NASA CONTRACTOR REPORT

NASA CR-1610

DETERMINATION OF WELDABILITY AND ELEVATED TEMPERATURE STABILITY OF REFRACTORY METAL ALLOYS

IV – Post Weld Annealing Studies of T-111

by G. G. Lessmann

Prepared by
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for

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16. Abstract

The weld structure of T-111 shows some aging effects (over the range 1500° to 2400° F or 815° to 1316° C) even after aging at 2400° to 3000° F (1316° to 1649° C) for 1 hour. The aging was followed by the bend DBTT (ductile-to-brittle-transition temperature) test and study of the microstructure. No post weld annealing cycle totally eliminated the aging effect. A mechanism to explain the complicated observed aging phenomenon is postulated. The loss of ductility observed does not result in welds which cannot be used because of low ductility. (Tensile ductility is $<$ 23 percent.) It is concluded that 2400° F (1316° C) for 1 hour is a satisfactory post weld anneal temperature.

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FOREWORD

This evaluation was conducted by the Westinghouse Astronuclear Laboratory under NASA contract NAS 3-2540. Mr. P. E. Moorehead, of the Lewis Research Center Space Power Systems Division, was the NASA Project Manager for this program. Mr. G. G. Lessmann was responsible for performance of this program at the Westinghouse Astronuclear Laboratory.

The objectives delineated and results reported herein represent the requirements of Task V of contract NAS 3-2540. Additional comprehensive investigations which were conducted as a part of this program are the subjects of additional reports. The final reports for this contract are the following:

I - Weldability of Refractory Metal Alloys (CR-1607)

II - Long-Time Elevated Temperature Stability of Refractory Metal Alloys (CR-1608)

III - Effect of Contamination Level on Weldability of Refractory Metal Alloys (CR-1609)

IV - Post Weld Annealing Studies of T-111 (CR-1610)

V - Weldability of Tungsten Base Alloys (CR-1611)

Additional salient features of this program have been summarized in the following reports:

G. G. Lessmann, "The Comparative Weldability of Refractory Metal Alloys," The Welding Journal Research Supplement, Vol. 45 (12), December, 1966.

G. G. Lessman and R. E. Gold, "The Weldability of Tungsten Base Alloys," The Welding Journal Research Supplement.

D. R. Stoner and G. G. Lessmann, "Measurement and Control of Weld Chamber Atmospheres," The Welding Journal Research Supplement, Vol. 30 (8), August, 1965.

G. G. Lessmann and D. R. Stoner, "Welding Refractory Metal Alloys for Space Power System Applications," Presented at the 9th National SAMPE Symposium on Joining of Materials for Aerospace Systems, November, 1965.

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I. INTRODUCTION

This investigation was initiated because T-111 welds responded to aging with an increase in the bend transition temperature.* The apparent mechanism of this response did not appear to compromise the usefulness of T-111 in long time application. However, the unquestioned importance of this alloy in space power technology dictated that a more complete understanding of this aging response be developed. Further, the observed behavior was found to be characteristic of several other gettered refractory metal alloys. Hence, this investigation was important not only in the application of T-111 but also on a general basis to the entire field of space power materials technology. Two basic approaches lend themselves to this situation:

> First, one can define a thermal treatment such as a post weld anneal which eliminates the response entirely. This was the purpose of this investigation, namely, to determine if the structure could be stabilized by annealing. As described in this report, the T-111 weld structure did not respond to this treatment.
>
> Second, the aging response can be investigated in greater depth to demonstrate that the alloys usefulness is not limited by the aging, or conversely to define any application limits due to aging. This latter approach has been pursued under Contract NAS 3-11827, "Fracture and Hot Resistance in T-111 and ASTAR-811C".

The particular aging response observed in T-111 welds is shown in Figure 1. This response, as measured by bend testing, is properly characterized as follows:

- The observed bend transitions cannot be viewed as classic transitions from ductile-to-brittle behavior. This stems from the fact that fractures did not propogate by brittle cleavage but rather by ductile tearing. Hence, the T-111 bend transitions merely represent the minimum temperature at which T-111 welds can sustain a 1t bend. This means that the transition curves of Figure 1 really define a go-no go limit for 33-1/3% outer fiber strain. This interpretation was verified by tensile testing aged weld specimens as indicated later in this report.

