Hydrogen Segregation in Ti-6 Al-4V Weldments Made With Unalloyed Titanium Filler Metal

Investigation indicates that hydride banding should not be an uncommon occurrence and suggests caution in the use of unalloyed filler metal when welding Ti-Al-4V alloy

BY D. N. WILLIAMS, B. G. KOEHL AND R. A. MUELLER

ABSTRACT. Bands of hydrides have been observed along the fusion zone:heat-affected zone interface in a Ti-6Al-4V weldment made using unalloyed titanium filler metal and are believed to have been the cause of failure of the weldment. A program was conducted to determine the conditions under which hydride segregation would occur in this type of weldment and to determine the effects of hydride segregation on mechanical properties.

During these studies, hydride banding was developed in welds in which the degree of weld dilution was 35%. Hydride banding appeared to be favored more by high hydrogen in the Ti-6Al-4V sheet than in the filler metal. Although obvious hydride segregation was developed, no detrimental effects on mechanical properties were encountered. Mechanical properties were measured using unnotched tensile tests at two strain rates, notched tensile tests, notched rupture tests, and constrained bend tests.

Despite the failure to observe a significant decrease in mechanical properties, the observation of hydride banding at a weld dilution level as high as 35% is considered to be cause for concern. The severity of banding, and its effect on properties, should increase as the weld dilution level decreases.

Introduction

A helium pressure vessel constructed of heat treated Ti-6Al-4V failed leading to the destruction of a Saturn S-IV B rocket during a test sequence.[1] Unalloyed titanium filler metal was inadvertently used in making the tank girth weld, a multipass weld joining 0.4 in. thick hemispheres. Although both the unalloyed filler metal and the Ti-6Al-4V hemispheres contained less than 100 ppm hydrogen, extensive hydride segregation was found at the weld metal:heat-affected zone interface. A major composition change also occurred at this point, the composition changing from essentially pure titanium to Ti-6Al-4V.[2] Since the pressure vessel had survived proof testing at pressures well above the service pressure, it appeared that failure may have resulted from hydride precipitation during service rather than from low weld strength, and that the use of unalloyed titanium filler metal was a contributing factor rather than the direct cause of failure.

The use of unalloyed filler metal in the welding of Ti-6Al-4V is common practice. However, there is usually a significant amount of weld dilution, such that the weld metal contains appreciable aluminum and vanadium after welding, and no weld failures attributed to hydride formation in this type of weldment have been reported. Nevertheless, the failure cited above indicated the need for an investigation of the conditions leading to hydride formation in Ti-6Al-4V weldments made with unalloyed filler metal. Factors of importance were considered to be hydrogen content of components and amount of weld dilution.

The objective of the program described in the present paper was to determine the conditions under which hydride segregation along the fusion zone:heat-affected zone interface might occur in Ti-6Al-4V welded with unalloyed filler metal and to determine the effects of hydride segregation on the mechanical properties of the weldment.

Materials

Ti-50A (unalloyed) and Ti-6Al-4V materials were obtained in the form of 36 in. long, 0.125 in. thick sheet. The Ti-50A was 2 in. wide, the Ti-6Al-4V, 4 in. wide. The analyses are shown in Table 1.

The Ti-6Al-4V sheet was hydrogenated and heat treated at Battelle to produce the desired hydrogen contents of 50 and 150 ppm and microstructure of 50% equiaxed alpha. Hydrogen levels of 50 and 150 ppm were selected as representing the limiting range of hydrogen content apt to be present in Ti-6Al-4V sheet. A hydrogen content of no more than 150 ppm is required by Aerospace Materials Specification 4928. The microstructural condition selected was that present in the failed pressure vessel and is representative of that considered desirable for optimum properties in heat treated Ti-6Al-4V.

Welds were made to join solution heat treated and partially aged Ti-6Al-4V. The aging treatment was completed after welding, according to common practice, to attain some degree of stress relief. The heat treatment procedure which was performed

Table 1—Analyses of the Ti-50A and Ti-6A1-4V Sheet, Wt-%

Material	Heat no.	C	Fe	Al	V	H	O	N
Ti-50A	G5411	0.022	0.10	—	—	0.0030	0.11	0.008
Ti-6A1-4V	G5398	0.025	0.10	6.0	4.3	0.0030	0.10	0.015
[a]	G4820	0.026	0.11	6.0	4.1	0.0050	0.11	0.014

[a] Two heats of the same material were received.

