AN IMPROVED DIFFUSION WELDING TECHNIQUE FOR TD-NiCr SHEET

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An improved diffusion welding technique has been developed for TD-NiCr sheet. In the most preferred form, the improved technique consists of diffusion welding 320-grit sanded plus chemically polished surfaces of unrecrystallized TD-NiCr at 760°C under 140 MN/m² pressure for 1 hr followed by postheating at 1180°C for 2 hr. Compared to previous work, this improved technique has the advantages of shorter welding time, lower welding temperature, lower welding pressure, and a simpler and more reproducible surface preparation procedure. Weldments were made that had parent-metal creep-rupture shear strength at 1100°C.
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AN IMPROVED DIFFUSION WELDING TECHNIQUE FOR TD-NiCr SHEET

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SUMMARY

An improved diffusion welding technique has been developed to join TD-NiCr sheet. Compared to previous work, this improved technique has the advantages of shorter welding time, lower welding temperature, lower welding pressure (in the most preferred form), and a simpler and more reproducible surface preparation procedure. Weldments were made that had parent-metal creep-rupture shear strength at 1100° C (2012° F).

Specially processed (unrecrystallized) and commercial TD-NiCr were welded successfully by using the improved welding technique. However, the specially processed material was preferred over the commercial material since the weld line could be eliminated, creep-rupture fracture of weldments took place in the parent material away from the weld line, and the creep-rupture shear strength of weldments was higher than that of welds made in commercial TD-NiCr. With the commercial TD-NiCr, a semicontinuous weld line resulted, and creep-rupture shear fracture took place at this weld line, which indicated a plane of weakness.

Specially processed material was also welded to commercial material with the improved technique. These welds looked very promising and performed similarly to the welds in specially processed material.

As a result of the present study a simpler and more reproducible surface preparation technique is recommended which consist of sanding the faying surface with 320-grit paper and then chemically polishing it. A one-step weld cycle at a temperature of 760° C (1400° F) for 1 hour is recommended for the following material combinations at the following pressures:

1. Specially processed material - 140 meganewtons per square meter (20 ksi)
2. Commercial material - 275 meganewtons per square meter (40 ksi)
3. Specially processed to commercial material - 210 meganewtons per square meter (30 ksi)

Postheating at 1180° C (2150° F) for 2 hours in hydrogen is also recommended to recrystallize the specially processed material weldments and cause grain growth and elimination of the weld line. Postheating is thought to strengthen the commercial material weldments by increased diffusion across the weld line.
A dispersion-strengthened nickel-base alloy, commercially designated TD-NiCr, 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 - 20-weight-percent-Cr matrix, which contains a fine dispersion (2 weight percent) of ThO\(_2\) particles, as described in reference 1. TD-NiCr sheet is being considered for applications where metal temperatures may reach about 1200\(^\circ\) C (2200\(^\circ\) F) in an oxidizing environment. Examples of potential applications include jet-engine components and the heat-shield panels of space shuttle vehicles (refs. 1 and 2).

Joining dispersion-strengthened materials such as TD-NiCr by conventional fusion welding processes results in joint efficiencies (joint strength x 100/parent-metal strength) of only about 40 to 50 percent at elevated temperatures (ref. 3). Fusion welding TD-NiCr destroys the ThO\(_2\) dispersion and the benefits of prior mechanical working. The resulting strength of the fusion weldment is similar to that of thoria-free Ni - 20 weight percent Cr. For this reason, solid-state welding (particularly diffusion welding where the deformation at the weld is low) is a promising approach to joining TD-NiCr since melting is avoided.

A recent study (ref. 4) has demonstrated the feasibility of diffusion welding TD-NiCr sheet. The diffusion lap welds had parent-metal strength as determined by creep-rupture shear testing at 1090\(^\circ\) C (2000\(^\circ\) F). Previous problems encountered in diffusion welding TD-NiCr, such as formation of a band of small, weak recrystallized grains at the weld interface, a continuous weld line, and unwelded areas, were avoided by carefully selecting surface preparation, metallurgical condition of the starting material, and welding cycle. Specifically, the following conditions were recommended in reference 4 for diffusion welding TD-NiCr sheet:

1. Surface preparation - 600-grit sanding plus electropolishing
2. Material - specially processed (unrecrystallized) TD-NiCr preferred over commercial TD-NiCr
3. Two-step weld cycle
   a. Heating at 705\(^\circ\) C (1300\(^\circ\) F) and 210 meganewtons per square meter (30 ksi) for 1 hour
   b. Heating at 1190\(^\circ\) C (2175\(^\circ\) F) and 15 meganewtons per square meter (2 ksi) for 2 hours

Although the study of reference 4 provided diffusion welds with parent-metal strength, there are several limitations that make it difficult to apply the results. First, the surface preparation used (sanding through 600-grit paper plus electropolishing) is time consuming and not always easy to reproduce. For example, it was difficult to produce a uniform, pit-free surface on every specimen by electropolishing since factors
such as age of the electropolishing solution, temperature, voltage, current, and time all affected the condition of the final surface. Second, the welding cycle used was long (3 hr plus heatup time) and required high-temperature (1190° C; 2175° F) welding tooling.

