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SOLID-STATE WELDING PROCESSES FOR AN OXIDE DISPERSION STRENGTHENED NICKEL-CHROMIUM-ALUMINUM ALLOY

Thomas J. Moore

Lewis Research Center

Cleveland, Ohio 44135

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16. Abstract

Three solid-state welding processes were evaluated for joining TD-NiCrAl (Ni-16Cr-4Al-2ThO_2) alloy sheet. Both hot-press and resistance spot welding techniques were successfully applied in terms of achieving grain growth across the bond line. Less success was achieved with a resistance seam welding process. In stress-rupture shear and tensile shear tests of lap joints at 1100° C, most failures occurred in the parent material, which indicates that the weld quality was good and that the welds were not a plane of weakness. The overall weld quality was not as good as previously attained with TD-NiCr, probably because the presence of alumina at the faying surfaces and the developmental TD-NiCrAl sheet, which was not of the quality of the TD-NiCr sheet in terms of surface flatness and dimensional control.

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CONTENTS

SOLID-STATE WELDING PROCESSES FOR AN OXIDE DISPERSION STRENGTHENED NICKEL-CHROMIUM-ALUMINUM ALLOY

by Thomas J. Moore

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SUMMARY

This study was designed to determine the feasibility of solid-state welding the oxide dispersion strengthened alloy, TD-NiCrAl (Ni-16Cr-4Al-2ThO_2). Lap welds were made in 0.4-millimeter sheet that was initially in both recrystallized and unrecrystallized conditions. Three welding processes were used: hot-press welding in vacuum, resistance spot welding in air, and resistance seam welding in argon. Weld quality was first evaluated using metallographic techniques to determine the extent of grain growth across the bond line. In assessing weld quality, microstructures of TD-NiCrAl weldments in the postheated (at 1315° C for 2-hr in H_2) condition were compared with those obtained in an earlier study for similar TD-NiCr weldments.

Mechanical testing involved stress-rupture shear tests primarily at 1100° C. Tensile shear tests were run at 1100° C and at room temperature. In analyzing the stress-rupture test results of lap joints in TD-NiCrAl, comparison was made with data obtained previously for TD-NiCr weldments.

The results of this study show that TD-NiCrAl sheet can successfully be diffusion welded using hot-press and resistance spot welding processes. Grain growth across the bond line was produced for both processes. However, the degree of elimination of the bond line was less complete than in previous studies on TD-NiCr weldments. Solid-state resistance seam welds in TD-NiCrAl showed some promise, although grain growth across the bond line was not achieved.

The stress-rupture shear strength of hot-press welds and resistance spot welds in TD-NiCrAl sheet at 1100° C was lower than that of similar TD-NiCr weldments made in a previous study. But since base-metal failure was involved, these lower strengths suggest a base-metal strength difference rather than a weld strength difference. The feasibility of producing a simulated heat shield using 0.4-millimeter TD-NiCrAl sheet was demonstrated. But optimum resistance spot welding parameters were not established for the heat shield.

INTRODUCTION

Oxide dispersion strengthened (ODS) nickel-base alloys are of interest for applications such as aircraft engine components and have been considered for re-entry heat shields of space vehicles (refs. 1 and 2). Previously, emphasis has been placed primarily on the Ni-20Cr-2ThO_2 compositions designated TD-NiCr (or DS-NiCr). More recently, additions of aluminum to produce alloys such as TD-NiCrAl (Ni-16Cr-4Al-2ThO_2) have been shown to improve oxidation resistance at temperatures near 1100° C (refs. 3 and 4). The stable surface oxides that are beneficial for oxidation resistance can be detrimental to joining in that the oxide can prevent the metal-to-metal interfacial contact required for solid-state welding processes.

This study was performed to evaluate the potential of several solid-state welding processes to join TD-NiCrAl sheet with emphasis on the enhanced-diffusion welding concept. The application of this concept to TD-NiCr (refs. 5 to 7) results in a solid-state weld without measurable deformation that is equal in strength to the base metal. No discontinuity in the base-metal microstructure is produced in the vicinity of the bond line. And thus, the enhanced-diffusion weld is indistinguishable from the base metal. In successful applications of the enhanced diffusion welding concept to TD-NiCr, a diffusion weld was made in unrecrystallized sheet by hot pressing or solid-state resistance spot welding under conditions that do not cause recrystallization. Postheating produced bulk recrystallization and grain growth across the bond line.

Three solid-state welding processes were evaluated in this study using lap joint configurations:

(1) Hot press welding (HPW)

(2) Resistance spot welding (RSW)

(3) Resistance seam welding (RSEW).

A portion of the resistance spot welding study involved the fabrication of a simulated heat shield to demonstrate that the TD-NiCrAl sheet could be formed and spot welded without visual evidence of forming or welding defects.

A number of lap joint specimens were prepared for each welding process to evaluate weld quality. Stress-rupture shear tests were run at 1100° and 1205° C. Tensile-shear tests were run at 1100° C and at room temperature. Failure modes of tested specimens were evaluated using metallography. Weld microstructure, strength, and failure modes for the TD-NiCrAl weldments were compared with results obtained earlier for TD-NiCr weldments. On the basis of the results of this study, conclusions are offered regarding the weldability of aluminum-modified ODS alloys such as TD-NiCrAl.

MATERIALS

The TD-NiCrAl sheet used in this study was a developmental product of 0.4-millimeter (nominal) thickness. Both recrystallized (fully processed sheet) and unrecrystallized sheet were used. Details of the sheet manufacture are described in reference 4. The unrecrystallized sheet grain structure is generally too fine to be resolved at a magnification of 500 (fig. 1(a)). Unrecrystallized sheet differs from recrystallized sheet only in that it has not received the final heat treatment that produces the recrystallized structure (fig. 1(b)). Unrecrystallized sheet was used because sheet in this processing condition is required to apply enhanced diffusion welding techniques.

Thickness variations for the 0.4-millimeter developmental TD-NiCrAl sheet of ±0.051 millimeter were about twice the amount measured (±0.025 mm) for commercial grade 0.4-millimeter TD-NiCr sheet. This greater thickness variation would tend to inhibit complete surface contact, which is a requirement for diffusion welding. Structural variations also were characteristic of the developmental sheet product in that there were differences in the recrystallized grain size and shape between heats as a result of the different processing procedures used (ref. 4). Heat numbers, chemistry, and processing condition of the TD-NiCrAl sheet for the various welding processes evaluated in this study are as follows:

Welding process	Heat	Code	Condition
Hot press welding	[a]3905	A-1	Unrecrystallized
Resistance spot welding:			
Single-spot welds	[a]3905	A-1	Unrecrystallized
Spot welded panel	[a]3905	B-2	Unrecrystallized
		2, 3	Recrystallized
Resistance seam welding	[b]3831	U	Unrecrystallized
		R	Recrystallized

[a]Ni-17.6 wt %Cr-3.7 wt %Al-2.2 wt %ThO_2 plus 418 ppm C and 43 ppm S.

[b]Ni-15.3 wt %Cr-4.0 wt %Al-2.3 wt %ThO_2 plus 430 ppm C and 30 ppm S.

WELDING PROCESSES AND EVALUATION

Three welding processes were evaluated: HPW, RSW, and RSEW. For all processes, emphasis was placed on the development of welding parameters that would result in the elimination of the bond line. As-received, 120-grit sanded surfaces were prepared for welding using either simple degreasing, chemical etching, or electropolishing. Earlier studies on diffusion welding of TD-NiCr had indicated that extensive grain growth could be produced across the bond line after faying surfaces were prepared by either electropolishing (ref. 5) or chemical etching (refs. 6 and 7). Such welds in TD-NiCr were as strong as the base metal in stress-rupture tests at 1100° C. Therefore, if similar grain growth across the bond line could be produced in TD-NiCrAl weldments, it was felt that base-metal strength would be achieved at the weld.

In establishing welding parameters, the lap joints were evaluated by metallography before mechanical testing. The weldments were examined to determine the quality of the solid-state welds and the extent of disruption in the microstructure (small grains at bond line, lack of grain growth across the bond line, etc.) at the weld. Metallographic specimens were etched electrolytically in a solution of 100 cubic centimeters of water, 2 grams of chromium oxide, and 10 cubic centimeters of sulfuric acid.

Once the maximum degree of grain growth across the bond line was achieved for each welding process, a number of weldments were made using the selected procedures and parameters. Before mechanical testing all weldments were postheated at 1315° C for 2 hours in hydrogen. Lap joints were used for all mechanical testing. These tests, conducted in air, included stress-rupture shear tests at 1100° and 1205° C and tensile shear tests at 1100° C and room temperature. The tensile shear tests were run at a crosshead speed of 1.3 millimeters per minute. Tested specimens were examined metallographically to determine the location and nature of the fractures. Each of the welding processes evaluated and details of the welding procedures are described in the following sections.

Hot Press Welding

Hot-press diffusion welded lap joints were made using only the unrecrystallized TD-NiCrAl sheet. The objective was to make diffusion welds at a temperature low enough to avoid recrystallization during welding and subsequently to postheat in order to produce bulk recrystallization and grain growth across the bond line.

Welding equipment. - Hot-press welding was conducted in a vacuum chamber evacuated to a pressure of 6.65×10^{-5} pascal (5×10^{-5} torr). A tantalum resistance heater was used to produce the welding heat. Sintered tungsten rams were used to transmit the welding force from a 220-kilonewton (25-ton) hydraulic press to the weld tooling.

Weld tooling was made from 3.2-millimeter-thick commercial TD-NiCr sheet. The weld tooling was designed with three protrusions so that three lap welds could be made simultaneously (see fig. 2). Three welds were made to increase the weld area so that enough welding force was required to allow use of existing hydraulic equipment above its minimum setting. This welding procedure had been used in an earlier study on TD-NiCr sheet (ref. 6).