D. N. WILLIAMS, B. G. KOEHL and R. A. MUELLER are with Battelle Memorial Institute, Columbus, Ohio.

Paper to be presented at the AWS 51st Annual Meeting held in Cleveland, Ohio, during June 8-12, 1970.

Table 2—Hydrogen Content of Medium Dilution Weldments

Weldment no.	Hydrogen content of Ti-6A1-4V sheet, ppm — Desired	Hydrogen content of Ti-6A1-4V sheet, ppm — Actual	Hydrogen content of Ti-50 filler metal, ppm — Desired	Hydrogen content of Ti-50 filler metal, ppm — Actual
1[a]	50	63	50	64
	50	—		
2[b]	150	189	50	64
	150	188		
3[b]	150	192	150	145
	150	313[d]		
4[a]	50	72	150	145
	50	—		
5[b, c]	150	170	150	136
	150	178		

[a] Ti-6A1-4V plate from heat G4820 used for this weldment.
[b] Ti-6A1-4V plate from heat G5398 used for this weldment.
[c] *Ti-6A1-4V filler metal for this weldment was from heat G5398.*
[d] Notches in test bars were located at the interface between the filler metal and the Ti-6A1-4V plate containing 192 ppm hydrogen.

Table 3—Welding Parameters

	1st pass	2nd pass
Arc voltage, v	13–14	13–14
Weld current, amp	159–160	159–160
Travel speed, ipm	8	8
Electrode (negative)	$^3/_{32}$ in. diameter—1% thoriated tungsten	
Electrode stick-out, in.	$^3/_4$	$^3/_4$

after hydrogenation was as follows:

1. Solution heat treat for 10 min. at 1740° F in air.
2. Water quench.
3. Surface grind to remove 3 mils per side.
4. Age between clamped stainless steel plates (to flatten) for 1½ hr at 1000° F and air cool.
5. Pickle lightly.
6. After welding, age for 2½ hr at 1000° F between clamped stainless steel plates and air cool.

The Ti-50A sheet was degreased thoroughly and used with no annealing treatment other than that applied during hydrogenation. Hydrogen contents of 50 and 150 ppm were introduced into this material at Battelle.

All material was cut into 18 in. lengths for easier accommodation in the equipment which was used for the hydrogen addition.

Experimental Procedures

In order to obtain the necessary hydogen levels, sheets of both Ti-50A and Ti-6Al-4V were hydrogenated by exposure to gaseous hydrogen at elevated temperatures. The sheets were sealed inside a Type 304 stainless steel tube which was attached to a modified Sieverts apparatus which was used to introduce a measured amount of hydrogen. The sheets were heated to 1350–1400° F by heating tapes wound on the outside of the tube. The desired quantities of hydrogen were added and the progress of the hydrogenization was followed with a mercury manometer. After absorption of the added hydrogen, usually in 15 to 30 min, the sheets were maintained at temperature for 1 to 2 additional hours to allow for homogenization of the hydrogen in the material, then cooled slowly to room temperature. This equipment proved difficult to control exactly, and the desired hydrogen contents were not always obtained.

It was originally planned to prepare weldments at low ($\approx$ 0%) medium ($\approx$ 30%), and high ($\approx$ 50%) dilution levels. Weld dilution was calculated as follows:

$$\frac{A}{A+B} \times 100 = \text{percent weld dilution}$$

where B is the volume of filler metal and A is the volume of base metal in the weld metal.[3]

While hydrogenation and heat treatment of material were in progress, work was initiated on development of weld procedures to control weld dilution. Despite extensive variations in welding conditions and joint design, it was not possible to obtain a weld dilution level of less than 35% while obtaining complete weld fusion. Moreover it was not possible to increase the weld dilution much above the 35% level without creating an excessively large weld bead. As a result, it was decided to discontinue efforts to produce the majority of the planned weldments and to concentrate upon evaluation of medium dilution weldments. These weldments included four combinations of hydrogen in Ti-6Al-4V and Ti-50A as follows:

Weldment no.	*Ti-6Al-4V sheet*	*Ti-50A filler metal*
1	Low hydrogen	Low hydrogen
2	High hydrogen	Low hydrogen
3	High hydrogen	High hydrogen
4	Low hydrogen	High hydrogen

A fifth weldment (no. 5) was prepared using high hydrogen Ti-6Al-4V sheet and high hydrogen Ti-6Al-4V filler metal. The hydrogen contents of these medium dilution weldments are shown in Table 2. As shown here, the hydrogen content of most components of these weldments was somewhat higher than the desired level.