The purpose of the study described in this report was to develop an improved diffusion welding technique for TD-NiCr sheet. Emphasis was placed on development of a simpler and more reproducible surface preparation technique and on reducing the time and temperature necessary for diffusion welding. Both specially processed (unrecrystallized) and commercial TD-NiCr sheet 0.4 millimeter (0.015 in.) thick were included in this study. Evaluation of the improved diffusion welding technique was done by creep-rupture shear testing of diffusion-welded specimens at 1100° C (2010° F) and by metallographic analysis.

MATERIALS AND PROCEDURE

TD-NiCr Sheet

Specially processed material. - Specially processed (SP) TD-NiCr 0.4 millimeter (0.015 in.) thick was used in the tests. The nominal composition is Ni - 20 weight percent Cr - 2 weight percent ThO₂. The special processing consisted of leaving out the final recrystallization heat treatment that is normally given to commercial TD-NiCr after thermomechanical processing, so that the SP TD-NiCr is actually in the fine-grained, unrecrystallized condition. TD-NiCr in the SP condition has a grain size that is too fine to see by light microscopy (refs. 1 and 4). However, this material is subsequently recrystallized to a much larger grain size by postheating after diffusion welding, as will be shown in this report.

This material was selected because a previous study (ref. 4) had shown that the weld line could be eliminated for diffusion welds in 1.6-millimeter- (0.060-in.-) thick SP material. The 0.4-millimeter- (0.015-in.-) thick SP TD-NiCr was expected to behave identically to the 1.6-millimeter- (0.060-in.-) thick SP material during during diffusion welding since the two materials have essentially the same metallurgical characteristics.

In a recent study (unpublished data from General Dynamics/Convair) the ductility (tensile elongation in 5 cm, 2 in.) was determined for 0.25- and 0.5-millimeter- (0.010- and 0.020-in.-) thick SP TD-NiCr at various elevated temperatures, as shown in figure 1. It is expected that the 0.4-millimeter- (0.015-in.-) thick SP TD-NiCr used in this study has similar ductility. The ductility of the SP material is poor at room temperature but increases with temperature. The ductility is best in the 760° to 815° C
(1400° to 1500° F) temperature range and varies from 11 to 27 percent elongation depending on the testing direction (fig. 1). Recrystallization begins at 815° to 870° C (1500° to 1600° F) (ref. 5).

Commercial material. - Commercial TD-NiCr 0.4 millimeter (0.015 in.) thick was also used in the tests. 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 in hydrogen. The commercial material is more ductile at room temperature than the SP material (15 percent as opposed to 2 percent tensile elongation in 2.54 cm, 1 in.) and is thus more formable. The commercial-material grain size is much larger than that of the SP material in order to provide good high-temperature strength (ref. 4). The nominal composition of the commercial material is Ni - 20 weight percent Cr - 2 weight percent ThO₂.

Combination of specially processed and commercial material. - Since the commercial material is formable at room temperature and the SP material is not, it would be desirable to be able to weld this dissimilar combination of materials for fabrication of some aerospace components. For example, in a two-component assembly (such as a corrugation-stiffened heat shield) the component most severely formed (the corrugation) could be made from commercial TD-NiCr, and the component least form (the face sheet) could be made from SP TD-NiCr. Therefore, a few specimens of the combined materials were included in this study.

Welding Procedure

Welding equipment. - A vacuum hot press was used to make diffusion lap welds in both commercial and SP TD-NiCr. The weld specimens were radiantly heated by a tantalum resistance heater. A 220-kilonewton (25-ton) hydraulic press was used to apply welding force. A pressure of 2×10⁻⁵ torr was maintained in the vacuum chamber during welding.

Sintered tungsten rams were used to transmit force from the hydraulic press to the weld tooling. Weld tooling was made from 3.2-millimeter- (0.125-in.-) thick commercial TD-NiCr and also from Inconel X. Although both materials were used successfully to make diffusion welds, the TD-NiCr underwent less warpage with extended use and was used for the majority of the study.

The weld tooling consisted of three protrusions to make three diffusion lap welds in each specimen simultaneously, as shown in figure 2. Three welds were made in order to increase the weld area so that enough welding force was required to allow use of existing hydraulic equipment above its minimum setting. The welds made were smaller than the overlap so that there was material available at the periphery of each weld for
expansion as deformation took place at the weld. This tooling was felt to be more representative of welding an actual lap configuration in hardware than tooling that would completely cover the overlap and not allow expansion.

**Specimen preparation.** - The as-received TD-NiCr sheet (both types) had nominal 120-grit belt-sanded surfaces with the surface scratches parallel to the principal rolling direction. Of course, as the finishing belt becomes smoother with use, the surface finish on the TD-NiCr sheet also becomes smoother. Thus, the as-received sheet can vary somewhat in surface finish.