Surface preparation. - The effect of surface preparation on weld quality was evaluated for several faying (mating) surface conditions. They included

(1) As received (degreased), 120-grit sanded surfaces

(2) Etched surfaces (buffered aqua regia, electrolytic)

(3) Electropolished surfaces using the procedure shown in figure 3.

Welding parameters. - Before making specimens for mechanical testing, a number of experimental hot-press welds were made to define the parameters required for producing quality diffusion welds. Weld quality as influenced by the welding variables were examined within the following ranges:

Temperature, ^{o}C	760 to 1100
Pressure, MPa (ksi)	41.4 to 207 (6 to 30)
Time at temperature, hr	1 to 6
Atmosphere	Vacuum
Deformation, percent reduction in thickness	0 to 6

Weld evaluation. - In addition to metallographic evaluation, lap-joint shear test specimens were prepared and punched from the weldments as shown in figure 2. Only the middle weld was tested as can be noted by the notching procedure shown in the top sketch in figure 2. The notching was designed to increase the shear stress on the weld plane by producing a 2t overlap (where t is the sheet thickness). A jig was used to hold the specimen. The notch was made with a file.

Resistance Spot Welding

In this part of the program, two RSW procedures involving the enhanced diffusion welding concept (refs. 6 and 7) were evaluated. One procedure, developed at Lewis, involved the preparation of single-spot welds in unrecrystallized sheet using welding parameters that would produce a diffusion weld without simultaneously recrystallizing the sheet. Postheating was used to produce bulk recrystallization and grain growth across the bond line. Once procedures and parameters were optimized, based on metallographic examination, a number of single-spot lap joints (see fig. 4) were prepared for mechanical testing.

Another RSW procedure was developed at the McDonnell-Douglas Corporation to fabricate a corrugation-stiffened, heat shield panel (simulated) using enhanced diffusion welding. In this variation of the enhanced diffusion welding concept, a material that is formable at room temperature, recrystallized TD-NiCrAl sheet, is formed into the corrugations. Unrecrystallized TD-NiCrAl sheet (which has poor formability) is used as the flat-face sheet. Ideally, spot diffusion welds are made between the corrugations and the face sheets in such a manner that no small recrystallized grains are formed at the bond line nor is bulk recrystallization produced in the unrecrystallized face sheet. Postheating is subsequently used to recrystallize the face sheet and eliminate the bond line by grain growth across the interface.

Welding machines. - A 400-kilovolt-ampere single-phase resistance welding machine was used to make the single-spot welds. For the panel fabrication study a 100-kilovolt-ampere, three-phase resistance welding machine was used. For both studies the spot welds were made in air using copper alloy electrodes.

Surface preparation. - For single-spot weldments as-received (120-grit sanded) specimens were electropolished using the procedure shown in figure 3. This surface preparation technique was used exclusively because of earlier favorable results obtained in the hot-press weldments. In the panel fabrication by the McDonnell-Douglas Corp., the sheet surfaces were cleaned and descaled using the procedure shown in figure 5.

Welding parameters. - Several experimental welds were made in developing parameters for single-spot welds. The results of variables evaluated were

Type of machine	Single phase
Welding atmosphere	Air
Number of welding heat cycles	1/2 to 7
Welding current, kA	14.9 to 28.4
Pneumatic force, kN (lb)	9.9 to 12.7 (2220 to 2860)
Approximate welding pressure (assuming 4.4-mm-diam spot weld), MPa (ksi)	646 to 841 (93.6 to 122.0)

Before fabricating the spot-welded panel, a cursory evaluation of welding parameters was made with the following variables:

Type of machine	Three phase
Welding atmosphere	Air
Number of weld impulses	1 or 2
Welding current (phase shift), percent	0 to 5

Weld evaluation. - Single spot lap joints of the type shown in figure 4 were used for metallographic evaluation and shear testing. The spot size was large compared with the sheet thickness: the ratio of the spot-weld diameter to the sheet thickness was about 11 to 1.

Sections were also taken through some of the welds in the spot welded panel to evaluate whether the goal of high-quality solid-state spot welds was achieved.

Resistance Seam Welding

The modified resistance seam welding (RSEW) process used by Solar to make the lap welds is a proprietary process developed by the Solar Division of International Harvester Company (ref. 8). The full thickness of the lap joint is held at the welding temperature for several tenths of a second, and sufficient pressure is applied to produce macrodeformation. Thus, this is essentially a deformation welding process rather than a diffusion welding process wherein no macrodeformation is produced.

Five lap-joint weldments were made, each with 150 to 300 lineal millimeters of weld. They included weldments in both recrystallized/recrystallized and unrecrystallized/unrecrystallized combinations.

Welding machine. - A programmed resistance seam welding machine was used to provide the welding heat. The seam welds were made for the full width of the overlapped sheet in an inert (argon) atmosphere using refractory metal wheels. The lap joints were normal to the rolling direction of the sheet.

Surface preparation. - After preliminary trails involving the use of hot alkaline cleaning and electrolytic polishing, satisfactory cleaning of the recrystallized sheet was ultimately achieved with the following two-step procedure:

(1) Remove the surface oxide using a 100-grit silicon carbide abrasive paper and follow by sanding with 400-grit silicon carbide paper.

(2) Electrolytic polish (with a proprietary polish).

For unrecrystallized sheet only step (2) was used on the as-received sheet surfaces.

Welding parameters. - The lap weldments were set up for a nominal 5t overlap. Temperature was programmed from a special power supply. Time-at-temperature was determined by the welding speed. The lap joints for mechanical testing were made at a rate of about 200 millimeters per minute and a squeeze force of 1.38 kilonewtons (310 lb). Welding current was about 65 amperes. The welding atmosphere was argon.

Weld evaluation. - Two types of specimens were used in mechanically testing the lap joints. For the room temperature and 1205° C tests conducted at Solar, bowtie shaped specimens, with 12.7-millimeter-wide ends, 6.3-millimeter-wide by 25.4-millimeter-long gage sections, and a 114-millimeter overall length, were used. For the 1205° C specimens, resistance-welded tabs were attached to extend the length to 254 millimeters for a cold-grip test apparatus. For tests run at 1100° C at Lewis the specimen designs shown in figure 6 were used. The shear stress at the weld produced with a 5t overlap (fig. 6(a)) was rather low (1/5 of the tensile stress). Failure at the weld plane was much more likely to occur with the 2t overlap notched specimen shown in figure 6(b). With this notched specimen, however, end effects of the weld were removed.

RESULTS

In this section, the metallographic analyses of weldments produced by HPW, RSW, and RSEW are presented. For the RSW process evaluation of both single-spot welds and a spot welded panel are included. The results of mechanical testing of lap joints in stress-rupture shear and tensile shear are also shown. Included is metallographic examination of the fractures.

As previously noted, the TD-NiCrAl sheet used in this study was a developmental product having about 60 to 75 percent of the strength of TD-NiCr sheet (ref. 4). The low creep strength of the developmental TD-NiCrAl was not sufficient to preclude bending of the lap-welded test specimens during the stress-rupture tests. Thus, loading the specimen produced bending in the test area such that the lap welds were subjected to a combination of shear and tensile stresses. Comparisons were made, however, of TD-NiCrAl weldment strength and failure mode from this study with data from previous studies of TD-NiCr weldments. These will be discussed later.

Hot Press Weldments

Microstructure. - Considering the maximum extent of grain growth across the bond line, best results were obtained using unrecrystallized sheet with electropolished surface preparation and the following HPW parameters:

Atmosphere	Vacuum
Temperature, $^{\circ}$C	990
Pressure, MPa (ksi)	69.0 (10.0)
Time, hr	4

A typical microstructure (from run 1, table I) that was achieved using these welding parameters and a postheating condition of 1315° C for 2 hours in hydrogen is shown in figure 7. No measurable deformation was produced at the weld. The objective of producing grain growth across the bond line was achieved, and small grains at the bond line were avoided. In the earlier studies on TD-NiCr, similar but more extensive grain growth was produced across the bond line (refs. 5 and 6). The lack of complete movement of the bond line grain boundary across the weld interface in TD-NiCrAl weldments is probably associated with the presence of alumina at the faying surfaces, which would tend to inhibit grain growth across the bond line. All lap joints for mechanical property determinations were made using these parameters (run 7, table I).

Other welding parameters evaluated are also listed in table I. The principal variables were faying surface preparation and HPW parameters.

Preparation of the faying surfaces of the unrecrystallized TD-NiCrAl sheet was an important factor in minimizing the extent of small grain formation at the bond line. This effect is shown in figure 8 for hot-press welds made at 1100° C and postheated at 1315° C for 2 hours in hydrogen (table I, runs 1 to 3). A continuous weld line with small grains on both sides of the weld interface are evident in figure 8(a) for degreased, as-received, 120-grit sanded surfaces. An etched surface preparation (buffered aqua-regia, electrolytic) resulted in fewer small grains at the weld interface (fig. 8(b)). Use of the electropolishing procedure (fig. 3) eliminated the small grains (see fig. 8(c)). Similar beneficial effects of electropolishing had been shown previously for diffusion weldments in TD-NiCr sheet (ref. 5).

Once the electroplishing procedure was established, HPW variables were evaluated in an attempt to maximize the extent of grain growth across the bond line. The HPW variables (table I, runs 3 to 14) and their ranges were

Welding temperature, °C	760 to 1100
Welding pressure, MPa (ksi)	41.4 to 207 (6 to 30)
Time at temperature, hr	1 to 6
Atmosphere	Vacuum
Deformation, percent reduction in thickness	0 to 6

The effect of HPW variables on weld microstructure is shown in figure 9 for four welds made between 870° and 1100° C (table I, runs 3, 6, 10, and 12). Good weld quality was obtained for all four runs, with the weld made at 990° C and 69.0 megapascals (10 ksi) for 2 hours being slightly superior to the others, based on grain growth across the bond line.