The titanium alloy sheets were welded using the gas tungsten-arc process in a vacuum-purged (<5 microns) argon back-filled welding chamber. The chamber was equipped with a movable work table providing precise control of the travel speed. An

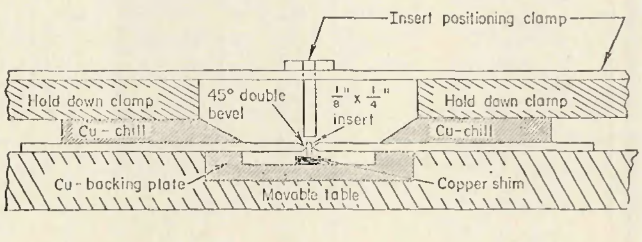
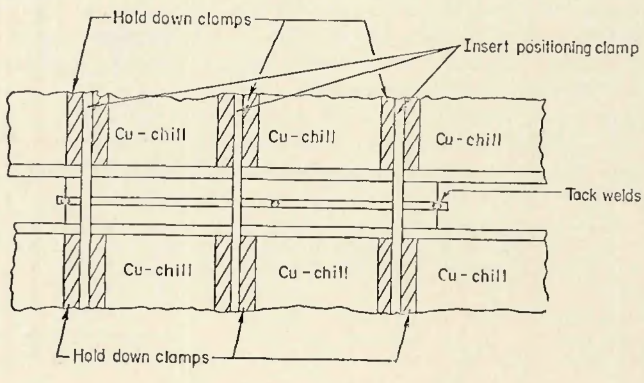


Fig. 1—Joint configuration and tooling for production of the weldments



automatic constant voltage controlled welding head, which maintains the proper arc length, and a d-c power supply with a high frequency start capability was used.

Each weldment was made from two 18 x 4 in. titanium sheets surface ground to a thickness of 0.110 in. and one 20 x 1/4 x 1/8 in. titanium insert. The insert constituted the filler metal. A double 45 deg. bevel was machined along one 18 in. edge of each Ti-6Al-4V sheet. The other 18 in. edge was faced off so that the total width of each pair of sheets was exactly 8 in.

Figure 1 shows, schematically, the joint configuration and tooling. The insert was placed between the 45 deg. bevels of each sheet. Two copper bars were used to provide a controlled chill and holddown tooling for assembly. The bars were placed about 1 in. from the weld center line. Steel holddown clamps were placed about every six inches along the length of the chill bars to provide the holddown force. Tooling at the bottom of the joint consisted of a copper backing plate inserted into the work table. The backing plate provided the bottom chill and was machined flush with the top of the work table. A 2 in. wide x 1/4 in. deep slot was machined into the backing plate so that it was not in contact with the filler metal insert or Ti-6Al-4V sheet near the weld area.

The vertical position of the filler metal insert was obtained by placing three small copper shims along the insert length between the insert and the backing plate. At these same locations on the top, steel bars containing a screw were used to clamp the insert into position.

The titanium alloy sheets and filler metal insert were inserted into the chamber and the tooling was clamped into place. After final alignment, the chamber was purged and back-filled with the high-purity argon to a slight positive pressure. Tack welds were then placed in the middle and at each end of the insert. The insert positioning clamps and copper shims were removed, and the tacked assembly reclamped and realigned. The top weld was then made along the length of the plates. The first pass melted the top portion of the insert achieving about 55% penetration. The partially welded assembly was then removed from the tooling, rotated 180 deg. about the longitudinal weld axis, reclamped, and realigned. The second pass weld was then made to complete the joint.

The range of the welding parameters for each pass as indicated by a recording volt-ammeter is shown in Table 3 along with other parameter information.