The as-received surface finish was evaluated both with and without chemical polishing. Without chemical polishing, the specimens were detergent cleaned, rinsed in methyl alcohol, and stored in trichlorotrifluoroethane before welding. With chemical polishing, the specimens were prepared by being placed consecutively in two heated acid solutions, as shown in figure 3. Uniformly polished surfaces were easily obtained with this technique, and no problems were encountered in reproducing the polished surfaces on consecutive batches of specimens. Less than 0.025 millimeter (0.001 in.) of material thickness was removed during chemical polishing.

Other surface finishes evaluated included 320-grit sanding and 600-grit sanding. Sanding was done only on the mating surfaces. These surface finishes were evaluated only after they were chemically polished in the same manner as described for the as-received surfaces (as shown in fig. 3).

The surfaces of the weld specimens in contact with the weld tooling were coated with alumina (Al₂O₃) to prevent sticking.

**Weld cycles.** - The specimens were overlapped approximately 13 millimeters (0.5 in.), and a vacuum of 2×10⁻⁵ torr was attained in the welding chamber. The specimens were heated to the welding temperature, welding force was applied, and diffusion welding was achieved.

The welding cycles used in this study are summarized in table I. The majority of the welds were made with the recommended one-step weld cycles shown in the top section of the table. For the SP material heating at 760°C (1400°F) and 140 meganewtons per square meter (20 ksi) for 1 hour is recommended. But for the commercial material, a higher pressure (275 MN/m², 40 ksi) is recommended. And for welding SP to commercial material, an intermediate pressure (210 MN/m², 30 ksi) is recommended. No measurable deformation was recorded after any of these recommended cycles was used. All welds made in this study were postheated at 1180°C (2150°F) for 2 hours in hydrogen. Comparison of the recommend weld cycles with the previously used two-step cycle shows that the recommended weld cycles require a lower temperature and shorter time (table I).

The SP material was welded at 760°C (1400°F), where ductility is high enough to obtain intimate contact (fig. 1), but the recrystallization temperature (approx. 815°C to
870° C, 1500° to 1600° F (ref. 5)) is not reached. It was the intent of this study to recrystallize the SP material during postheating and eliminate the weld line by grain growth, without welding pressure, in a conventional furnace. The advantage is that very high temperature (approx. 1180° C, 2150° F) hot-press tooling would not be required as in the previous study (ref. 4). The commercial material was also diffusion welded at a lower temperature (760° C, 1400° F) than previously used (table I). At 760° C (1400° F) commercial TD-NiCr has sufficient ductility (approx. 10 percent) to obtain intimate contact during diffusion welding. Postheating was used to increase diffusion across the weld line and increase weld strength. The combination of SP and commercial material was welded at 760° C (1400° F) for the reasons already cited. Postheating was used to recrystallize the SP material and eliminate the weld line by grain growth across the interface.

During the development of the recommended weld cycles, other cycles were tried, some of which are shown in the bottom section of table I. As can be seen from this table, the principal variable explored was diffusion welding pressure. Pressure was varied from the point where the specimens fell apart on removal from the tooling after the weld cycle to the point where excessive deformation took place at the weld. (The effect of excessive deformation on weld microstructure will be illustrated in the section Effect of weld cycle.) Temperature was held at approximately 760° C (1400° F).

Weld Evaluation

The weldments were evaluated both metallographically and by creep-rupture shear testing. The creep-rupture shear specimens were punched from the weldments to achieve the configuration shown in figure 2. Part of the diffusion weld was left intact on either side of the punched gage section and was used for metallographic evaluation.

As shown in figure 2, the middle weld was creep-rupture shear tested by notching to a nominal 2t overlap (where t is the thickness of the parent material). Although this was not a pure shear test because of associated bending stresses, it is representative of actual lap welds in service which usually are stressed similarly. The specimen gage section was 4 millimeters (0.16 in.) wide. Testing was done in air at 1100° C (2012° F) with deadweight loading (approx. 35 N, 8 lb). Failure occurred either in shear (at the weld or in the parent material) between the notches, in tension at the base of the notches, or by a combination of the two.

Creep-rupture shear tests were used to evaluate the quality of these diffusion welds mechanically because previous studies (refs. 4 and 6) have shown that the creep-rupture test is a more severe mechanical test of diffusion weld quality in TD-NiCr and TD-Ni than elevated- or room-temperature tensile tests. For this reason, creep-
rupture shear tests were used to determine if improvements in the diffusion weld cycle and surface preparation altered diffusion weld quality. Only the welds with no excessive deformation or unwelded areas were used for these shear tests. It was not considered feasible to develop a parent-metal creep-rupture shear curve for this thin sheet because of anticipated difficulties in machining notches in and testing the 0.4-millimeter-(0.015-in.-) thick material. Therefore, 1.6-millimeter-(0.060-in.-) thick TD-NiCr creep-rupture shear data from reference 4 were used for a parent-metal strength comparison.