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*See CR-1608 listed in Foreword of this report.

- The primary fracture mode was by grain boundary tearing rather than by transgranular cleavage. Again, this indicates that the aging behavior cannot be described as a classic shift in the ductile-to-brittle transition temperature. The scanning electron fractographs of Figure 2 show the typical grain boundary fracture mode of aged T-111 weld metal.

- Aging had essentially no effect on the strength of T-111. This is shown in Figure 3. (This incidentally demonstrated the usefulness of the bend test as a screening tool since ductility as measured by bend testing is sensitive to a variety of subtle as well as pronounced metallurgical interactions.)

- The combination of observed fracture mode and excellent tensile stability indicated that the engineering application of T-111 was not limited by the aging response.

- Only weld metal displayed an aging response implying that constitutional segregation was responsible in part for the observed aging response.

- The aging response was accompanied by grain boundary and interdendritic precipitation in the weld and heat affected zones, Figure 4. A definite correlation between the observed precipitate and aging response was not established since the precipitates did not lend themselves to positive identification. However, their implied relationship to the aging response was accepted in postulating an aging mechanism as summarized in Section V, Conclusions.

This report then describes the effects of post weld annealing as a method of stabilizing T-111 weld structure, while future reports under Contract NAS 3-11827 will provide an engineering definition of the characteristics of the aged structure in T-111 welds.