This technique resulted in a weld dilution of approximately 35% when using unalloyed titanium filler metal. Analysis of the weld metal showed it to contain approximately 2.0% aluminum and 1.4% vanadium.*

Upon completion of the welding, the weldments were aged an additional 2½ hr at 1000° F in air and air cooled, then ground flat. Test samples were cut from the weldments as shown in Fig. 2 to provide material for bend, tensile, notched tensile, notched stress rupture, and metallographic tests. Constrained bend samples measured 3/4 x 8 in. and were fastened onto a radiused steel mandrel to provide a maximum surface strain of 0.33%. Notched tensile and stress rupture samples were machined to provide a stress concentration factor (K_t) of 6 as shown in Fig. 2b. The notch was located in the weldment, such that the root of the notch was offset slightly from the centerline of the weld nugget—Fig. 2b. The notch cut through regions of weld metal and heat-affected zone and in some locations coincided with the hydride. Unnotched tensile samples were machined according to the drawing in Fig. 2c.

* Measurements of weld dilution were made only on test welds. Since it was found that weld dilution was reasonably constant with the technique used, it was considered unnecessary to run analyses on the five weldments prepared for mechanical property studies.

Results

Because of the significant weld dilution which was present in these samples, it was initially thought that segregation of hydrogen would not be obtained. However, as shown in Fig. 3, several of the welds showed obvious hydrogen segregation appearing as bands of hydride along portions of the fusion zone:heat affected zone interface. Banding was evident only along the interface formed in the second weld pass. The banding along this interface was most prominent in weldment 3 where both the base metal and filler metal were high in hydrogen—Fig. 3c. Weldment 2, made with low hydrogen filler metal and high hydrogen Ti-6Al-4V, also showed obvious banding—Fig. 3b.

Only a slight indication of banding is seen on the two welds made with unalloyed filler metal and low hydrogen Ti-6Al-4V, weldments 1 and 4 (Figs. 3a and 3d), while no hydride was seen in weldment 5 made with Ti-6Al-4V filler metal even though both base metal and filler metal contained high hydrogen contents (Fig. 3e). The amount of hydride distributed through the weld nugget appeared to decrease in the following order: 3 (Fig. 3c), 2 (Fig. 3b), 4 (Fig. 3d), 1 (Fig. 3a), and 5 (Fig.