The metallographic sections were polished and etched electrolytically with a solution of 100 cubic centimeters of water (H₂O), 2 grams of chromium oxide (CrO₃), and 10 cubic centimeters of sulfuric acid (H₂SO₄). Only the middle weld (fig. 2) is shown for each specimen described in this report. The unwelded areas that naturally occurred between the middle and edge welds (fig. 2) were used to locate the weld line for each photomicrograph shown in this report. So, at the edge of most of the photomicrographs unwelded areas are apparent and are not indicative of weld quality. Also, failed creep-rupture shear specimens were metallographically evaluated for location and mode of failure.

RESULTS AND DISCUSSION

High-quality, diffusion lap welds in TD-NiCr were produced with the improved welding technique. When the metallurgical condition of the starting material, the weld specimen surface preparation, and the weld cycle were carefully selected, diffusion welds as strong as the parent material were produced. The preferred metallurgical condition was the SP (unrecrystallized) form of TD-NiCr. The recommended surface preparation was 320-grit sanding plus chemical polishing, as shown in figure 3. And the recommended weld cycles are the one-step cycles shown in the top section of table I followed by postheating. Specific results are discussed in the following sections.

Creep-Rupture Shear Tests

The results of the creep-rupture shear tests are summarized in table II and figure 4. As can be seen from the failure locations and modes of fracture shown in table II, it was difficult to force shear failure in the overlap region even with the 2t overlap used. One of the two specimens that failed in shear in the parent material (line 9 in table II) lasted 95 hours at a shear stress of about 20 meganewtons per square meter (3 ksi). This compares very closely with the 100-hour parent-metal creep-rupture...
The shear stress of 1.6-millimeter- (0.060-in.-) thick TD-NiCr, which is 21 meganewtons per square meter (3.1 ksi), as shown in figure 4. Thus, 20 meganewtons per square meter (3 ksi) is probably close to the 100-hour creep-rupture shear stress of the 0.4-millimeter- (0.015-in.-) thick TD-NiCr. This further illustrates the similarity of properties in 1.6 and 0.4-millimeter- (0.060- and 0.015-in.-) thick TD-NiCr.

The diffusion welds in SP TD-NiCr had excellent creep-rupture shear strength, as shown in table II and figure 4. At no time did a SP diffusion weld fail at the location of the original weld line. The failures were always in the parent material. This was true for both the 320-grit and 600-grit plus chemically polished surfaces prior to welding, as shown in table II. Most of the parent-metal failures were tensile-type failures caused by tensile and bending stresses adjacent to the overlap. An example of this type of failure is shown in figure 5(a). It can be seen in figure 5(a) that the weld was highly stressed, as shown by the porosity formation and grain boundary separation that occurred in the parent-metal grain boundaries in the overlap region. Had the weld been weaker than the parent metal, failure would have occurred through the original weld interface. The welds in the SP material are therefore as strong as the parent material, and a 320-grit plus chemically polished surface preparation is adequate.

Diffusion welds in the commercial material were weaker than welds in the SP material when tested in creep-rupture shear, as shown in table II and figure 4. Shear failure through the original weld interface commonly occurred either during testing (as described in ref. 4) or on cooling to room temperature after a discontinued test. This was true for both the 320-grit and 600-grit plus chemically polished surfaces (as shown in table II). For welds in commercial TD-NiCr, creep-rupture shear strength was consistently lower than for welds in SP TD-NiCr. Shear failure at the weld line (which occurred while cooling a discontinued test to room temperature) is shown in figure 5(b). This type of failure is thought to be due to the formation of an oxide (principally chromium oxide, \( \text{Cr}_2\text{O}_3 \)) at grain boundaries during creep-rupture testing (ref. 7). Although this happens at grain boundaries under stress throughout the entire specimen (as at other grain boundaries in figure 5(b)), in welds in commercial material, the oxide forms continuously at the weld line which is in effect a continuous grain boundary. On cooling, differential thermal contraction between the oxide and metal presumably caused failure at the oxide-metal interface.

Only a limited amount of creep-rupture shear testing was done on the welds between SP and commercial TD-NiCr, as shown in table II and figure 4. However, the creep-rupture strengths were excellent, and no failures occurred at the location of the original weld line. As shown in figure 4, creep-rupture shear strengths of diffusion welds between SP and commercial TD-NiCr were higher than for welds in commercial material. The welds between SP and commercial material failed in tension in the parent material.
either during a test or on cooling to room temperature after discontinuation of the test (a result of the oxide problem previously described). The strength and failure mode of these welds were very similar to the results obtained for welds in SP material to itself.

Microstructural Evaluation

Diffusion welds prepared from SP, commercial, and the combination of SP and commercial material were examined by light microscopy. Various weld cycles, including the recommended cycles, were used. And the as-received (120-grit), 320-grit, and 600-grit sanded plus chemically polished surface preparations were evaluated.