Further studies were conducted at the 990° C welding temperature. In general, the most satisfactory results (fig. 7) were obtained with a welding time of 4 hours. Run 7, welded under these conditions, was considered the optimum setting for the preparation of lap welds for mechanical testing. No improvement in weld quality was observed with a 6-hour weld time (table I, run 8).

Attempts to produce high quality hot-press welds below 960° C were unsuccessful except for run 12, which was made at 870° C and 138 megapascals (20 ksi) for 2 hours. Other runs at 930°, 870°, and 760° C resulted in either poor quality welds or no welding at all, even with 5 and 6 percent deformation (table I, runs 11, 13, and 14).

Mechanical properties. - Results of tests conducted on notched, lap joint specimens are shown in table II. The 1100° C stress-rupture shear data are plotted in figure 10. For comparison, similar data (from ref. 6) are shown for TD-NiCr hot-press weldments that were also made in unrecrystallized sheet and postheated to produce bulk recrystallization. All but one of these weldments, in both materials, failed in the base metals, indicating that the welds were not a plane of weakness. One of the TD-NiCrAl weldments failed at the weld plane on heating (see table II). The reason for this anomaly is unknown but dimensional variations in the developmental sheet product may have prevented the intimate surface contact which is essential for diffusion welding.

On the basis of the stress-rupture data shown in figure 10, the weldment strengths in TD-NiCr and TD-NiCrAl sheet are similar for periods up to about 30 hours (ignoring the one TD-NiCrAl weldment that failed on heating). But for longer times the TD-NiCr weldments are stronger. This strength difference is believed to be a result of less bending moment at the weld in the TD-NiCr tests because of the greater creep strength of TD-NiCr base metal. Relative weld joint strengths could not be determined because failures took place away from the weld for both materials.

In duplicate room-temperature tensile-shear tests of the TD-NiCrAl welds, failure took place in the base metal at a calculated shear stress of 292 megapascals (42.3 ksi) in the weld (table II).

Single-Spot Weldments

Microstructure. - On the basis of metallographic evaluation, the best RSW results in the single-spot test specimen study were obtained using a welding current of one cycle and a pneumatic force of 12.7 kilonewtons (2860 lb). The parameters used to produce the optimum one-cycle weld are shown in table III. Weld quality was good as judged by grain growth across the bond line, and the absence of small grains at the bond line (fig. 11). Spot welds with more extensive grain growth across the bond line with complete elimination of the initial interface had been made earlier in 0.4-millimeter-thick TD-NiCr sheet (ref. 7). This probably reflects the absence of alumina at the faying surfaces of the TD-NiCr welds.

The settings shown in table III were used to produce the single-spot lap joints in TD-NiCrAl sheet for mechanical testing. No macrodeformation was produced at the spot welds.

In the spot welding parameter development, a distinct microstructural pattern was observed and related to the welding parameters used. A summary of these results is shown in table IV. The major welding variables are listed along with a sketch of the microstructures in the postheated condition (1315° C for 2 hr in H_2). In the lower-pneumatic-force series (9.87 kN or 2220 lb), a clear trend was observed as the amperage was decreased. The molten zone was reduced in size and then eliminated. Reduction of the fine-grained recrystallized zone followed a similar pattern. Since the objective was to produce a diffusion weld without melted regions or small recrystallized grains, the higher-pneumatic-force series (12.7 kN or 2860 lb) was conducted. At the highest amperage both melting and a fine-grained recrystallization zone was produced. But, as the amperage was reduced, the melted zone and fine-grained region were eliminated. The weld made at 22.9 kiloamperes produced the desired structure with grain growth across the bond line.

In addition to the one-cycle welds, experimental single-spot welds were also made for one-half, three, and five cycle weld times. Less satisfactory results were achieved using these weld times in regard to weld quality and reproducibility.

Mechanical properties. - Stress-rupture shear and tensile shear data for the single-spot welds are presented in table V. Two of eleven single-spot test specimens fell apart before testing. Dimensional variations in the developmental TD-NiCrAl sheet and possible alumina on the faying surfaces are felt to be responsible for this anomaly.

For the 1100° C stress-rupture shear tests (table V), failure occurred at the weld plane for one weldment and in the base metal for two other weldments. One test specimen was unloaded before failure. The two base-metal failures were of the button pull-out type and occurred due to a combination of bending and tensile stresses that were produced by joint rotation (fig. 12). In previous tests of solid-state spot welds made in unrecrystallized TD-NiCr sheet, failure always occurred in the base metal but not by button pullout (ref. 7). For the TD-NiCr specimens bending did not occur in the test area because of the excellent creep-rupture strength of the sheet. A plot of weld plane shear stress against time to rupture at 1100° C is shown in figure 13. Also shown (from ref. 7) are comparable data for solid-state spot welds that were made in unrecrystallized 0.4 millimeter TD-NiCr sheet. The higher stress-rupture shear strength of the TD-NiCr weldments is apparent.

One 1100° C tensile shear test specimen exhibited a button failure in the base metal at a weld shear stress of 43.5 megapascals (6.3 ksi). (See table V). A duplicate test specimen failed at 29.7 megapascals (4.3 ksi). In triplicate room-temperature tensile shear tests failure occurred by button pullout at an average calculated weld shear stress of 266 megapascals (38.5 ksi).

Spot Welded Panel

Fabrication of a simulated heat shield panel was successfully accomplished in this cursory study in that the material was formed and welded without evidence of external defects. A photograph of the welded panel is shown in figure 14.

Twelve cross sections of spot welds taken from the welded panel revealed that the goal of producing high quality, solid-state spot welds was not achieved. Seven of the welds showed various degrees of melting, even though all welds were made using the same settings (table VI). A cross section of a portion of a fusion spot weld appears in figure 15(a). The solid-state spot weld in figure 15(b) is indicative of a fine-grained recrystallized nugget of the type sketched in table IV. Further work would be necessary to eliminate melting and produce solid-state spot welds without fine grains in the vicinity of the weld.

Microstructure. - A summary of the parameters used to prepare the lap-joint weldments are shown in table VII. Weld quality was reasonably good in runs 1 (recrystallized sheet) and 2 (unrecrystallized sheet) in that continuous bond lines were evident, but the bond lines were virtually free of small grains. However, there was no grain growth across the bond line in either case. Thus, the weld interface was essentially a planar grain boundary.

Run 4 (unrecrystallized sheet) had excellent surface appearance (fig. 16) and a 5t overlap. The average deformation at the weld joint was 11 percent. Examination of a cross section from this weldment (fig. 17) shows a fine-grained, partially recrystallized structure with many long, thin grains. No grain growth across the bond line is evident and the bond line is difficult to distinguish from other parallel grain boundaries.

The lap joint (run 5, recrystallized sheet) shown in figure 18 had an unintentional 3t overlap and some defective areas. Thus, as shown in figure 19(a), excessive deformation was produced (21 percent). The bond line (fig. 19(b)) is a continuous grain boundary containing a few small grains. Base-metal grain size and shape were unexpectedly different for the recrystallized sheet (fig. 19) compared with the initially unrecrystallized sheet (fig. 17) in the heat-treated condition. The reason for the difference in grain structure is probably related to different processing conditions used to produce the sheet material.

Mechanical properties. - Stress-rupture shear and tensile shear data are shown in table VIII for both recrystallized and unrecrystallized starting material. The 1205° C stress-rupture shear tests all were conducted using unnotched 5t overlap weldments. Fracture occurred in the base metal, adjacent to the weld for each test. This would be expected because the weld shear stresses were low due to the 5t overlap.

For the 1100° C stress-rupture shear tests, most of the specimens failed in the base-metal adjacent to the weld or at the base of the notch (figs. 20 and 21). Two of the notched specimens failed partly at the weld, and one unnotched 3t overlap specimen failed at the weld interface. Note that the unnotched 5t weldments failed in the base metal at lower stress levels. (See table VIII.) Apparently, the combined bending and tensile stresses produce the greatest weakening of the base metal adjacent to the weld with the 5t overlap.

With the 5t overlap and in all tensile shear tests run at 1093° C, failure occurred in the base metal. The average weld shear stress for duplicate tests in initially unrecrystallized weldments was 15.9 megapascals (2.3 ksi) and for duplicate weldments in recrystallized sheet it was slightly lower, or 12.8 megapascals (1.9 ksi).

Room-temperature tensile-shear test specimens also had a 5t overlap. As opposed to all other mechanical testing in this program, these specimens were tested in the as-welded condition. All failures were located in the base metal. (See table VIII.)

Average weld shear stress at rupture was 164 megapascals (23.8 ksi) for four tests in unrecrystallized sheet. For duplicate tests in recrystallized sheet the weld shear stress was slightly lower, 144 megapascals (20.9 ksi).

DISCUSSION

The enhanced diffusion welding concept, previously shown to be applicable to TD-NiCr sheet, is also applicable to 0.4-millimeter-thick TD-NiCrAl sheet. In this program the feasibility of using the enhanced diffusion welding concept for TD-NiCrAl sheet was demonstrated using either vacuum hot pressing or resistance spot welding in air. The weldability of TD-NiCrAl sheet was, however, not as good as TD-NiCr in that a lesser extent of grain growth across the bond line was achieved. In addition, some inconsistency was noted in that several TD-NiCrAl weldments fell apart before testing.

In stress-rupture shear tests at 1100° C and tensile shear tests at 1100° C and room temperature, failure almost always took place in the base metal. This indicated that the diffusion welds in TD-NiCrAl sheet were of good quality and not a plane of weakness. Hot-press weldments in TD-NiCrAl sheet were somewhat weaker than similar weldments in TD-NiCr. Resistance spot weldments in the TD-NiCrAl sheet were significantly weaker in stress-rupture shear tests. But, since base-metal failure was involved, these lower strengths suggest a base-metal strength difference rather than a weld strength difference. The feasibility of producing a simulated heat shield in 0.4-millimeter-thick TD-NiCrAl sheet was demonstrated, but optimum resistance spot welding parameters were not established.

