Section was taken parallel to rolling direction. Etchant, 100 milliliters H₂O, 2 grams CrO₃ and 10 milliliters H₂SO₄ (electrolytic, 3 V dc). Mag: 500X



Fig. 9—Effect of 120-grit surface preparation (without electro-polishing) on weld quality of weld made in specially processed material with recommended weld cycle. Section is parallel to rolling direction and shows unwelded area and extensive recrystallization at weld line. Etchant, 100 milliliters H₂O, 2 grams CrO₃ and 10 milliliters H₂SO₄ (electrolytic). Mag: 500X

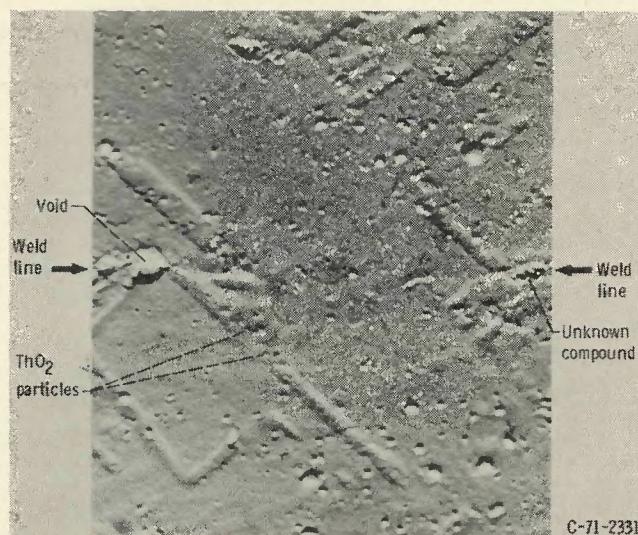
with areas of grain growth across the weld line (approximately 10 to 20% of the weld area). Isolated areas of nonwelding were noticed (not shown).

These results are better than those reported for more conventional diffusion-welding joints³ in that small, recrystallized grains at the joint and a continuous weld line were avoided. When these small grains and a continuous weld line are present, inconsistent mechanical properties results are obtained. Welds in commercial TD-NiCr failed at the weld line with a smooth fracture surface. This was expected since an almost continuous weld line was observed.

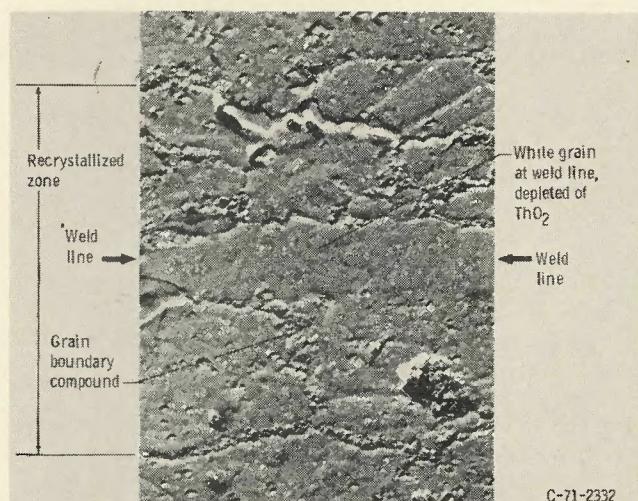
The importance of the electropolishing step in the weld specimen surface preparation is illustrated in Fig. 8. A welded specimen of SP material sanded through 600-grit paper but not electropolished is shown. The recommended weld cycle was used. A continuous weld line bordered by small recrystallized grains is apparent. Isolated unwelded areas were noticed (not shown). These results were typical of this surface preparation regardless of the weld cycle used. Similar results were obtained with the commercial material. Probably the 600-grit sanding did not completely eliminate surface roughness and introduced some cold work in the surfaces. Then during welding these small asperities were deformed and along with the energy released from prior surface cold working, local recrystallization at the weld line resulted. The type of weld shown in Fig. 8 gave widely varying mechanical strengths when tested at elevated temperatures.

The effect of sanding with coarser grits is illustrated in Fig. 9. Shown is a welded specimen, made with as-received, 120-grit sanded surfaces in SP material. Large unwelded areas and extensive recrystallization at a continuous weld line were typical of the 120-grit sanded surface preparation. Although it is not readily apparent in Fig. 9, examination at higher magnifications indicated a line of white grains was present at the weld line with small grains on either side.