Effect of surface preparation. - The recommended surface preparation consisted of sanding the as-received surface with 320-grit paper on the side to be welded and then chemically polishing it, as shown in figure 3. Typical microstructures of welds in SP and commercial TD-NiCr made with this surface preparation and the recommended weld cycles (table I) are shown in figure 6. The 600-grit plus chemically polished surface preparation (not shown) produced the same results. Specimens with this preparation had strengths similar to those of the 320-grit sanded specimens, as shown in table II. Since the 600-grit sanding offered no advantage over the 320-grit sanding and involved an additional sanding step, the 320-grit sanding plus chemical polishing is recommended.

As shown in figure 6(a) the weld line in SP material has been eliminated by using the recommended surface preparation and weld cycle. During postheating, recrystallization and grain growth occurred across the weld line and thereby eliminated it. Elimination of the weld line is further proven by the fact that creep-rupture shear testing always resulted in parent-metal failure, away from the weld line. These same results were previously obtained for 1.6-millimeter- (0.060-in.-) thick SP TD-NiCr (ref. 4) with the more complicated surface preparation and weld cycle shown in the middle section of table I.

Figure 6(b) shows a lap weld in commercial TD-NiCr made with the recommended surface preparation and weld cycle. A fairly continuous weld line is evident with some areas of possible grain growth across the weld line. The semicontinuous weld line is similar to a grain boundary, and failure occurred at the weld line in 1100° C (2012° F) creep-rupture shear tests (table II). This is expected since grain boundaries are weaker than the matrix at elevated temperatures. Similar results were previously obtained for 1.6-millimeter- (0.060-in.-) thick commercial TD-NiCr (ref. 4), again with a more complicated surface preparation and weld cycle.

A limited amount of work was done to determine the feasibility of welding SP to commercial material. A cross section of a typical weld made with the recommended
cycle in table I between these two materials, after postheating, is shown in figure 7. It is evident that the weld line has been eliminated. Further evidence of the good weld quality is given by the parent-metal creep-rupture failures that occurred when this type of specimen was tested as described in the preceding section. It is thought that the weld line was eliminated when recrystallization and grain growth of the SP material occurred during postheating. The commercial material grains are stable at the postheating temperature and probably nucleated the SP grains. Both materials were prepared by sanding with 600-grit paper and chemical polishing, as shown in figure 3. Unfortunately, one specimen was accidentally welded upside down for each of the three welds listed in the bottom section of table II. Consequently, an as-received plus chemically polished surface was welded to a 600-grit sanded plus chemically polished surface (both combinations, see bottom section of table II). This did not harm weld quality as all three welds exhibited high creep-rupture shear strength (table II) and elimination of the weld line (fig. 7). The fact that the specimen with the as-received plus chemically polished surface had improved flatness from 600-grit sanding the opposite slide probably helped to insure high weld quality. However, 320-grit plus chemical polishing surface preparation is still recommended for this combination of materials to insure consistently high quality diffusion welds.

The as-received (120-grit sanded) surface preparation was investigated both with and without chemical polishing for welds in SP and commercial TD-NiCr to themselves as this preparation offered the advantage of not requiring additional sanding. Without chemical polishing, the same problem of small, recrystallized grain formation at the weld line resulted for SP material as previously encountered (ref. 4). When a weld made with the as-received surface preparation was creep-rupture shear tested at a low shear stress (17 MN/m², 2.4 ksi), failure on loading occurred in shear at the weld line. With chemical polishing, the as-received surface preparation usually resulted in the formation of a semicontinuous weld line in the SP material, as shown in figure 8, and the formation of small grains after recrystallization (not shown) regardless of the welding cycle used. This was observed in other welds with the same surface preparation also. Perhaps the semicontinuous weld line forms with this surface preparation because the surface roughness and sheet waviness present even after chemical polishing, result in excessive deformation at high points and inadequate deformation at the low points. Inadequate deformation (and pressure) was seen to result in a continuous weld line and unwelded areas (described in the next section). And excessive deformation (and pressure) was seen to result in the formation of small grains after recrystallization (described in the next section). Therefore, even though the weld shown in figure 8 has some grain growth across the weld line, other problems (such as unwelded areas and small grains after recrystallization) can occur with the as-received plus chemically polished surface preparation.
The as-received plus chemically polished surface preparation also was not suitable for diffusion welding commercial TD-NiCr sheet, as shown in figure 9. With the recommended weld cycle, large unwelded areas occurred (see fig. 9(a)). The unwelded areas are due to the waviness and thickness variations of the TD-NiCr sheet and the difficulty in forcing these uneven surfaces together during diffusion welding. Simply increasing the welding pressure (fig. 9(b)) did not solve the problem, as excessive deformation then occurred in the TD-NiCr sheet and unwelded areas still resulted. The excessive deformation will be discussed in the next section.

By 320-grit sanding before chemical polishing, the sheet waviness, thickness variation, and surface roughness are reduced to the point where reproducible diffusion welds can be made in SP and commercial TD-NiCr without excessive deformation or large unwelded areas.