Solid-state welds, produced in TD-NiCrAl sheet using a modified resistance seam welding process, showed less promise than the hot-press and resistance spot diffusion welds because the bond line was a stable grain boundary with no grain growth across the weld interface. Because most of the stress-rupture shear and tensile shear specimens tested had 5t overlaps, the welds were not highly stressed, and failures occurred in the base metal. Lap joints notched to a 1t or 2t overlap and tested at 1100° C also failed partly or entirely in the base metal. Because this seam welding process was not applied to TD-NiCr sheet, an assessment of the relative strengths of TD-NiCrAl and TD-NiCr weldments could not be made.

SUMMARY OF RESULTS AND CONCLUSIONS

1. The feasibility of joining 0.4-millimeter-thick TD-NiCrAl sheet by enhanced diffusion welding techniques was demonstrated. The solid-state welds can be made by hot pressing in vacuum or by resistance welding in air. These solid-state welding

techniques are also believed to be applicable to other aluminum containing dispersion-strengthened alloys.

2. As judged by the extent of grain growth across the bond line, best diffusion welding results were obtained using the following process conditions and postheating at 1315° C:

a. Hot press welding conditions -

Temperature, °C	990
Pressure, kN (ksi)	69.0 (10.0)
Time, hr	4
Atmosphere	Vacuum

b. Resistance spot welding conditions -

Electrodes:	
Material	Copper
Class	III
Diameter, mm	12.7
Dome radius, mm	203
Pneumatic force, kN (lb)	12.7 (2860)
Percent heat	21
Number of heat cycles	1 (60 Hz)
Peak welding current, kA	22.9
Atmosphere	Air

3. Solid-state resistance seam welds were also successfully produced in TD-NiCrAl sheet, but grain growth across the bond line was not achieved.

4. The elevated temperature strength of TD-NiCrAl weldments as obtained from the stress-rupture tests at 1100° C was generally lower than simular TD-NiCr weldments.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, December 9, 1974,
505-01.

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3. Centolanzi, Frank J.; Probst, Herbert B.; Lowell, Carl E.; and Zimmerman, Norman B.: Arc Jet Tests of Metallic TPS Materials. Presented at the Space Shuttle Mater. Conf., Huntsville, Ala., Oct. 5-7, 1971.

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8. Continuous Seam Diffusion Bonding Process. U.S. Patent 3,644,698, February 22, 1972.

TABLE I. - VACUUM HOT-PRESS WELDING PARAMETERS USED FOR UNRECRYSTALLIZED 0.4-MILLIMETER-THICK TD-NiCrAl SHEET

[Surface preparation, electropolishing (see fig. 3) unless otherwise noted; chamber pressure, 5×10^{-5} torr.]

Run	Temperature, °C	Pressure		Time, hr	Deformation, percent reduction in thickness	Microstructure[a]
		MPa	ksi			
[b]1	1100	41.4	6	1	None	Small grains at bond line
[c]2	1100	41.4	6	↓	----	Small grains at bond line
3	1100	41.4	6	↓	----	Grain growth across bond line
4	990	55.2	8	↓	None	↓
5	↓	69.0	10	↓	----	↓
6	↓	↓	↓	2	2	↓
7	↓	↓	↓	4	None	↓
8	↓	↓	↓	6	2	↓
9	↓	82.7	12	1	3	↓
10	960	69.0	10	2	None	↓
11	930	103	15	↓	[d]6	Fell apart (no welding)
12	870	138	20	↓	6	Grain growth across bond line
13	870	103	15	↓	----	No grain growth across bond line
14	760	207	30	↓	[d]5	Fell apart (no welding)

[a]After Postheating (at 1315° C for 2 hr in H_2).
[b]Degreased only.
[c]Etched in buffered aqua regia.
[d]Estimated.

TABLE II. - HOT-PRESS WELDMENT TEST DATA

[Lap joints in 0.4-mm unrecrystallized TD-NiCrAl sheet notched to 2t overlap as shown in fig. 2; all weldments postheated at 1315° C for 2 hr in H_2.]

Base metal tensile stress		Weld plane shear stress		Hours to rupture	Failure location
MPa	ksi	MPa	ksi		
1100° C Stress-rupture shear tests					
36.6	5.3	18.6	2.7	29.0	Base metal
31.1	4.5	15.9	2.3	161	Base metal
28.3	4.1	14.5	2.1	47.8	Base metal
22.8	3.3	11.7	1.7	239+	(a)
22.8	3.3	11.7	1.7	-----	Weld shear on heating
Room-temperature tensile shear tests					
577	83.6	288	41.8	-----	Base metal
591	85.6	296	42.8	-----	Base metal

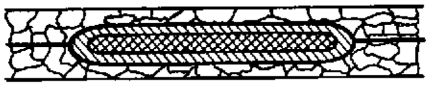
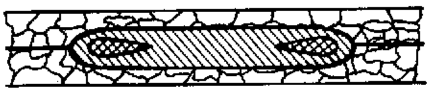
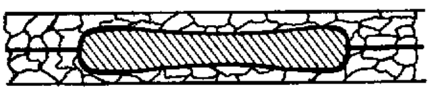
[a]Test stopped; base metal and weld failure on cooling.

TABLE III. - RESISTANCE SPOT WELDING SCHEDULE FOR SINGLE SPOT LAP WELDS IN 0.4-MILLIMETER-THICK TD-NiCrAl SHEET

[Welds made in air.]

Base metal	Specially processed (unrecrystallized) condition
Faying surface preparation	Electropolished
Welding machine	Single phase, 400 kVA
Welding electrodes	Class III copper alloy, 12.7-mm diam., 203-mm-rad.
Pneumatic force	12.7 kN (2860 lb)
Control settings:	
Percent heat	21
Number of squeeze cycles	120
Number of heat cycles	1
Number of hold cycles	30
Peak welding current	22.8 to 23.1 kA
Deformation at weld, % Δt	None
Spot diameter	11 t (approximately 4.4 mm)
Postheating	1315° C for 2 hr in H_2

TABLE IV. - EFFECTS OF WELDING CURRENT AND PNEUMATIC PRESSURE ON ONE-CYCLE (60 HZ) RESISTANCE SPOT WELDS IN UNRECRYSTALLIZED TD-NiCrAl SHEET

Welding current, peak kA	Deformation, percent reduction in thickness	Microstructure of postheated[a] weldments	
Pneumatic force, 9.87 kN (2220 lb)			
26.3	11		Fusion nugget enclosed within fine-grained recrystallized "nugget."
25.1	2		Donut-shaped fusion nugget within fine-grained recrystallized nugget.
23.6	2		Fine-grained recrystallized nugget
21.9	1		Tapered donut-shaped recrystallized nugget
Pneumatic force, 12.7 kN (2860 lb)			
28.4	3		Fusion nugget enclosed within fine-grained recrystallized nugget
26.0	3		Fine-grained recrystallized nugget
22.9	None		Grain growth across bond line with very few small grains at bond line

[a]Postheated at 1315° C for 2 hr in H_2.

TABLE V. - RESISTANCE SPOT WELDMENT TEST DATA

[Single spot lap joints in 0.4-mm-thick unrecrystallized TD-NiCrAl sheet (fig. 4); spot diameter, 4.36 mm; weldment postheated at 1315° C for 2 hr in hydrogen.]

Base-metal tensile stress		Weld plane shear stress		Hours to rupture	Failure location
MPa	ksi	MPa	ksi		
1100° C Stress-rupture shear tests					
40.0	5.8	26.9	3.9	0.1	Shear at weld
29.5	4.3	20.0	2.9	8.9	Base metal (button)
24.2	3.5	16.6	2.4	44.6	Base metal (button)
20.7	3.0	13.8	2.0	312+	Unloaded before failure
1100° C Tensile shear tests					
64.9	9.4	43.5	6.3	-----	Base metal (button)
44.1	6.4	29.7	4.3	-----	Shear at weld
Room-temperature tensile shear tests					
360	52.2	242	35.1	-----	Base metal (button)
409	59.3	276	39.9	-----	Base metal (button)
415	60.2	280	40.5	-----	Base metal (button)

TABLE VII. - RESISTANCE SEAM WELDING[a] SCHEDULES FOR LAP-JOINT WELDMENTS IN 0.4-MILLIMETER-THICK TD-NiCrAl SHEETS

Base metal:	
Runs 1, 3, and 5	Recrystallized sheet
Runs 2 and 4	Unrecrystallized sheet
Faying surface preparation	Electropolishing (proprietary "Summa" polish)
Lap joint overlap	5 times sheet thickness (nominal)
Machine	Programmed resistance seam welding machine
Welding atmosphere	Argon
Welding wheel material	Molybdenum
Pneumatic force	1.38 kN (310 lb)
Welding current:	
Runs 1 to 3	60 to 75 A
Runs 4 and 5	65 A
Welding speed	200 mm/min
Postheating:	
Runs 1 to 3	1315° C for 1 hr in argon
Runs 4 and 5	1315° C for 2 hr in hydrogen

[a]Proprietary process developed by Solar Division of International Harvester Corp. (see ref. 8).

TABLE VI. - RESISTANCE SPOT WELDING SCHEDULE FOR SIMULATED HEAT SHIELD PANEL IN 0.4-MILLIMETER-THICK TD-NiCrAl SHEET

[Welds made in air.]