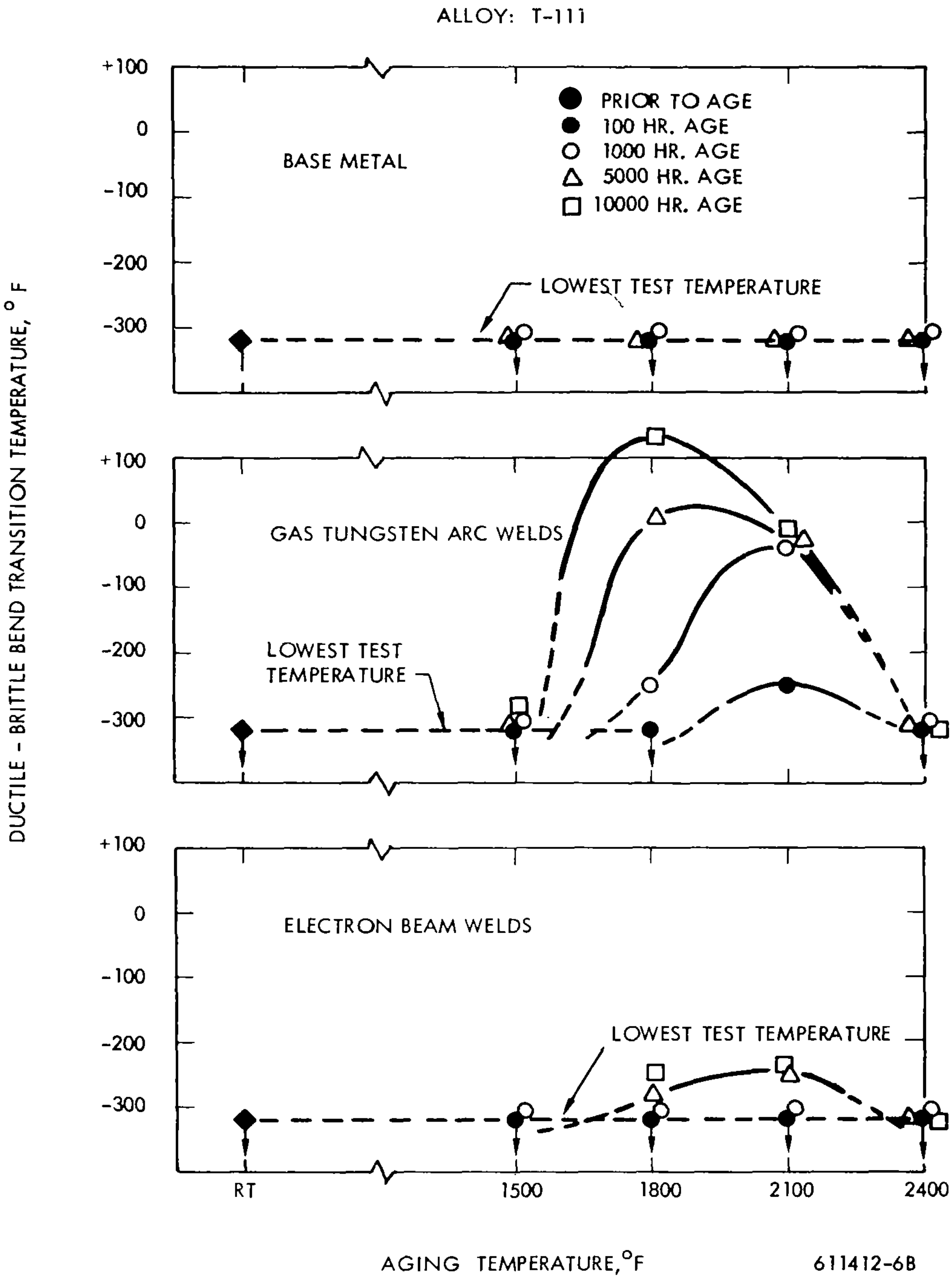
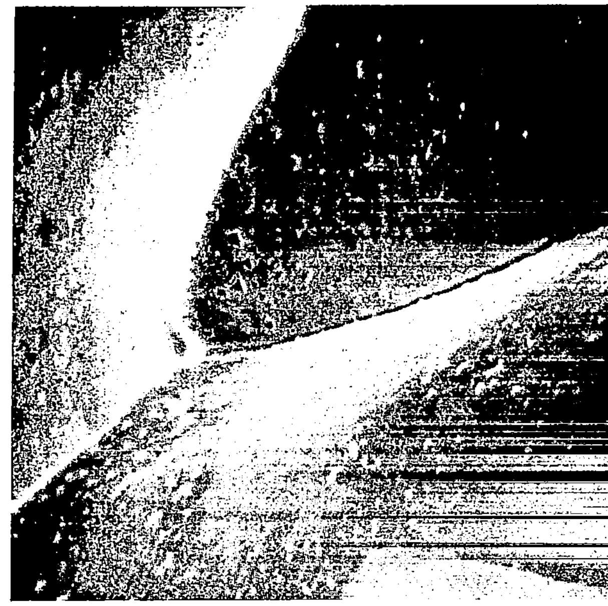


FIGURE 1 - Response of T-111 to Aging as Determined by Bend Testing (Data for 0.035 inch sheet, annealed 1 hour at 2400°F prior to aging, bend tested using a 1t radius.)

270X



2000X

820X

820X

FIGURE 2 - Scanning Electron Fractographs of T-111 Weld Specimen. Fractured by Bend Testing Following Aging 5000 Hours at 1800°F. Annealed 1 Hour at 2400°F Prior to Aging.