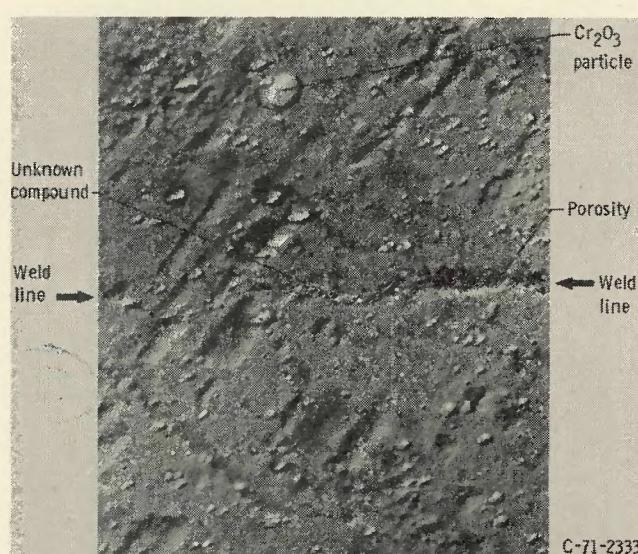
Electron microscopy was used to study the recrystallized area, as will be described. The extensive recrystallization is believed to be due to the deformation of asperities and the release of energy stored from cold working during sanding, as previously described. The unwelded areas are probably due to the unevenness of the TD-NiCr sheet. The high creep resistance of TD-NiCr prevents complete intimate contact of the faying surfaces during the weld cycle.



(a) Specially processed material with 600-grit sanded plus electropolished surface preparation.



(b) Specially processed material with 120-grit sanded as-received surfaces.



(c) Commercial material with 600-grit sanded plus electropolished surface preparation.

Fig. 10—Effect of surface preparation on ThO_2 distribution near original weld line. Sections were taken parallel to rolling direction and recommended weld cycle was used. Mag: 26,000X

Table 4—Weld Cycles Employed

Specially Processed TD-NiCr					
Surface preparation	Temperature		Pressure		Time, hr
	°C	°F	MN/m ²	ksi	
120-grit sanded	1040	1900	76.0	11.0	0.05
120-grit sanded	1040	1900	41.3	6.0	0.5, 1.0
120-grit sanded plus electropolished	1040	1900	68.9	10.0	0.05
600-grit sanded	1040	1900	55.0–68.9	8.0–10.0	0.05
	845	1550	76.0	11.0	0.05
	955	1750	62.0	9.0	0.05
	1040	1900	41.3	6.0	0.5, 1.0
Recommended cycle ^a					
600-grit sanded plus electropolished	1040	1900	68.9–82.5	10.0–12.0	0.05
	1040	1900	41.3	6.0	0.5, 1.0
	1150	2100	82.5	12.0	0.05
	1150	2100	20.6	3.0	1.0
	815	1500	96.4	14.0	3.0
	705	1300	172.0–276.0	25.0–40.0	1.0
	1190	2175	13.8	2.0	2.0
Recommended cycle ^a					
Commercial TD-NiCr					
120-grit sanded	1040	1900	41.3	6.0	0.5, 1.0
120-grit sanded	1090	2000	62.0–103.2	9.0–15.0	0.05
120-grit sanded plus electropolished	1090	2000	68.9	10.0	0.05
Recommended cycle ^a					
600-grit sanded	955–1090	1750–2000	65.4	9.5	0.05
600-grit sanded	1040	1900	41.3	6.0	0.5, 1.0
600-grit sanded plus electropolished	1090	2000	68.9	10.0	0.05
600-grit sanded plus electropolished	1040	1900	41.3	6.0	1.0
	815	1500	96.4	14.0	3.0
	1150	2100	20.6	3.0	1.0

^a See table I for recommended cycle

The weld shown in Fig. 9 was made with the recommended weld cycle and the same results were observed for all weld cycles evaluated. This type of weld was extremely weak when tested at elevated temperatures; in fact, it commonly failed on loading in creep-rupture testing.

Electron microscopy was undertaken to determine the effects of surface preparation on ThO₂ distribution and general structure near the weld line. Three as-welded lap joints were studied with the weld cycle as recommended in Table 1:

Fig- ure	Sheet material	Surface preparation
10(a)	SP TD-NiCr	600-grit sanded and electro-polished
10(b)	SP TD-NiCr	as-received (120-grit sanded)
10(c)	Commercial TD-NiCr	600-grit sanded and electro-polished

An electron photomicrograph at the weld in SP material with 600-grit sanded plus electropolished surfaces is shown in Fig. 10(a) and indicates no change in ThO₂ distribution in the vicinity of the weld. The microstructure appears to be continuous across the weld line. Light microscopy of this

joint (Fig. 7) revealed no defects. But at the weld line in Fig. 10(a) and in other electron photomicrographs (not shown) a few small voids were observed. An unknown compound was also seen at the weld line. This compound was not continuous at the weld line but it was identical in appearance to a compound located at the parent material grain boundaries (not shown).