Base metal	Unrecrystallized sheet for flat face of panel and recrystallized sheet for the corrugations
Faying surface preparation	Clean in descaling solution (see fig. 7)
Welding machine	100 kVA, three-phase machine
Welding electrodes:	
Top	6.35-mm-diam. by 50.8-mm tip rad.
Bottom	15.9-mm-diam. by 305-mm tip rad.
Pneumatic force	8.9 kN (2000 lb)
Control settings:	
Percent heat	6
Number of heat cycles	4
Number of cool cycles	1
Number of impulses	1
Postheating conditions	1315° C for 2 hr in H_2

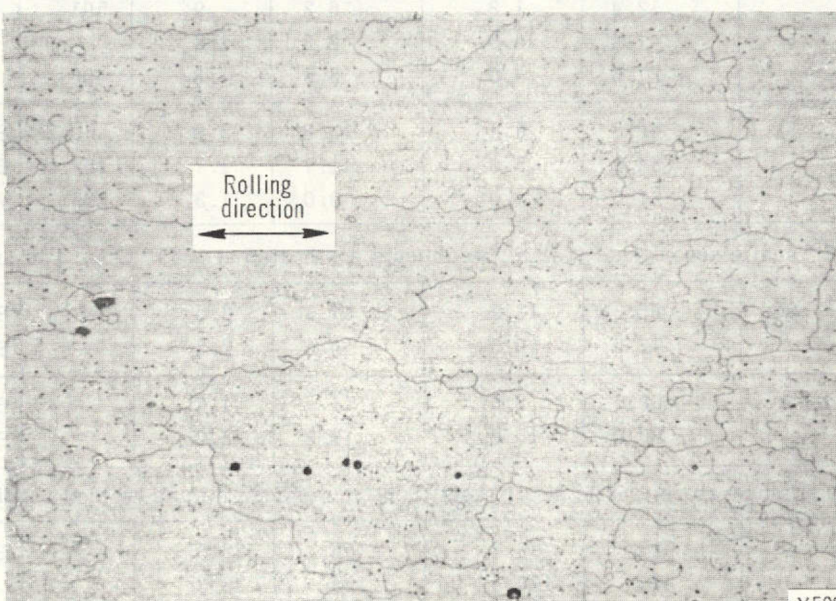
TABLE VIII. - RESISTANCE SEAM WELDMENT TEST DATA

[Lap joints in 0.4-mm-thick TD-NiCrAl sheet.]

Initial base metal condition	Overlap	Base-metal tensile stress		Weld plane shear stress		Hours to rupture	Failure location
		MPa	ksi	MPa	ksi		
1205° C Stress-rupture shear tests (Solar); weldments postheated at 1315° C for 1 hr in argon)							
Unrecrystallized	5t	44.9	6.5	9.0	1.3	0.1	Base metal
		34.5	5.0	6.9	1.0	72.2	Base metal
Recrystallized	5t	51.7	7.5	10.4	1.5	0.2	Base metal
		51.7	7.5	10.4	1.5	1.6	↓
		38.0	5.5	7.6	1.1	2.6	
		31.1	4.5	6.2	.9	11.1	
		24.2	3.5	4.8	.7	79.7	
1100° C Stress-rupture shear tests (Lewis); weldments postheated at 1315° C for 2 hr in hydrogen							
Unrecrystallized	[a]1t	12.4	1.8	12.4	1.8	429+	Unloaded
		9.7	1.4	9.7	1.4	600	Base metal and weld
	[a]2t	27.6	4.0	13.8	2.0	5.0	Base metal and weld
		19.3	2.8	9.7	1.4	22.0	Base metal
		12.4	1.8	6.2	.9	501	↓
	5t	41.4	6.0	8.3	1.2	.9	
		27.6	4.0	5.5	.8	406	
Recrystallized	3t	40.7	5.9	13.8	2.0	50.2	Base metal
		34.5	5.0	11.7	1.7	167	Base metal
		27.6	4.0	9.0	1.3	265	Weld interface
1093° C Tensile shear tests (Solar); weldments postheated at 1315° C for 1 hr in argon							
Unrecrystallized	5t	86.3	12.5	17.3	2.5	-----	Base metal
		72.5	10.5	14.5	2.1	-----	Base metal
Recrystallized	5t	58.6	8.5	11.7	1.7	-----	Base metal
		69.0	10.0	13.8	2.0	-----	Base metal
Room-temperature tensile shear tests (Solar); weldments in as-welded condition							
Unrecrystallized	5t	915	132.5	155	22.5	-----	Base metal
		874	126.5	175	25.3	-----	↓
		849	123.0	170	24.6	-----	
		780	113.0	156	22.6	-----	
Recrystallized	5t	766	111.0	155	22.5	-----	Base metal
		666	96.5	133	19.3	-----	Base metal

[a]Notched.

(a) As-received, unrecrystallized condition.



(b) Recrystallized at 1315° C for 2 hours in hydrogen.

Figure 1. - Base metal microstructure for 0.4-millimeter-thick TD-NiCrAl sheet. Sulfuric-chromic acid etch.

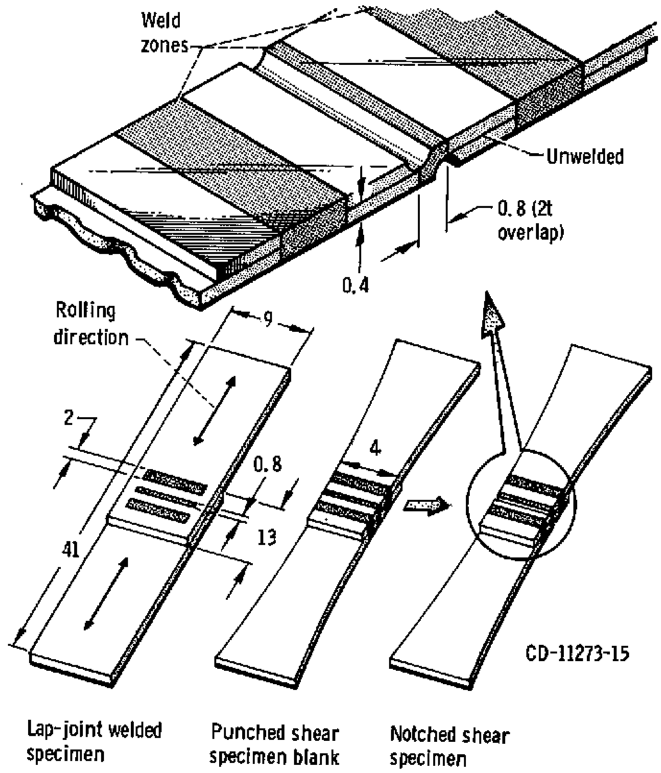


Figure 2. - Configuration of hot-press weldment showing how specimens for tensile shear and stress-rupture shear tests were obtained. (Dimensions are in mm).

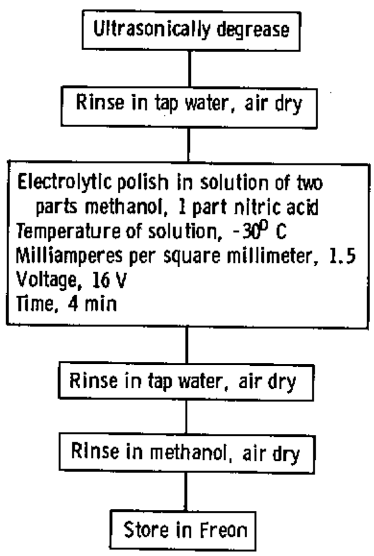


Figure 3. - Electropolish surface preparation procedure which was used before hot-press welding.

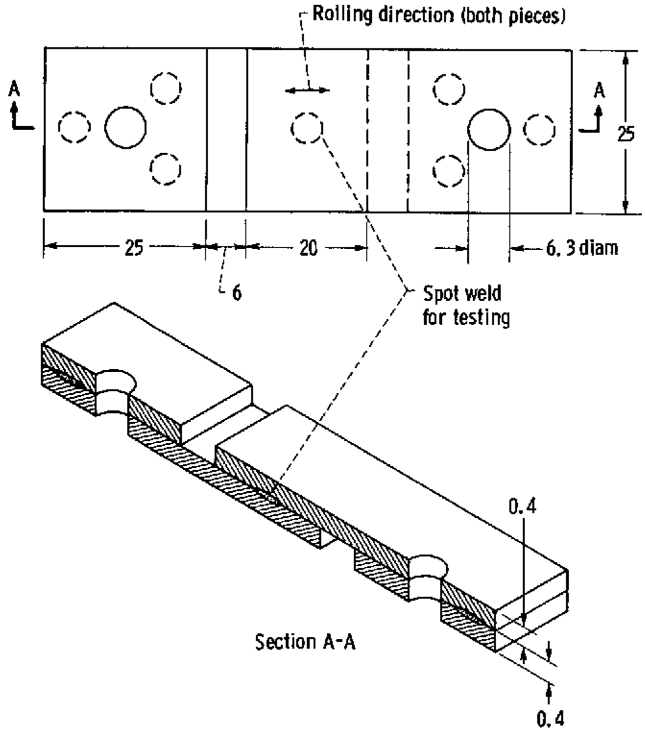


Figure 4. - Resistance spot-weld lap joint specimen used for tensile shear and stress-rupture shear tests. Reinforcing tabs are spot welded to specimen to prevent failure at pin holes. Dimensions are in millimeters. Section A-A was used for metallographic examination of weld.

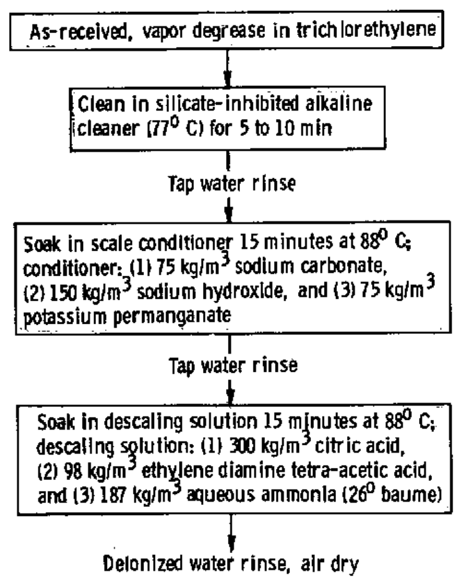
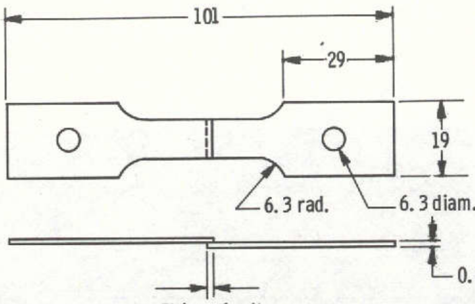
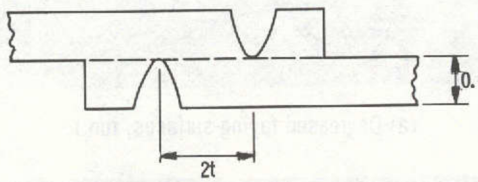


Figure 5. - Cleaning and descaling procedures used before heat shield panel fabrication by resistance spot welding.