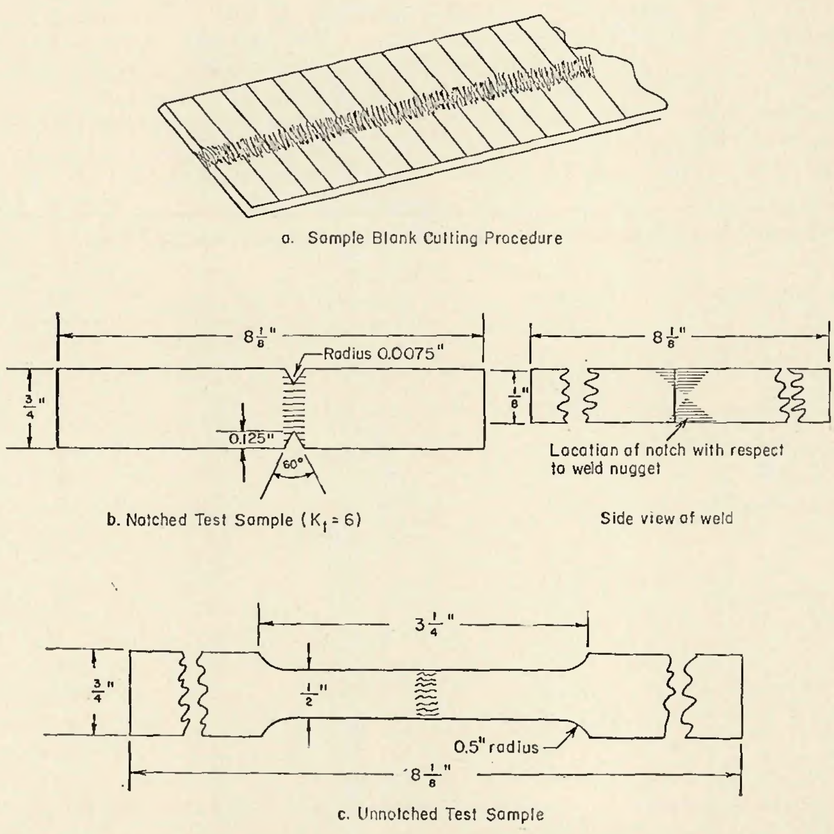
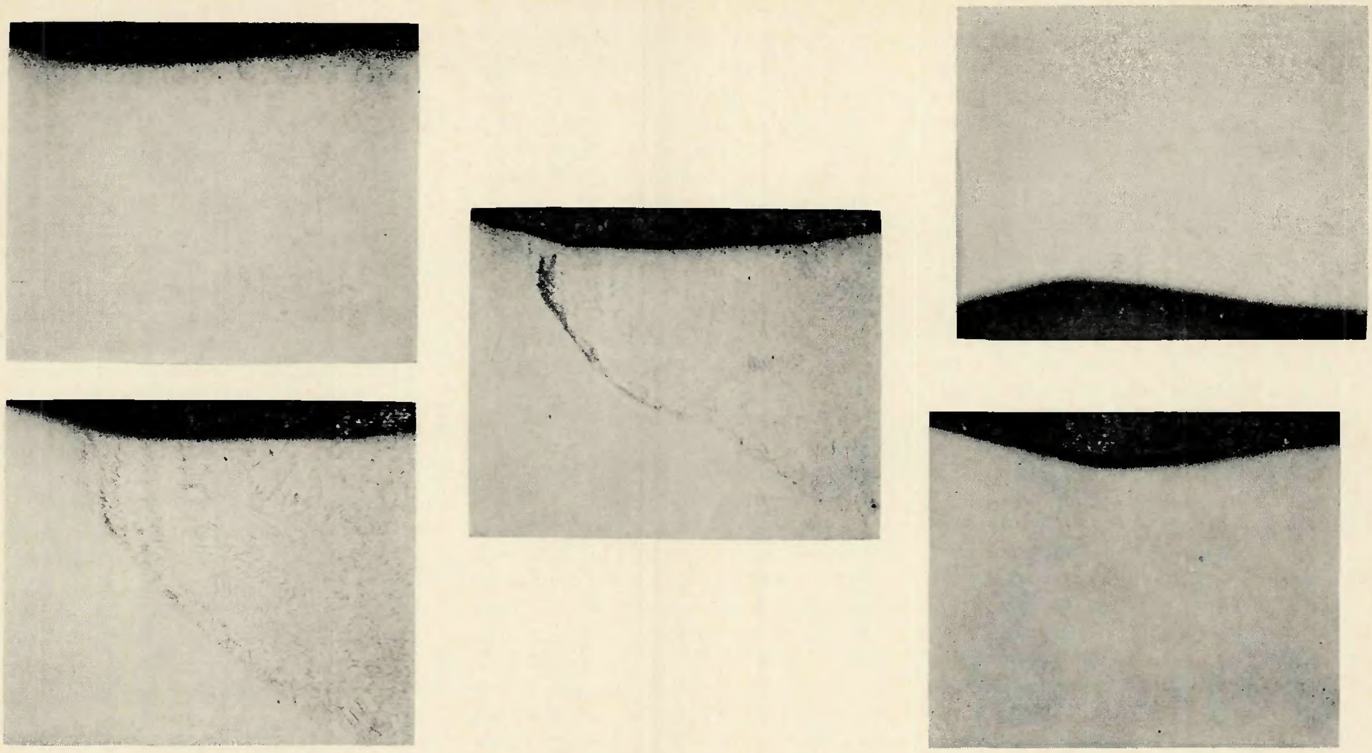


Fig. 2—Test sample location and sample dimension



Weldment no.	1	2	3	4	5
Base metal H_2 (ppm)	65	190	190	70	170
Filler metal H_2 (ppm)	65	65	145	145	135

Fig. 3—Hydride precipitated in weld area with Ti-6Al-4V base metal at left and Ti-50A filler metal at right in weldments 1-5. Top left (a)—weldment no. 1; bottom left (b)—weldment no. 2; center (c)—weldment no. 3; top right (d)—weldment no. 4; bottom right (e)—weldment no. 5. X50 (reduced 50% on reproduction)

3e).

Since the hydrogen content was initially over twice as high in the filler metal used to make weldments 3 and 4 as in the filler metal used to make weldments 1 and 2, it appears that significant quantities of hydrogen passed from the Ti-6Al-4V sheet to the weld metal during welding to cause the large amount of hydride observed in weldment 2. As will be noted later, it is possible that weldment 2 was contaminated slightly during welding, and this factor may have contributed to the larger than expected amount of hydride formation. The absence of hydrides in weldment 5 is due to the high solubility of hydrogen in Ti-6Al-4V.