Effect of weld cycle. - For SP TD-NiCr, the recommended welding cycle is heating at 760° C (1400° F) and 140 meganewtons per square meter (20 ksi) for 1 hour followed by postheating at 1180° C (2150° F) for 2 hours in hydrogen. Diffusion welding at temperatures much below 760° C (1400° F) resulted in welds too weak to handle after removal from the hot press. For example, welds made at 705° C (1300° F) fell apart during removal from the hot press. Welding temperatures around 815° to 870° C (1500° to 1600° F) could result in the beginning of premature recrystallization (ref. 5). Recrystallization at these temperatures would probably result in a grain size smaller than desirable for good high-temperature strength. Too little welding pressure (approx. 70 MN/m², 10 ksi) resulted in unwelded areas and a tendency to form a continuous weld line for diffusion welds in SP TD-NiCr. Too much welding pressure (approx. 210 MN/m², 30 ksi) often resulted in excessive deformation (approx. 1 percent) and the formation of small grains after recrystallization. The formation of small grains due to excessive deformation is shown in figure 10. Evidently, excessive deformation somehow changed the texture of SP TD-NiCr so that it could not recrystallize to a large grain size. The small grains are very weak at elevated temperatures (see fig. 10(c)), as evidenced by creep-rupture shear specimens that failed at the small grains before reaching the test temperature.

For commercial TD-NiCr, the recommended welding cycle (shown in table I) is heating at 760° C (1400° F) and 275 meganewtons per square meter (40 ksi) 1 hour followed by postheating at 1180° C (2150° F) for 2 hours. Diffusion welding at temperatures much below 760° C (1400° F) resulted in welds that were too weak to handle. Temperatures above 760° C (1400° F) could probably be used, although the ductility of commercial TD-NiCr begins to drop significantly above 870° C (1600° F) (ref. 8). But, intimate contact would be more difficult to obtain during welding at the higher temperatures, and unwelded areas would be more difficult to avoid (ref. 4). Too little pressure (approx. 210 MN/m², 30 ksi) resulted in unwelded areas. Conversely, excessive pres-
sure (approx. 310 MN/m², 45 ksi) resulted in deformation twinning and grain boundary cracking, as shown in figure 9(b). These metallurgical changes are expected to decrease the strength of TD-NiCr. Of course, the chance of excessive deformation is increased by sheet waviness and variations in sheet thickness (for the same nominal welding pressure) as these factors caused localized pressure increases. With the proper surface preparation, higher welding pressures can be tolerated since localized pressure increases tend to be avoided.

For welds between SP and commercial TD-NiCr, the recommended welding cycle is heating at 760° C (1400° F) and 210 meganewtons per square meter (30 ksi) for 1 hour followed by postheating at 1180° C (2150° F) for 2 hours. Diffusion welding at lower pressure (e.g., 140 MN/m², 20 ksi, bottom section of table I) resulted in welds that fell apart on removal from the hot press. Welding at pressures greater than 210 meganewtons per square meter (30 ksi) would probably result in excessive deformation in the SP material, as previously described.

It should be emphasized that postheating at 1180° C (2150° F) for 2 hours is essential in developing optimum diffusion welds in SP and commercial TD-NiCr. Postheating recrystallizes the SP material and eliminates the weld line by grain growth. For commercial TD-NiCr postheating causes increased diffusion across the weld line and is thought to strengthen the diffusion weld greatly. Although hydrogen was used exclusively for postheating in this study and is used commercially for recrystallizing SP TD-NiCr, it is probable that any inert atmosphere, such as argon, would be suitable.

**Applicability of the Process**

The improved diffusion welding technique described in this report was tested for only one thickness of TD-NiCr sheet. But the welding technique should be applicable to all gages of TD-NiCr that are capable of undergoing recrystallization and grain growth with heat treatment. After other gages of TD-NiCr are diffusion welded in the unrecrystallized condition, recrystallization and grain growth during postheating should eliminate the weld line and provide welds with parent-metal strength. Also, it is quite probable that other dispersion-strengthened materials, such as TD-NiCrAl, may also be joined by this improved diffusion welding technique.

The successful joining of specially processed (unrecrystallized) TD-NiCr to commercial TD-NiCr is perhaps the most important single result of this study. The potential of being able to diffusion weld a material that is formable at room temperature (commercial TD-NiCr) to a material capable of eliminating the weld line (specially processed TD-NiCr) opens a much wider range of applicability for this welding process. For example, a corrugation-stiffened heat shield panel could use SP TD-NiCr for the
flat, face sheet and commercial TD-NiCr for the corrugations. Also, diffusion welding commercial TD-NiCr to itself with the use of a specially processed TD-NiCr interlayer could be considered. This should eliminate both weld lines and produce parent-metal strength in the joints. Another possible application is the joining of directionally recrystallized dispersion-strengthened materials (ref. 9) for jet engine components. This offers the possibility of diffusion welding two turbine blade halves together at temperatures below their recrystallization temperature and then eliminating the weld line while directionally recrystallizing the assembly. This application conceivably could greatly reduce the manufacturing costs for producing hollow turbine blades.