(a) Lap joint test specimen.



(b) Lap joint notched to 2t overlap.

Figure 6. - Tensile shear and stress-rupture shear test specimens used to test resistance seam welded joints. (Dimensions are in millimeters.)

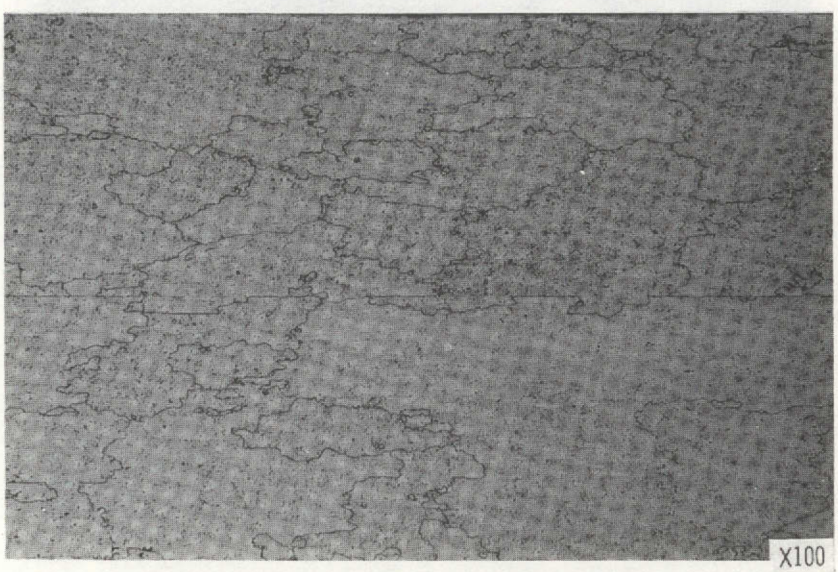
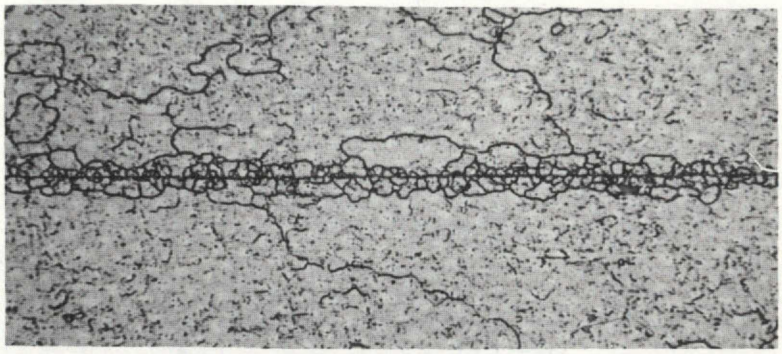
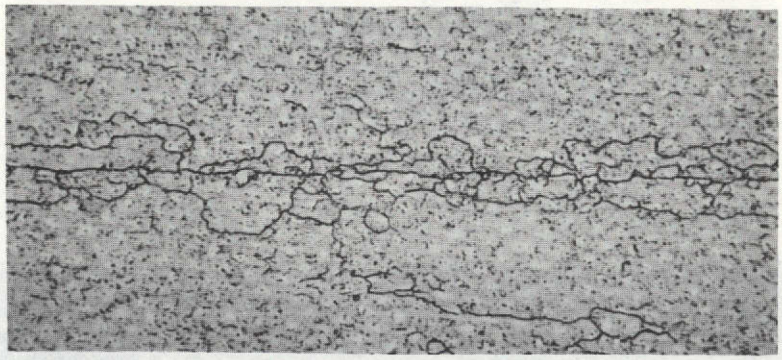


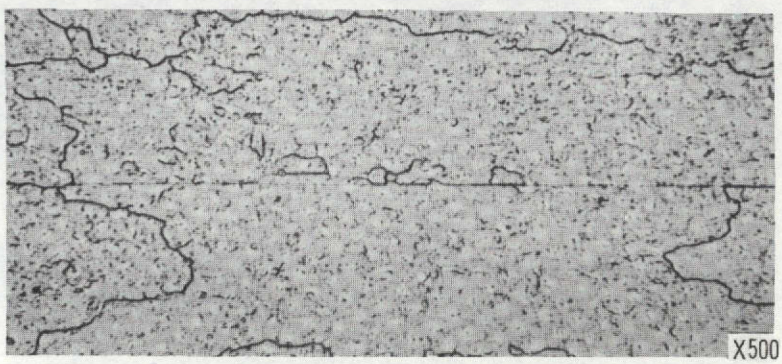
Figure 7. - Typical microstructure of hot-press lap welds in TD-NiCrAl sheet produced for mechanical testing. Welding conditions: temperature, 990° C; pressure 69.0 megapascals (10 ksi); time, 4 hours, in vacuum. Weldments postheated at 1315° C for 2 hours in hydrogen.



(a) Degreased faying surfaces, run 1.

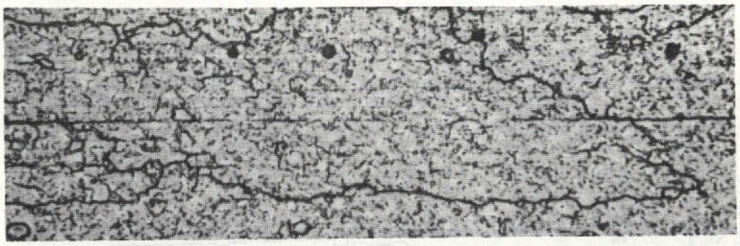


(b) Etched faying surfaces, run 2.

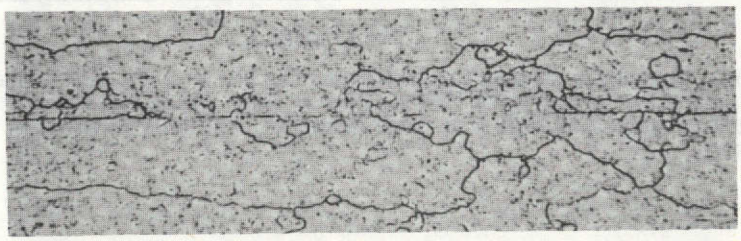


(c) Electropolished faying surfaces, run 3.

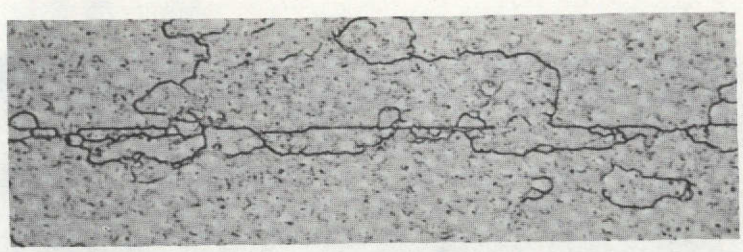
Figure 8. - Effect of surface preparation on microstructure of hot-press welds in TD-NiCrAl sheet. Welded at 1100° C and 41.4 megapascals (6 ksi) for 1 hour in vacuum and postheated at 1315° C for 2 hours in hydrogen.



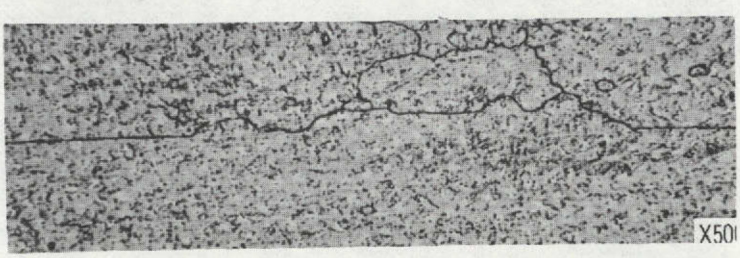
(a) Run 3 welding conditions: temperature, 1100° C; pressure, 41.4 megapascals (6 ksi); time, 1 hour.



(b) Run 6 welding conditions: temperature, 990° C; pressure, 69 megapascals (10 ksi); time, 2 hours.



(c) Run 10 welding conditions: temperature, 960° C; pressure, 69 megapascals (10 ksi); time, 2 hours.



(d) Run 12 welding conditions: temperature, 870° C; pressure, 138 megapascals (20 ksi); time, 2 hours.

Figure 9. - Effect of vacuum hot-press welding variables on bond line microstructure for electropolished TD-NiCrAl specimens. Postheated at 1315° C for 2 hours in hydrogen.

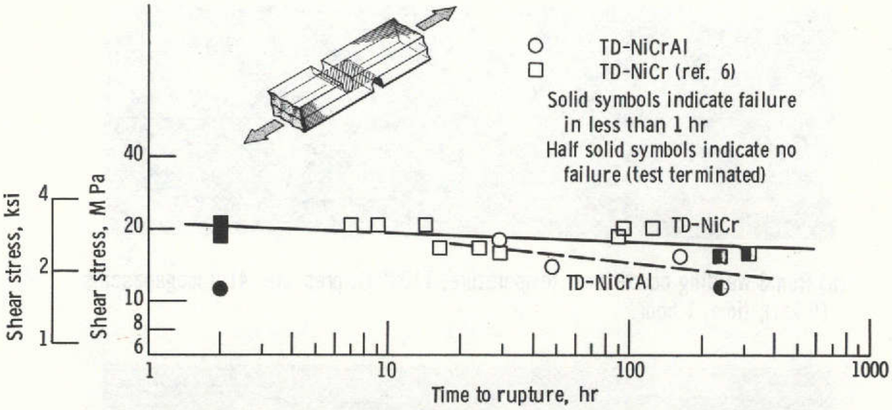


Figure 10. - Comparative weld shear stress at fracture versus time to rupture at 1100° C in air for notched hot-press weldments in 0.4-millimeter-thick sheet. Weldments postheated at 1315° C for 2 hours in hydrogen for TD-NiCrAl and at 1180° C for 2 hours in hydrogen for TD-NiCr.