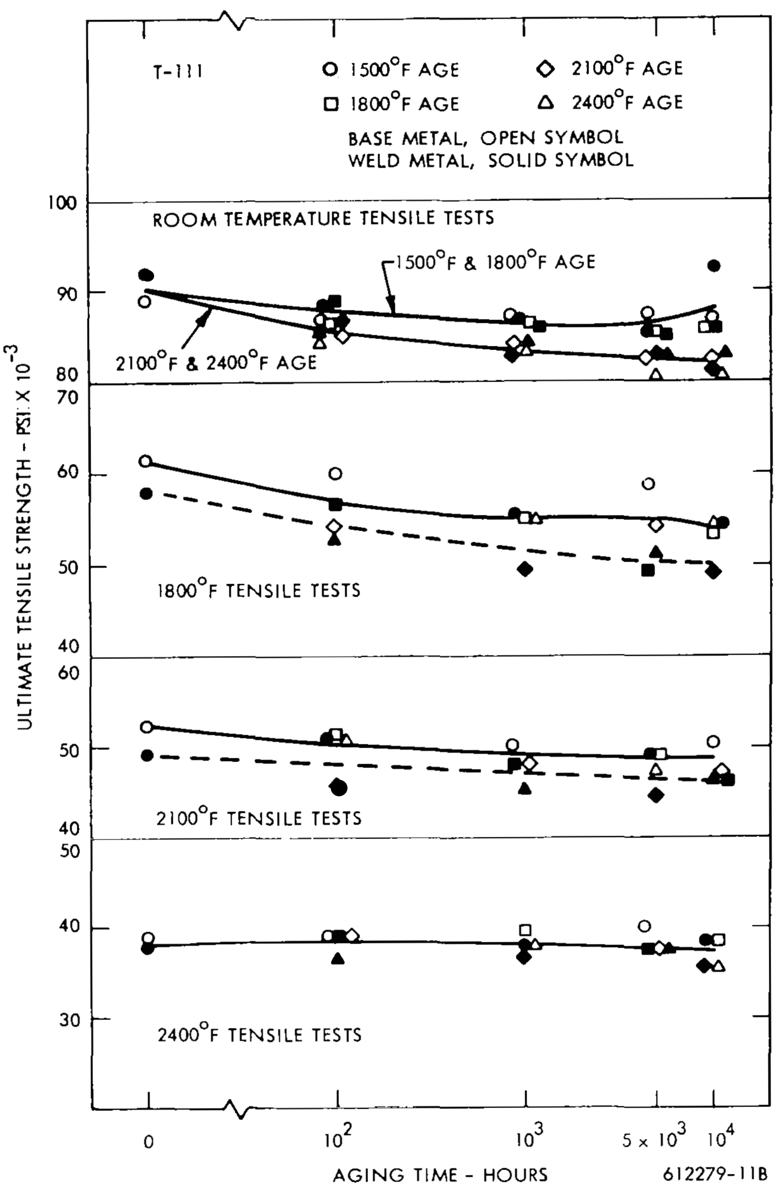
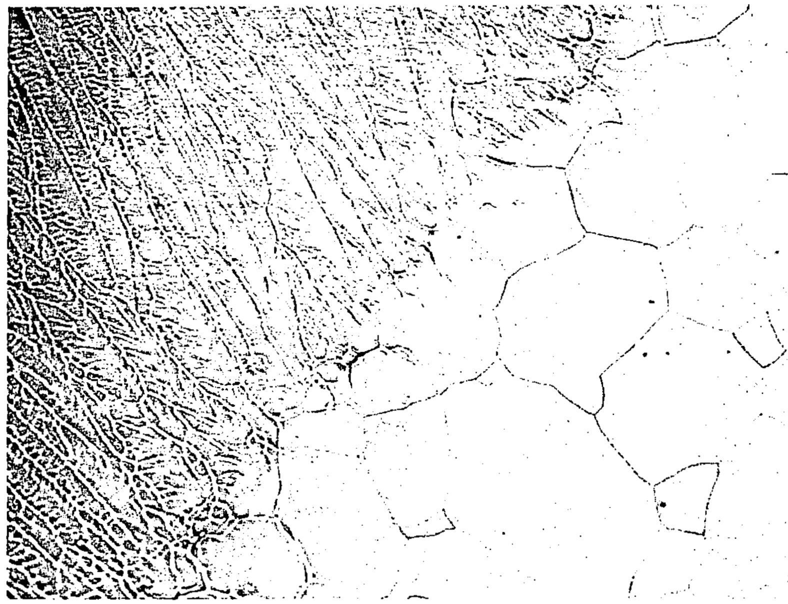
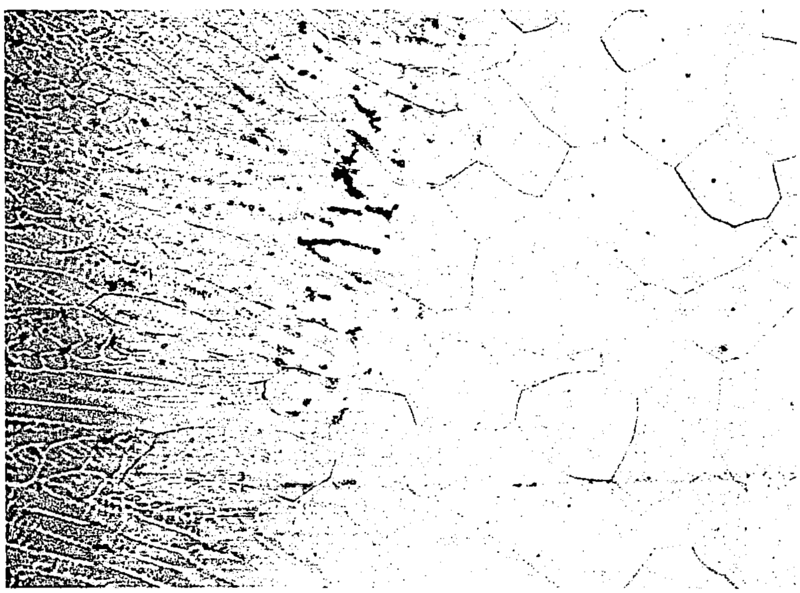