CONCLUSIONS

An improved diffusion welding technique has been developed for joining TD-NiCr sheet. Diffusion-welded lap joints made with 0.4-millimeter- (0.015-in.-) thick TD-NiCr had 1100° C (2012° F) shear strengths equal to that of the parent material. Compared to the previous two-step welding process, this improved one-step welding method has the advantages of shorter welding time, lower welding pressure (in the most preferred form), and a simpler and more reproducible surface preparation procedure.

Specifically, the following conclusions resulted from this study:

1. The diffusion welding process is applicable to joining both commercial-grade and specially processed (unrecrystallized) TD-NiCr sheet to themselves or to each other. Use of the specially processed material is preferred because of better reliability of joint quality.

2. The conditions recommended for the one-step weld cycles developed in this study are heating at 760° C (1400° F) for 1 hour for the following material combinations at the following pressures:
   a. Specially processed TD-NiCr - 140 meganewtons per square meter (20 ksi)
   b. Commercial TD-NiCr - 275 meganewtons per square meter (40 ksi)
   c. Specially processed to commercial TD-NiCr - 210 meganewtons per square meter (30 ksi)

Postheating at 1180° C (2150° F) for 2 hours in a nonoxidizing atmosphere is recommended for all of these cycles to produce recrystallization and/or grain growth across the weld line.
3. The recommended preweld joint preparation method involves surface sanding with 320-grit paper followed by chemical polishing.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, November 9, 1972,
502-21.

REFERENCES


TABLE I. - DIFFUSION WELD CYCLES USED TO JOIN 0.4-
MILLIMETER- (0.015-IN.-) THICK TD-NiCr SHEET

<table>
<thead>
<tr>
<th>Weld material</th>
<th>Temperature</th>
<th>Pressure</th>
<th>Time,</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>°C</td>
<td>°F</td>
<td>MN/m²</td>
</tr>
<tr>
<td><strong>Recommended weld cycles from this study</strong>^a</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Specially processed</td>
<td>760</td>
<td>1400</td>
<td>140</td>
</tr>
<tr>
<td>Commercial</td>
<td>760</td>
<td>1400</td>
<td>275</td>
</tr>
<tr>
<td>Specially processed to</td>
<td>760</td>
<td>1400</td>
<td>210</td>
</tr>
<tr>
<td>commercial</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Previous two-step weld cycle used (ref. 4)</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Specially processed</td>
<td>760</td>
<td>1400</td>
<td>210</td>
</tr>
<tr>
<td>and commercial</td>
<td>760</td>
<td>1400</td>
<td>240</td>
</tr>
<tr>
<td><strong>Other weld cycles tried in this study</strong>^a</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Specially processed</td>
<td>760</td>
<td>1400</td>
<td>140</td>
</tr>
<tr>
<td></td>
<td>170</td>
<td>30</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>210</td>
<td>35</td>
<td>1</td>
</tr>
<tr>
<td>Commercial</td>
<td>760</td>
<td>1400</td>
<td>210</td>
</tr>
<tr>
<td></td>
<td>240</td>
<td>35</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>275</td>
<td>40</td>
<td>1</td>
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<tr>
<td></td>
<td>310</td>
<td>45</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>345</td>
<td>50</td>
<td>1</td>
</tr>
<tr>
<td>Specially processed to</td>
<td>760</td>
<td>1400</td>
<td>140</td>
</tr>
<tr>
<td>commercial</td>
<td>760</td>
<td>1400</td>
<td>210</td>
</tr>
</tbody>
</table>

^aPostheating at 1180°C (2150°F) for 2 hr in hydrogen is also recommended.
^bStep 1.
^cStep 2.
TABLE II. - CREEP-RUPTURE SHEAR STRENGTHS OF DIFFUSION LAP WELDS IN 0.4-MILLIMETER- (0.015-IN.-) THICK TD-NiCr AT 1100° C (2012° F)