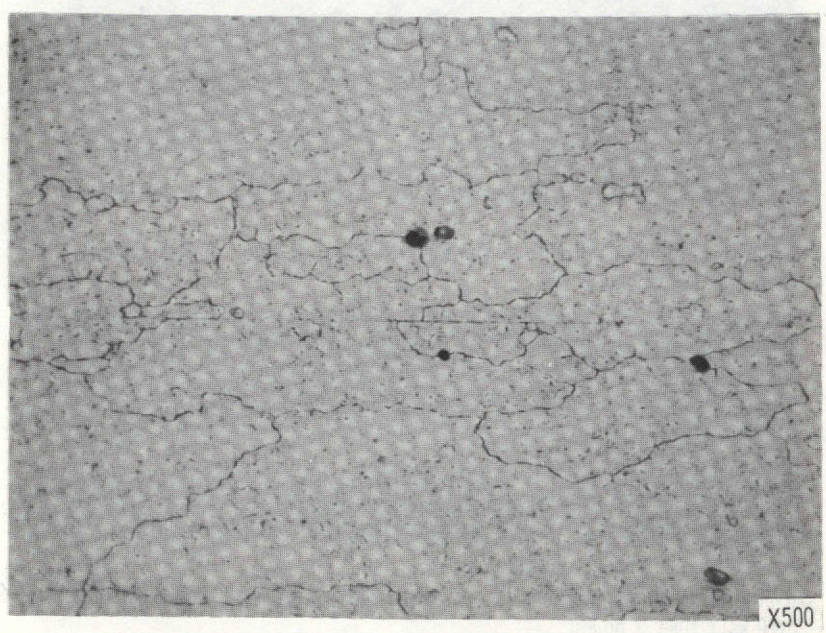


Figure 11. - Typical microstructure near center of solid-state spot weld made with one cycle of welding current in 0.4-millimeter-thick electropolished TD-NiCrAl sheet. Weldments postheated at 1315° C for 2 hours in hydrogen.

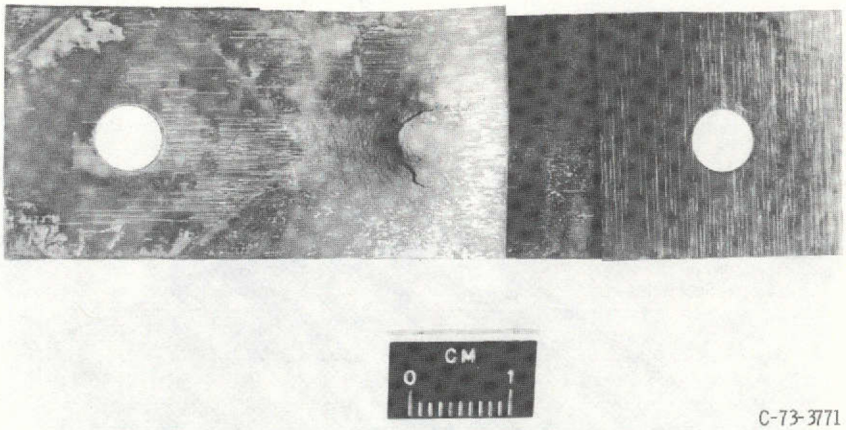


Figure 12. - Typical resistance spot weldment failure in stress-rupture test. Fracture occurred in TD-NiCrAl sheet adjacent to spot weld.

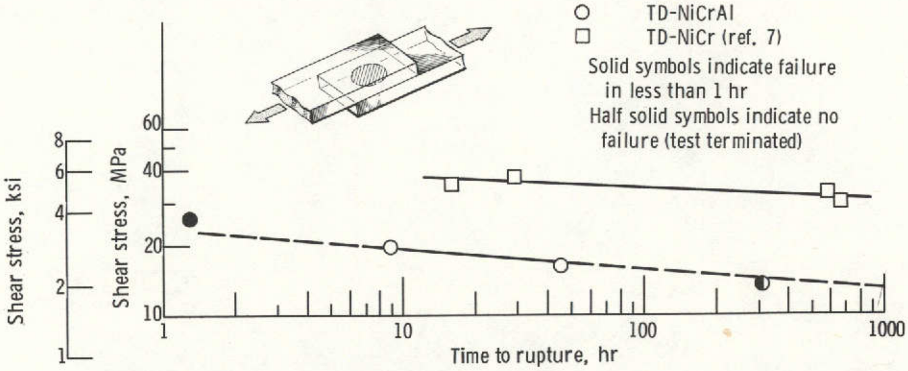


Figure 13. - Comparative weld shear at fracture versus time to rupture at 1100° C for resistance spot welds in TD-NiCrAl and TD-NiCr sheet. Single-spot weldments in 0.4-millimeter-thick sheet postheated at 1315° C for 2 hours in hydrogen for TD-NiCrAl and at 1200° C for 2 hours in hydrogen for TD-NiCr.

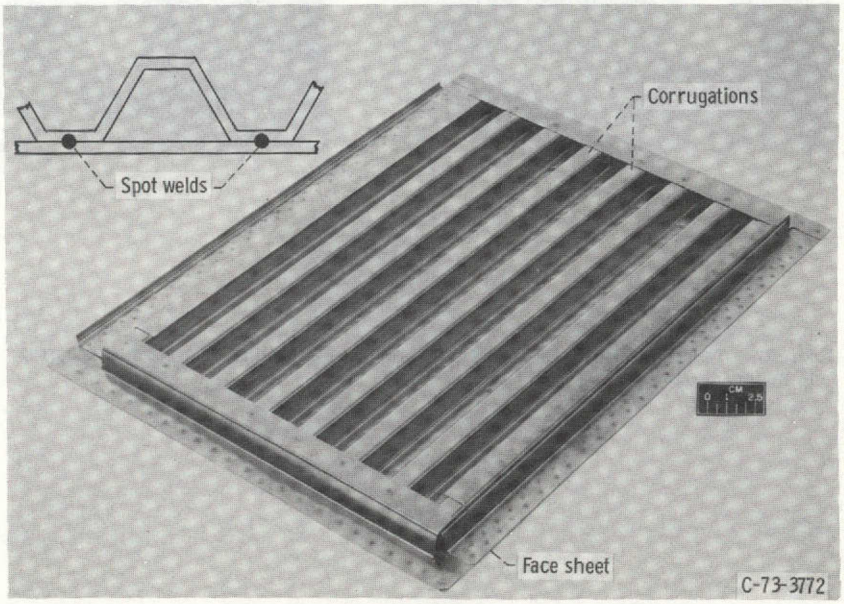
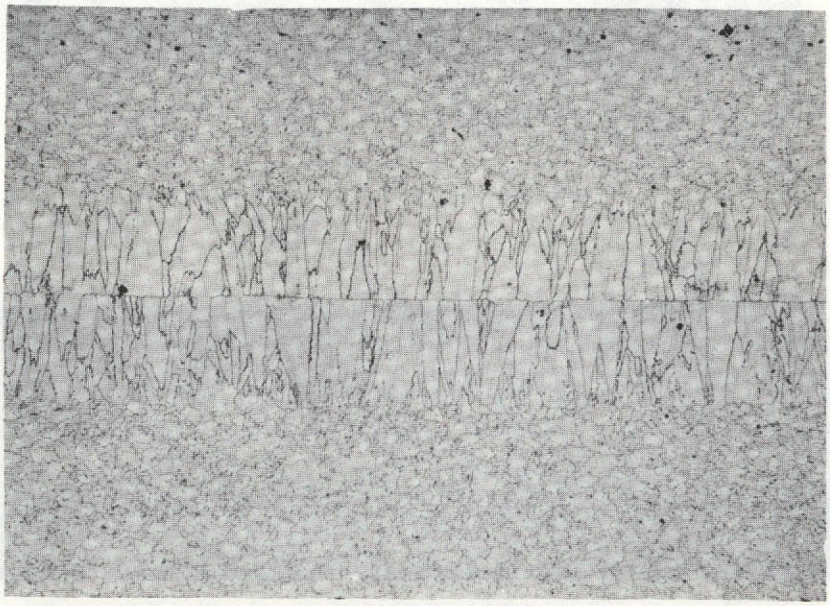
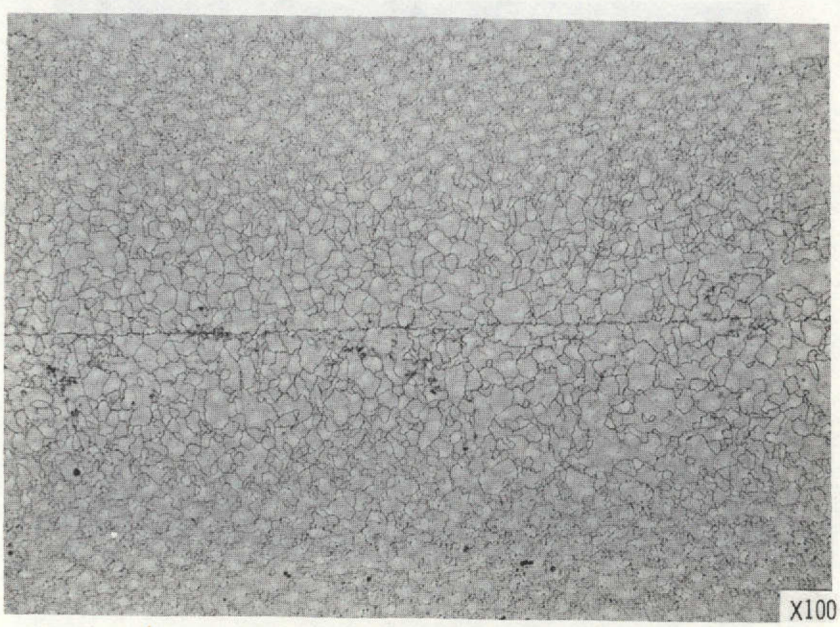


Figure 14. - Simulated heat shield panel in 0.4-millimeter-thick TD-NiCrAl sheet.