FIGURE 3 - Response of T-111 to Aging as Determined by Tensile Testing. (Data for 0.035 inch sheet tested transverse to both weld and rolling direction, annealed 1 hour at 2400°F prior to aging.)



200X

Weld Interface As Welded
Met. 16,697



200X

Weld Interface Aged 5000 Hours at 2100°F
Met. 18,268

FIGURE 4 - Effect of Aging on Weld Structure of T-111

II. TECHNICAL PROGRAM

The welding, annealing and aging parameters as well as the particular tests conducted are summarized in Table 1, Test Plan - Specimen Record. The basic program plan consisted of preparing specimens by welding, post weld annealing and aging employing the following variables:

- Two heats of T-111 were evaluated, see Table 2, one of which was also used in the Task III aging study. This was included as a pedigree reference heat.
- Three welding conditions were selected primarily for broad variability in total heat input. This was achieved using both the GTA and EB welding processes. Different welding speeds were used thereby screening for additional structural effects controlled by weld freezing rates such as grain and cell size, orientation and solute segregation. The parameters selected are indicated in Table I, Notes 2 and 3.
- Six different 1 hour post weld annealing temperatures up to 3000°F were investigated.
- Aging at 2100°F was evaluated for times of 100, 1000 and 5000 hours.

Evaluation consisted primarily of bend testing which was found to be an excellent screening tool in earlier tasks of this program. A 1t bend radius was used throughout. Both longitudinal and transverse bend transition temperatures were determined for each thermal history listed in Table I. A modest amount of tensile testing using longitudinal weld specimens was conducted in support of the bend testing. Tensile tests were conducted using a 0.005 in/in/min strain rate to 0.6% offset yield point and 0.05 in/in/min strain rate to failure. Specimen has a 1 inch by 0.250 inch wide gage section which contained the entire weld. Specimens were ground flat and parallel on surfaces. Hence, the finished gage thickness was 3 to 4 mils under the original sheet thickness. Metallographic examination was conducted as deemed necessary. Welding procedures and bend test procedures were qualified in Task I of this program and detailed in the Task I and II final report. Aging procedures were qualified in Task III. The salient