When hydrides are present in titanium alloys, they normally result in a loss in tensile ductility in high strain rate unnotched tensile tests and a loss in tensile strength and ductility in notched tensile tests. This type of embrittlement is known as impact embrittlement. Unnotched tensile tests were run at two cross head speeds, slow (0.005 in./min. to yielding and 0.05 in./min. to failure) and fast (0.5 in./min.), and notched tensile tests were run at 0.005 in./min. in an effort to detect impact embrittlement. All tests were conducted at room temperature.

Table 4—Results of Unnotched Tensile Tests Performed at a Slow Test Rate[a]

Weldment no.	Ultimate strength, psi	Yield strength psi	Elongation, %[b]	Reduction in area, %
1	102,000	88,400	5.5	34
	101.200	89,600	5.0	32
Average	101,600	89,000	5.2	33
2	104,200	89,600	5.0	24
	104,800	89,900	4.0	26
Average	104,500	89,800	4.5	25
3	98.900	84,300	4.0	27
	102.300	89,000	4.0	31
Average	100,600	86,600	4.0	29
4	101,200	86,400	4.5	31
	100,300	84,900	4.5	29
Average	100,800	85,600	4.5	30
5	141,500	123,500	5.0	23
	141,900	123,400	4.5	16
Average	141,700	123,400	4.8	20

[a] Cross head speed of 0.005 ipm to yield, then 0.05 ipm to failure.
[b] Measured between 2 in. gage marks. All samples fractured in weld metal.

The results of duplicate unnotched tensile tests performed at a slow testing rate are shown in Table 4. It is seen that weldment 1, in which both filler metal and base metal were low in hydrogen has the highest ductility of the four weldments made with unalloyed filler metal. Weldment 5, made with Ti-6Al-4V filler metal, shows considerably higher strength than the other four weldments, as would be expected, and also shows good ductility. Weldment 2 shows a higher ultimate strength and lower reduction in area than weldments 1, 3, or 4. This is tentatively attributed to slight oxygen contamination during welding.

If the yield strength of weldment 2 is also assumed to have been increased by contamination, perhaps as much as 3000 psi higher than it would normally have been, weldment 1 is seen to

Table 5—Results of Unnotched Tensile Tests Performed at 0.5 ipm

Weldment no.[a]	Tensile strength, psi	Elongation, %[a]	Reduction in area, %
1	106,500	5	32
	104,600	5	36
Average	105,600	5	34
2	109,600	4	19
	110,400	4	18
Average	110,000	4	18
3	104,600	5	29
	106,000	4	28
Average	105,300	4.5	28
4	105,400	5	37
	105,800	5	35
Average	105,600	5	36
5	148,900	5	24
	147,700	5	20
Average	148,300	5	22

[a] Measured between 2 in. gage marks. All samples fractured in weld metal.

Table 6—Results of Notched Tensile Tests (K_t = 6)

Weldment no.	Notched tensile strength, psi	Elongation, %[b]	Notched: unnotched strength ratio[a]
1	119,800	4	
	116,500	3	
Average	118,200	3.5	1.16
2	117,200	3	
	118,700	3	
Average	118,000	3.0	1.13
3	112,600	2	
	115,300	3	
Average	114,000	2.5	1.13
4	113,200	3	
	114,700	3	
Average	114,000	3.0	1.13
5	153,000	3	
	147,100	1	
Average	150,000	2.0	1.06

[a] Based on comparisons with the results of unnotched tensile tests run at a slow strain rate (Table 4).
[b] Measured between 1/2 in. gage marks.

Table 7—Results of Notched Stress Rupture Tests Performed at Room Temperature

Weldment no.	Stress, psi	Time to failure, hr
1	111,900	0.2
	105,600	0.7
	97,000	112.4 discontinued
2	116,600	0.01
	110,000	0.3
	97,000	92.5
3	111,600	0.03
	100,000	43.6
	97,000	3.0
	96,000	37.0
4	111,900	0.05
	100,300	6.4
	97,000	100.0 discontinued
5	148,300	0.06
	133,500	0.10
	120,000	100.0 discontinued

have the highest yield strength of the four weldments made with unalloyed filler metal. Except for slight differences in ductility and possibly yield strength, the hydride distribution had little effect on tensile properties at a slow testing speed.