<table>
<thead>
<tr>
<th>Surface preparation</th>
<th>Shear stress MN/m²</th>
<th>Time, hr</th>
<th>Failure location</th>
<th>Mode of fracture</th>
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<tbody>
<tr>
<td>Specially processed material</td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>320-grit sanded</td>
<td>21.3</td>
<td>3.1</td>
<td>14.6</td>
<td>Parent metal</td>
</tr>
<tr>
<td>21.3</td>
<td>3.1</td>
<td>7.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>17.2</td>
<td>2.5</td>
<td>16.2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>21.2</td>
<td>3.08</td>
<td>9.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>16.5</td>
<td>2.4</td>
<td>29.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>17.2</td>
<td>2.5</td>
<td>24.4</td>
<td></td>
<td></td>
</tr>
<tr>
<td>15.8</td>
<td>2.3</td>
<td>236+</td>
<td></td>
<td></td>
</tr>
<tr>
<td>16.2</td>
<td>2.35</td>
<td>310+</td>
<td></td>
<td></td>
</tr>
<tr>
<td>20.5</td>
<td>2.98</td>
<td>95.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>600-grit sanded</td>
<td>19.1</td>
<td>2.77</td>
<td>0.1</td>
<td>Parent material</td>
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<tr>
<td>20.5</td>
<td>2.98</td>
<td>125.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>19.3</td>
<td>2.81</td>
<td>91.0</td>
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</tr>
<tr>
<td>22.0</td>
<td>3.2</td>
<td>25</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Commercial material</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>320-grit sanded</td>
<td>17.2</td>
<td>2.5</td>
<td>310+</td>
<td>Weld</td>
</tr>
<tr>
<td>17.2</td>
<td>2.5</td>
<td>145.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>17.2</td>
<td>2.5</td>
<td>316+</td>
<td></td>
<td></td>
</tr>
<tr>
<td>18.6</td>
<td>2.7</td>
<td>01</td>
<td></td>
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</tr>
<tr>
<td>600-grit sanded</td>
<td>17.2</td>
<td>1.8</td>
<td>164+</td>
<td>Parent metal</td>
</tr>
<tr>
<td>21.3</td>
<td>3.09</td>
<td>(e)</td>
<td>Weld</td>
<td>Shear</td>
</tr>
<tr>
<td>20.9</td>
<td>3.02</td>
<td>1.7</td>
<td>Weld</td>
<td>Shear</td>
</tr>
<tr>
<td>19.0</td>
<td>2.76</td>
<td>18.6</td>
<td>Parent metal</td>
<td>Tensile and shear</td>
</tr>
<tr>
<td>Specially processed to commercial material</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>As-received 600-grit sanded</td>
<td>18.5</td>
<td>2.68</td>
<td>160+</td>
<td>Parent metal</td>
</tr>
<tr>
<td>As-received 600-grit sanded</td>
<td>18.7</td>
<td>2.72</td>
<td>281+</td>
<td></td>
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<tr>
<td>As-received 600-grit sanded</td>
<td>18.6</td>
<td>2.69</td>
<td>138.4</td>
<td></td>
</tr>
</tbody>
</table>

aAll specimens were chemically polished after sanding; for specially processed to commercial material section the specially processed surface preparation appears first.
bTest discontinued, no failure.
cAt room temperature on removal.
dOn cooling.
eFailed on loading.
Figure 1. - Effect of temperature on tensile elongation of specially processed TD-NiCr sheet (unpublished data from General Dynamics/Convair).

Figure 2. - Configurations of diffusion-welded and creep-rupture shear specimens. (Dimensions are in millimeters (in.).)
As-received, 320-grit sanded, or 600-grit sanded surfaces

Detergent cleaning

Hydrochloric acid - 92 percent
Nitric acid - 31 percent
Sulfuric acid - 5 percent
90°C for 3 min

Distilled water rinse

Nitric acid - 40 percent
Hydrofluoric acid - 20 percent
Water - 40 percent
90°C for 3 min

Distilled water rinse

Methyl alcohol rinse

Storage in trichlorotrifluoroethane

Figure 3. Flow diagram for chemical polishing of weld specimen surfaces. All compositions are in volume percent.
(a) Specially processed material; typical parent-metal tensile and bending failure.

(b) Commercial material; shear failure at weld line during cooling to room temperature with minimal external load.

Figure 5. - Cross sections of diffusion lap welds in 0.4-millimeter-(0.015-in.-) thick TD-NiCr sheet that have been notched to 2t overlap and creep-rupture shear tested at 1100° C (2012° F) in air. X100.

Figure 6. - Effect of material difference on weld quality in TD-NiCr sheet obtained by using recommended surface preparation and weld cycles. Welds were postheated at 1180° C (2150° F) for 2 hours in hydrogen.
Figure 7. - Effect of diffusion welding specially processed to commercial TD-NiCr on elimination of weld line. Weld was postheated at 1180°C (2150°F) for 2 hours in hydrogen.

Figure 8. - Effect of as-received plus chemically polished surface preparation on formation of semicontinuous weld line in specially processed TD-NiCr sheet. Weld was postheated at 1180°C (2150°F) for 2 hours in hydrogen. X100.

(a) Recommended diffusion welding cycle (760°C, 1400°F; 275 MN/m², 40 ksi; 1 hr).

(b) Increased welding pressure used (760°C, 1400°F; 310 MN/m², 45 ksi; 1 hr).

Figure 9. - Effect of as-received plus chemically polished surface preparation on weld quality in commercial TD-NiCr sheet. Welds were postheated at 1180°C (2150°F) for 2 hours in hydrogen. X100.
Figure 10. Effect of excessive deformation on diffusion weld in specially processed TD-NiCr sheet after recrystallization heat treatment of 1180°F (2150°C) for 2 hours in hydrogen.
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