Electron photomicrographs (Fig. 10b) of the weld in SP material with as-received 120-grit sanded surfaces showed evidence of ThO₂ depletion in recrystallized grains at the weld line as a result of the rough surface preparation. These ThO₂ depleted grains are about 1 micron wide. At 500 magnification, they appeared as a line of white grains (Fig. 9). The material at the grain boundaries of the recrystallized grains on either side of the white grains is believed to be rich in ThO₂ (Fig. 10b). If this is the case the mechanism by which distribution of the ThO₂ changed is unknown. ThO₂ depleted grains have been observed⁴ in diffusion welds in another dispersion-strengthened alloy, TD-Ni.

The appearance of a weld made with smooth surface preparation (600-grit sanded plus electropolished) in commercial material is shown in Fig. 10c. This weld evaluation was

intended as a comparison base for the SP material (Fig. 10a). The commercial TD-NiCr weldment had shown a nearly continuous weld line in light microscopy (Fig. 7b). The electron photomicrograph (Fig. 10c) reveals normal ThO₂ distribution at the weld, the presence of an unknown weld line compound and perhaps very fine porosity. A few isolated larger void areas were also observed at the weld line.

Effect of Varying Weld Cycles

As shown in Table 4, various combinations of weld cycle variables (time, temperature and pressure) were tried. Consistently good metallurgical and mechanical testing results were only obtained for the recommended two-step welding cycle shown in Table 1.

For other welding cycles, such as the representative early cycle shown in Table 1, variable results were obtained. For example, two specimens with a 600-grit sanded surface preparation were welded at 1040°C (1900°F) and 60 MN/m² (9 ksi) for 5 minutes. When tested in creep-rupture shear at 1090°C, both specimens had 100% joint efficiency. However, two specimens welded under identical conditions, failed on loading at similar stresses. Because of inconsistencies like these we strongly recommend the two-step welding cycle.

TD-NiCr is a difficult material to diffusion weld because of its high creep resistance and low ductility at elevated temperatures. Unevenness and lack of contact at the mating surfaces to be welded are difficult to correct because of the high creep resistance. If the welding pressure is increased at conventional diffusion welding temperatures (circa 1090°C (2000°F) to bring uneven surfaces into intimate contact for welding, cracking is likely to occur in the TD-NiCr parent material.

To solve the problem of obtaining intimate contact at the mating surfaces, high welding pressure (210 MN/m²) (30 ksi) was applied at 705°C (1300°F) where SP TD-NiCr has about 13% elongation and commercial TD-NiCr has about 9% elongation, both in 25 mm (1 in.). After intimate contact and incipient welding were attained, the pressure was reduced to a low value and the temperature raised to 1190°C (2175°F) to accelerate diffusion (and cause recrystallization for SP TD-NiCr) and grain growth across the weld line. The pressure was then increased to 15 MN/m² (2 ksi). In this manner, consistently good diffusion welds were attained.

To determine if both parts of the recommended weld cycle are really

necessary, a variation of the recommended weld cycle was tried. Omission of the low temperature, high pressure portion of the recommended weld cycle (with 600-grit sanded plus electro-polished specimen surfaces) resulted in a lack of intimate contact and a poor diffusion weld. This is shown in Fig. 11a by the presence of unwelded areas and a continuous weld line at the original interface (compare with Fig. 7a).

A second variation of the recommended weld cycle was tried to determine the effects of recrystallization of the SP material just prior to the application of the recommended weld cycle in the hot press. Again, a poor solid-state weld resulted. This is shown by a continuous weld line and unwelded areas in Fig. 11b.

So, it is seen that both parts of the recommended weld cycle are necessary and recrystallization of the SP TD-NiCr must take place during the weld cycle.

Concluding Remarks

A vacuum hot press welding procedure (termed enhanced diffusion welding) is recommended to produce lap welds in TD-NiCr sheet. Recrystallization and grain growth across the initial weld interface (and thus enhanced movement of the weld interface) is achieved by using specially processed parent material. Other important procedural features include the use of flat electropolished surfaces and a two-step weld cycle. Electropolishing the surfaces makes it possible to eliminate the weld line during welding. The first step of the two-step cycle provides the intimate contact required for diffusion welding. The second step accelerates diffusion and weld formation.

Although this program did not include an exhaustive study of the variables, the work reported herein does offer some clues as to how the recommended procedure might be improved. There were for example some isolated instances of high strength welds made with much shorter welding times. We suggest that the two-step welding cycle approach be retained but that the times for both steps be shortened. Also, electropolishing the surfaces prior to welding may be replaced by a less expensive chemical polishing technique.