TABLE 1 - Test Plan-Specimen Record

	Welding(2,3)			Post Weld Anneal One Hour at Indicated Temperature							Aging Hrs. at 2100°F				Evaluation						
															Mechanical			Metallographic			
Identification No. (1)	GTA (Low Heat Input)	EB (Minimum Heat Input)	GTA, Slow Speed (High Heat Input)	As-Welded	2200°F	2400°F	2500°F	2600°F	2700°F	3000°F	0	100	1000	5000	Long. BDBTT	Trans. BDBTT	Long. Tensiles (2100°F)	Metallography (Optical)	Hardness Traverse	Phase Identification	Metallography ID Number
1	●			●							●				●	●		●	●		16,699
2A	●			●							●						●				
2B	●											●					●				
3	●			●								●			●	●		●	●		16,700
4	●			●									●		●	●		●	●		16,701
5A	●			●									●				●				
5B	●						●						●				●				
6	●					●							●		●	●		●	●		16,702
7	●						●						●		●	●					
8A	●								●				●				●				
9	●							●					●		●	●		●	●		16,704
10	●								●				●		●	●					
11A	●								●					●			●				
12	●									●			●		●	●		●	●		16,705
13	●						●							●	●	●		●			18,264
8B	●						●							●			●				
14	●							●						●	●	●		●			18,265
15	●								●					●	●	●		●			18,266
16	●									●				●	●	●		●			18,267
17	●			●										●	●	●		●			18,268
11B	●			●										●			●				
18	●					●					●				●	●		●	●		16,698

TABLE 1 – Test Plan-Specimen Record (Continued)

Identification No. (1)	Welding(2,3)			Post Weld Anneal One Hour at Indicated Temperature							Aging Hrs. at 2100°F				Evaluation						
															Mechanical			Metallographic			
	GTA (Low Heat Input)	EB (Minimum Heat Input)	GTA, Slow Speed (High Heat Input)	As-Welded	2200°F	2400°F	2500°F	2600°F	2700°F	3000°F	0	100	1000	5000	Long. BDBTT	Trans. BDBTT	Long. Tensiles (2100°F)	Metallography (Optical)	Hardness Traverse	Phase Identification	Metallography ID Number
19	●						●				●				●	●					
20A	●						●				●						●				
21	●							●			●				●	●		●	●		16,699
22	●								●		●				●	●					
23	●							●				●			●	●		●	●		16,703
24	●																				
25B	●						●				●						●				
26		●		●										●	●	●		●			18,269
27		●					●							●	●	●		●			18,270
28		●						●						●	●	●		●			18,271
29		●							●					●	●	●		●			18,272
30		●					●						●		●	●					
31		●							●				●		●	●					
32		●						●					●		●	●					
33		●																			
34		●																			
35			●				●				●				●	●					
36			●					●			●				●	●		●	●		16,708
37			●						●		●				●	●					
38			●			●							●		●	●		●	●		16,707
39A			●			●							●				●				
39B			●				●					●					●				
40			●				●					●			●	●					

TABLE 1 - Test Plan-Specimen Record (Continued)

Identification No. (1)	Welding(2,3)			Post Weld Anneal One Hour at Indicated Temperature							Aging Hrs. at 2100°F				Evaluation						
															Mechanical			Metallographic			
	GTA (Low Heat Input)	EB (Minimum Heat Input)	GTA, Slow Speed (High Heat Input)	As-Welded	2200°F	2400°F	2500°F	2600°F	2700°F	3000°F	0	100	1000	5000	Long. BDBTT	Trans. BDBTT	Long. Tensiles (2100°F)	Metallography (Optical)	Hardness Traverse	Phase Identification	Metallography ID Number
41			●	●									●		●	●		●	●		16,709
42A			●					●					●				●				
42B			●						●			●			●	●	●				
43			●					●					●		●	●		●	●		16,706
44			●	●							●				●	●					
45			●																		
46			●																		
47	●			●									●		●	●					
48	●			●										●	●	●					
49	●						●						●		●	●					
50	●							●					●		●	●					
51		●			●									●	●	●		●			18,273
52		●				●							●		●	●					
53		●						●						●	●	●		●			18,274
54			●					●					●		●	●					
55			●						●				●		●	●					
56			●							●			●		●	●					
57A	●							●					●				●				
58			●			●							●		●	●					
59-2	●			●								●			●	●					
60	●			●								●			●	●					

TABLE 1 - Test Plan-Specimen Record (Continued)

NOTES:

(1) Welds 1 thru 46 from Program Heat Task V.
Welds 47 thru 60 from Reference Heat Task III.
Heat data per Table 2.

(2) Welding parameters as follows:

GTA, Low Heat Input: 9730 joules/inch using 15 ipm weld speed, 135 amperes DCSP, 3/8 inch clamp spacing.