(a) Fusion spot weld.



(b) Solid-state spot weld.

Figure 15. - Typical fusion and solid-state resistance spot welds in simulated heat shield panel. Weldments postheated at 1315° C for 2 hours in hydrogen.

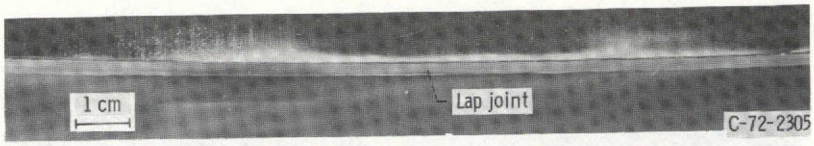
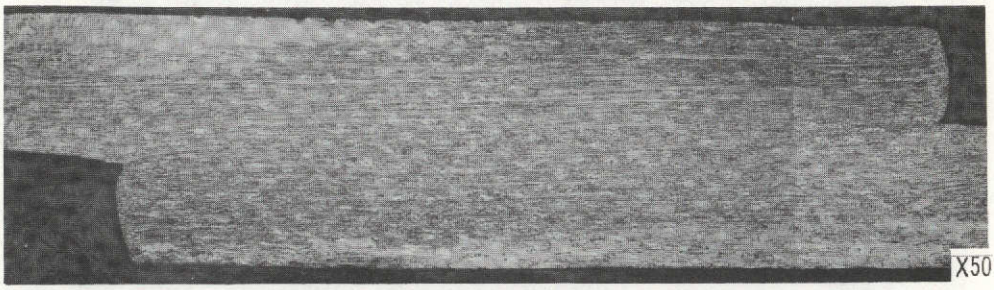
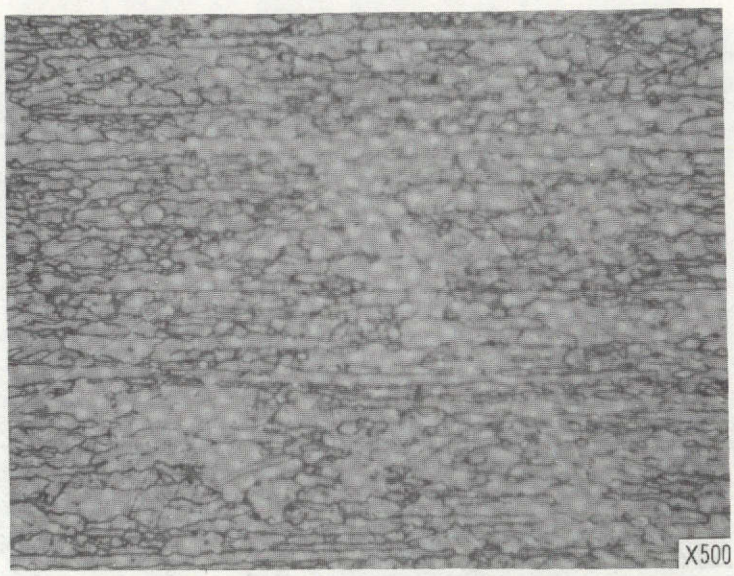


Figure 16. - Resistance seam weld run 4 in. 0.4-millimeter thick unrecrystallized TD-NiCrAl sheet. Joint overlap, 5t.



(a) Cross section of lap weld.



(b) Microstructure near bond line.

Figure 17. - Resistance seam weld (run 4) made in unrecrystallized 0.4-millimeter-thick TD-NiCrAl sheet. Weldment postheated at 1315° C for 2 hours in hydrogen.

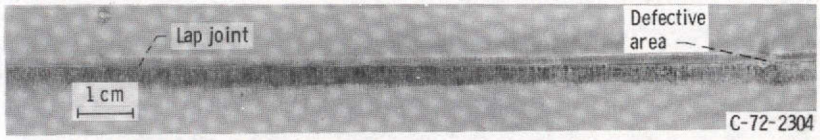
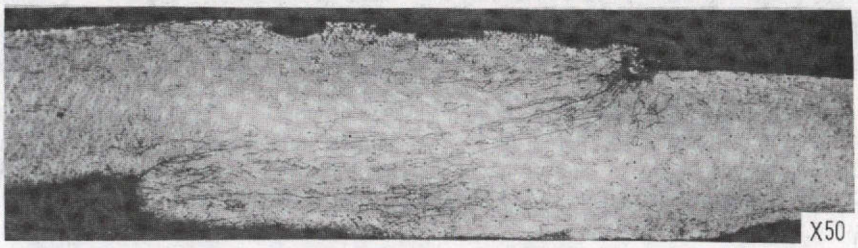
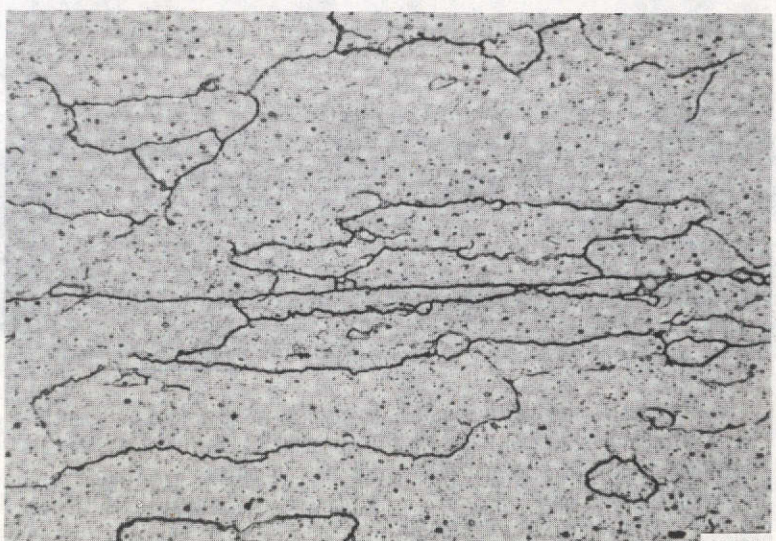


Figure 18. - Resistance seam welded panel in 0.4-millimeter-thick recrystallized TD-NiCrAl sheet. Joint overlap, 3t.



(a) Lap joint, showing excessive deformation.



(b) Microstructure near bond line.

Figure 19. - Resistance seam weld (run 5) made in recrystallized 0.4-millimeter-thick TD-NiCrAl sheet. Weldments postheated at 1315° C for 2 hours in hydrogen.

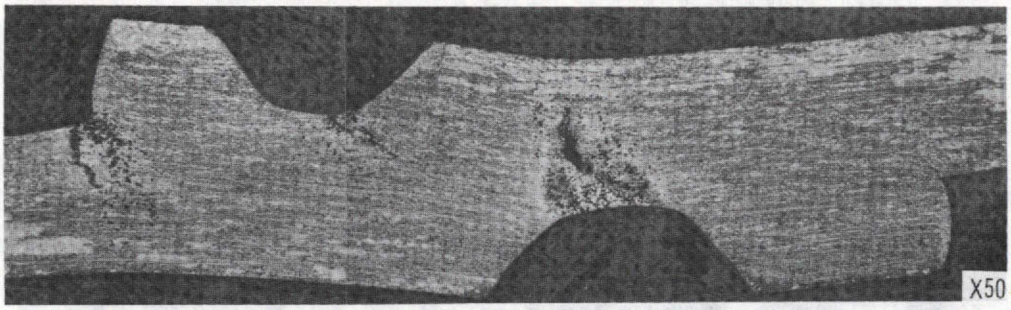


Figure 20. - Failure initiation in notched seam welded lap joint in unrecrystallized 0.4-millimeter-thick TD-NiCrAl sheet. Stress-rupture test stopped before complete fracture. Weldments postheated at 1315° C for 2 hours in hydrogen. Stress-rupture test conditions: temperature, 1100° C; stress (weld shear), 9.7 megapascals (1.1 ksi); time of test, 22 hours.

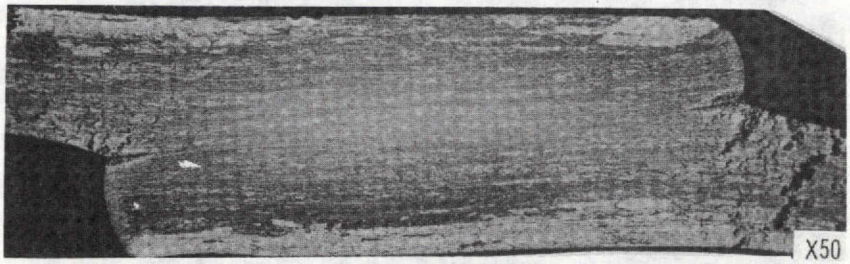


Figure 21. - Base-metal stress-rupture failure adjacent to seam welded lap joint in unrecrystallized 0.4-millimeter-thick TD-NiCrAl sheet. Weldments postheated at 1315° C for 2 hours in hydrogen. Stress-rupture test conditions: temperature, 1100° C; stress (weld shear), 8.3 megapascals (1.2 ksi); time to rupture, 0.9 hour.

NASA-Langley, 1975