Conclusions

An enhanced diffusion welding method has been developed to join TD-NiCr sheet with parent metal strength at the weld. Diffusion welded joints were made in specially processed TD-NiCr that equalled the ten-

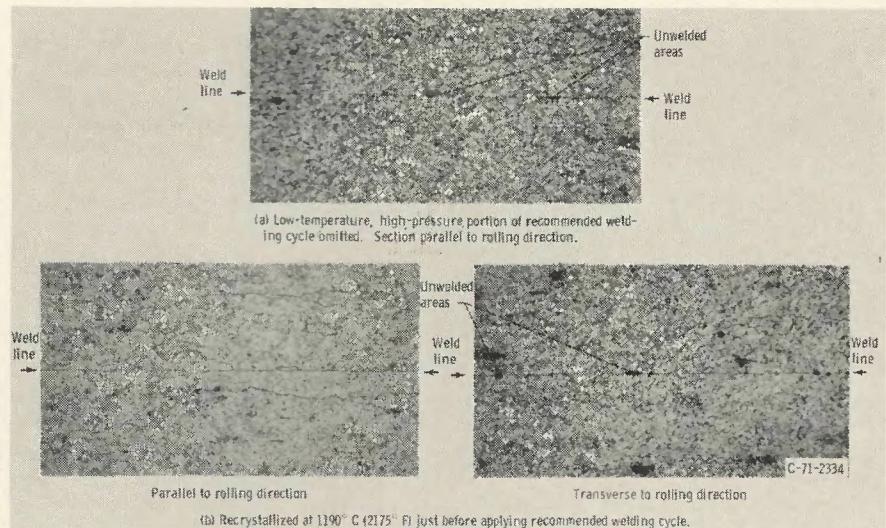


Fig. 11—Effect of variations from recommended weld cycle on weld quality. Welds were made in specially processed material with 600-grit sanded plus electropolished surfaces. Etchant, 100 milliliters H_2O , 2 grams CrO_3 and 10 milliliters H_2SO_4 (electrolytic, 3 V dc). Mag: 500X

sile-shear and creep-rupture shear strengths of the parent material at 1090°C (2000F). Previous work done without this method produced weak welds. With the enhanced diffusion welding methods, TD-NiCr sheet was diffusion welded in a vacuum hot press in a lap configuration. Specifically, the following conclusions are made about this welding method:

1. Specially processed TD-NiCr is preferred over commercial TD-NiCr for diffusion welding. The weld line can be eliminated with specially processed material and test specimen fracture takes place in the parent material away from the original interface. With commercial material, smooth fracture takes place at the weld line and tensile shear strength at 1090°C (2000F) is low.

2. The weld line can be eliminated when joining specially processed TD-NiCr by 600-grit sanding and electropolishing the faying surfaces prior to welding. The same surface preparation applied to commercial TD-NiCr prior to welding results in the presence of a weld line with a small degree of grain growth across the weld line. But the small, recrystallized

grains at the weld line that can decrease weld strength were eliminated.

3. A two-step weld cycle is preferred for diffusion welding TD-NiCr. The first step is the application of high pressure at relatively low temperature (207 MN/m² (30 ksi) and 705°C (1300F)) to provide micro-alignment and the intimate contact at the faying surfaces required for a solid-state weld to form. The second step consists of a low pressure, high temperature (15 MN/m² (2 ksi) and 1190°C (2175F)) diffusion heat treatment necessary for complete weld formation and grain growth across the original weld interface.

References

1. Saunders, N. T., "Dispersion-Strengthened Alloys for Space Shuttle Heat Shields," *Space Transportation System Technology Symposium, Vol. III, Structures and Materials*, NASA TM X-52876, 1970, pp. 159-174.
2. Moore, T. J., "Solid-State Welding of Dispersion-Strengthened Materials," *Aerospace Structural Materials*, NASA SP-227, 1970, pp. 119-134.
3. Yount, R. E., "Joining Techniques for Thorium Dispersion-Strengthened Materials," *SAMPE Proceedings*, Western Periodicals Co., Vol. 14, Article II-2B-1, 1968.
4. Moore, T. J. and Holko, K. H., "Solid-State Welding of TD-Nickel Bar," NASA TN D-5918, 1970.

Burrows and O'Keefe
(concluded from p. 63-s)
reliable process for fabricating hardware items where helium leak-tight joints are required.

Recommendations

It is recommended that braze alloy development be continued to develop commercial braze alloys for vacuum use that will not only be compatible with the base metal, but will also provide a flow temperature to permit the brazing operation to be accomplished at the solution treating or austenitizing temperature of the material. Preferred braze alloy flow temperatures for various materials follow:

Material	Desired flow temperature °F
Aluminum	
2014	935-950
2219	985-1010
Low alloy steels	
4130	1500-1600
4340	1500-1600
Maraging 18Ni250	1500-1600
Titanium 6Al-4V	1650-1725