The results of duplicate unnotched tensile tests performed at a fast testing speed are shown in Table 5. As was observed in slow tensile tests, weldment 2 was somewhat stronger and less ductile than weldments, 1, 3, or 4, while weldment 5 was considerably stronger than the other four. The ductility differences between the four weldments made with unalloyed filler metal are small except for the reduction in area of weldment 2 and are probably not significant. On the other hand, weldments 1 and 2, which showed obvious hydride banding, were the least ductile. Some contamination of weldment 2 is also indicated by the tensile data attained in a fast strain rate.

The results of duplicate notched tensile tests are shown in Table 6. In these tests, the ductility correlated with the metallographic observations for the four weldments made using unalloyed filler metal. Moreover, the notched strength and the notched: unnotched strength ratio suggested that weldment no. 1, in which both base metal and filler metal were low in hydrogen, was superior to the other three. These differences were quite small, however. Weldment 5, made with Ti-6Al-4V filler metal, was more notch-sensitive than any of the

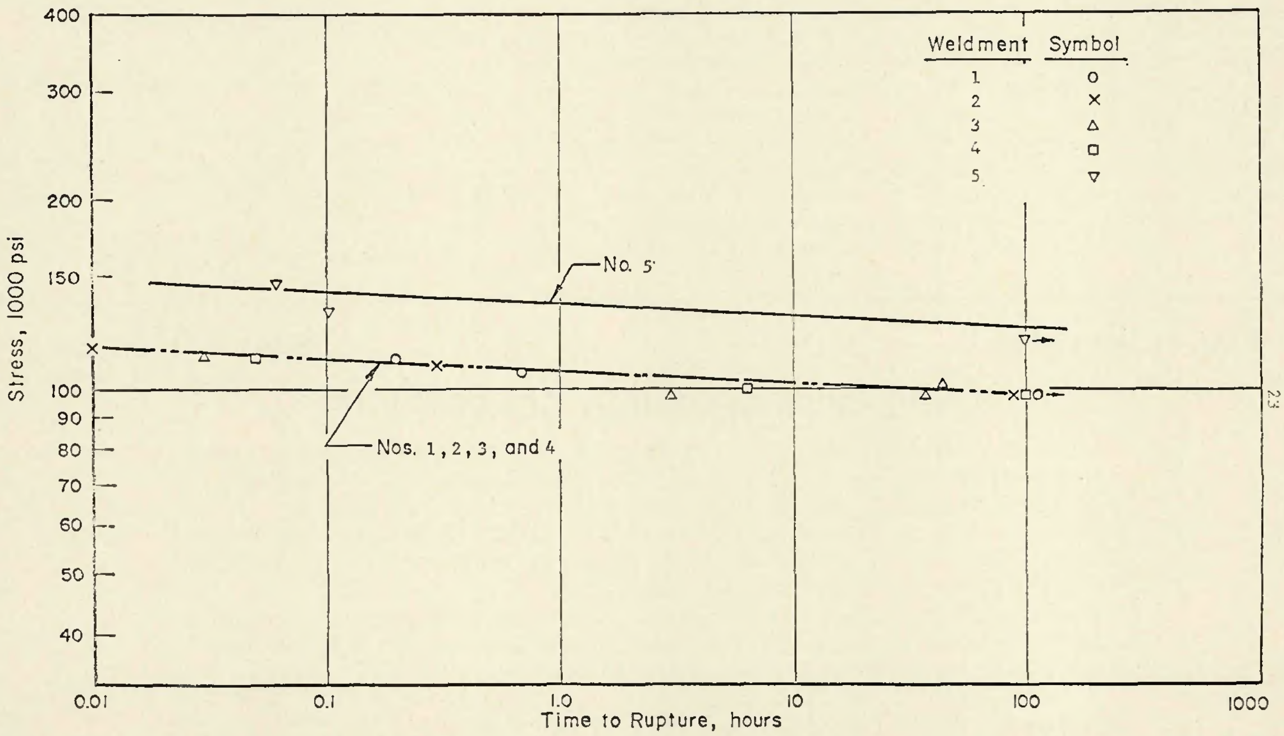


Fig. 4—Stress rupture curves for notched sheet samples at room temperature

weldments made with unalloyed filler metal as indicated by a lower notched:unnotched strength ratio.

It was obvious from these tests that the hydride present in the weldments had little effect on tensile properties. There was no evidence of significant impact embrittlement. Since the pressure vessel failure discussed in the Introduction occurred after considerable service under stress, it was considered possible that additional hydride precipitation or hydride agglomeration might occur during prolonged loading—that is, the weldments would be susceptible to the type of hydrogen embrittlement referred to as low strain rate embrittlement. Two test procedures were used to detect low strain rate embrittlement—notched stress rupture tests and constrained bend tests.

The results of notched stress rupture tests on the five weldments are presented in Table 7 and plotted in Fig. 4. Although there is some scatter in the data, it appears that all four of the weldments made with unalloyed filler metal had approximately the same 100 hr rupture strength (about 95,000 psi), while the rupture strength of the weldment made with Ti-6Al-4V was about 25,000 psi higher. Samples from weldments 3 and 2 failed in tests at 97,000 psi, while samples from weldments 1 and 4 did not fail within 100 hr. A sample of weldment 3 was also placed in test at 96,000 psi and failed in 37 hr. However, as shown in Fig. 4, these results are well within the normal scatter band of a single rupture curve including data from all four weldments made with unalloyed filler metal. Low strain rate embrittlement is not an obvious factor in these results.

Constrained bend tests were run in two ways. In the first tests, a sample of each weldment was fastened to a curved steel block at room temperature. The radius of the block was such that the sample outer fibers were strained in tension 0.33% transverse to the weld. In the second test, both the block and the sample were heated to 400° F for 17 hr, the samples were attached to the steel block, and the strained samples and test block were cooled to room temperature. It was hoped that moderate heating followed by slow cooling under stress would accelerate hydrogen redistribution. In all cases, the second weld pass was placed in tension. Both groups of samples were examined periodically for cracks in the weld regions using a fluorescent penetrant technique, but none were found. The samples stressed at room temperature were held in test for 51 days. Those stressed at 400° F were held in test at room temperature for 23 days.

The results of the notched stress rupture and constrained bend tests showed no evidence of low strain rate embrittlement. Since the notch stress rupture test is quite sensitive to low strain rate embrittlement tendencies, it is concluded that these weldments are not susceptible to low strain rate embrittlement. This was not unexpected since most of the hydrogen present was obviously already precipitated in the form of hydrides. Only that portion in solution can contribute to low strain rate embrittlement.

Despite the failure to detect evidence of significant embrittlement in these weldments, the observation that a major segregation tendency existed in weldments involving Ti-6Al-4V sheet and unalloyed titanium filler metal, even when a moderate amount of weld dilution was present, is disturbing. Hydride banding of the type observed, although apparently not harmful in the weldments studied, is likely to prove quite damaging if it becomes much more extensive. Hydride banding will undoubtedly increase in severity as weld dilution is decreased. Further studies of the factors leading to hydride banding and its effect on properties are needed.

Conclusion

During the course of this study, it was possible to produce hydride banding along the fusion zone:heat-affected zone interface of Ti-6Al-4V sheet welded with unalloyed filler metal. Bands of hydride were present along the fusion zone:heat-affected zone interface in two of five weldments prepared in this study. The hydrogen contents of the base metal and filler metal used to prepare these weldments were only slightly above current specification limits and weld dilution was about 35%.

It is thus concluded that hydride banding should not be an uncommon occurrence in welds of this type. However, despite the fairly extensive hydride banding observed, no significant evidence of hydrogen embrittlement, either impact embrittlement or low strain rate embrittlement, was detected in mechanical property studies. Apparently, the amount of hydride segregation present, although quite obvious metallographically, was too small to cause embrittlement.

Acknowledgement

These studies were supported by the National Aeronautics and Space Administration, Manned Spacecraft Center, Houston, Texas, under Contract NAS 9-7549. Mr. S. V. Glorioso of NASA Manned Spacecraft Center provided technical management. His interest in and contributions to these studies have been a significant factor in their successful completion.

References

1. "S-4B Explosion", Aviation Week & Space Technology, p 22 (February 20, 1967).
2. Henry, D. L., and Whiteson, B. V., "Localized Migration of Hydrogen in Titanium Alloy Welds," paper presented at the 1969 WESTEC Metal and Tool Conference, Los Angeles, California (March 10, 1969).
3. American Welding Society, *Welding Handbook*, 6th Edition, Section 1, p 2.37.