GTA, High Heat Input: 18,900 joules/inch using 6 ipm weld speed, 105 amperes DCSP, 3/8 inch clamp spacing.

EB, Minimum Heat Input: 4190 joules/inch using 150 KV x 3.8 ma beam, 15 ipm welding speed.

GTA amperage lowered to 115 and 95 for Reference Heat.

GTA voltage at 0.060 arc gap approximately 18 volts in helium.

(3) All welds bead-on-plate fusion welds no filler metal added. GTA weld parameters selected for 0.180 inch weld width. EB weld width approximately 0.040 inch. Welding direction parallel to rolling direction.

TABLE 2 - T-111 Test Heat Data

Designation	Program Heat (New For This Task)	Reference Heat (Used For Task III Aging Study)
Supplier	Wah Chang	Wah Chang
Heat Number	65080	G-65042-Ta
Thickness	0.040 inch sheet	0.035 inch sheet
Condition	Recrystallized 1 Hr. at 3000°F	Recrystallized 4 Hrs. at 2400°F
Grain Size, ASTM	8	7
Hardness, DPH	224	221
Chemistry		
W (w/o)	8.83	8.8
Hf (w/o)	1.98	2.0
C ppm Certified/Check	80/37	80/48
O ppm Certified/Check	60/10	50/15
N ppm Certified/Check	10/5	35/18
Ductility 1t Bend Test at -320°F Longitudinal & Transverse	Bent	Bent

features of the welding and aging procedures are:

- GTA welding in monitored helium atmospheres containing less than 5 ppm oxygen and less than 5 ppm moisture.
- EB welding in a chamber evacuated into the 10^{-6} torr range.
- Aging in completely sealed sputter-ion pumped vacuum furnaces at less than 10^{-8} torr total pressure.
- No detectable weld contamination following processing and testing.

III. RESULTS

BEND DUCTILITY RESPONSES

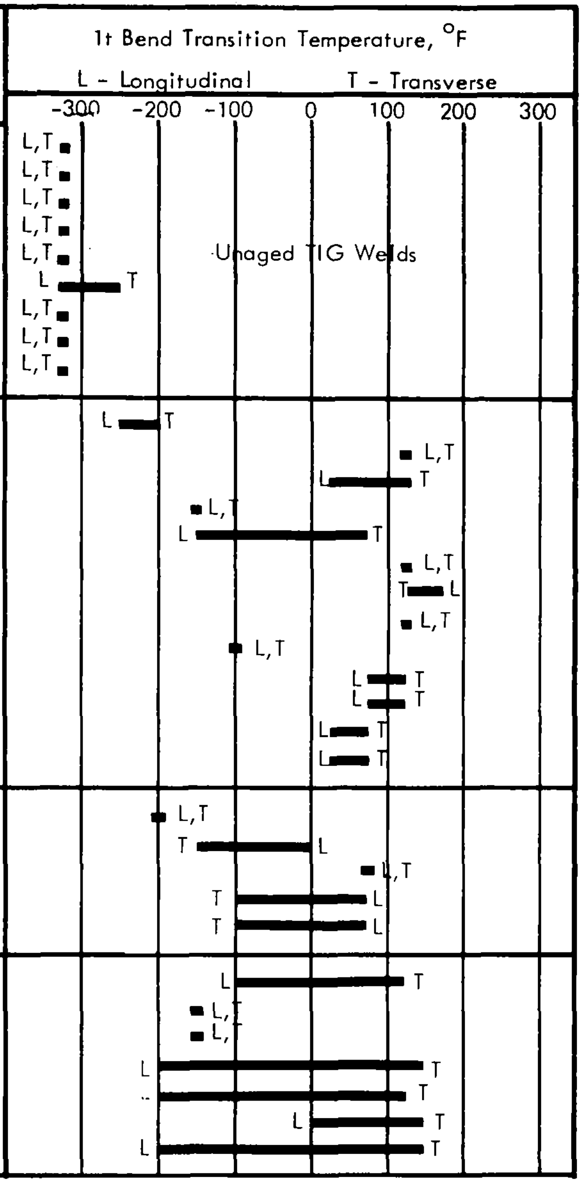
Bend testing was emphasized in the following aging and annealing responses in this program. Bend test results are presented in three forms in this report as follows:

- Detailed bend test data showing the results of each individual bend test. These are compiled in the appendix to this report.
- A tabulated summary showing the spread between longitudinal and transverse tests for each thermal history. This presentation is shown in Tables 3 and 4. The Go-No Go temperature for the 1t bend is conventionally labeled "Bend Ductile Brittle Transition Temperature" in these summaries. As previously explained, however, this is a misnomer in the case of T-111 since fractures occur by ductile tearing rather than brittle cleavage
- Finally, a comparative summary from which the effects of welding parameters, heat to heat variability and effect of post weld annealing temperatures can be ascertained. This is given in Figures 5 and 6. The longitudinal and transverse bend test transition temperatures are averaged for presentation in these figures. The technique of averaging these temperatures was established in Task I of this program in which it was verified that this provided the most rational presentation of weld bend test data.

These results lend themselves to a very simple interpretation, namely, that post weld annealing had little effect in influencing the bend ductility response to aging of either heat of T-111. Only a modest heat to heat variability was observed with no basic general difference in trends. The 3000°F post weld anneal for GTA welds produced a singular deviation at 1000 hours aging which did not carry over into the 5000 hour age. EB welds of the program heat appear to be more sensitive to aging than in the reference heat. It is not certain what caused this. EB welds in the program heat had somewhat rougher surface texture which may have resulted in moderately higher transitions. In an engineering sense the post weld anneals were not however beneficial and did not stabilize the weld structures.

TABLE 3 - Aging Responses of Welds in 0.040 Inch T-111 Sheet (Program Heat)

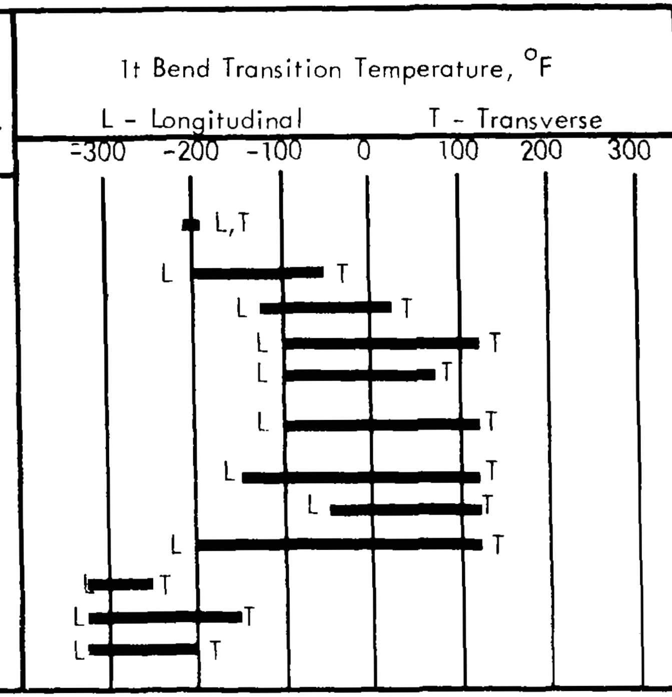
Material Sheet Tk. (in.)	Weld Type*	1 Hour Anneal (°F)	Hours Age at 2100°F	Weld Spec. No.
0.040	1	--	--	1
"	1	2400	--	18
"	1	2500	--	19
"	1	2600	--	21
"	1	2700	--	22
"	2	--	--	44
"	2	2500	--	35
"	2	2600	--	36
"	2	2700	--	37
0.040	1	--	100	3
"	1	--	1000	4
"	1	--	5000	17
"	1	2600	100	23
"	1	2400	1000	6
"	1	2500	1000	7
"	1	2600	1000	9
"	1	2700	1000	10
"	1	3000	1000	12
"	1	2500	5000	13
"	1	2600	5000	14
"	1	2700	5000	15
"	1	3000	5000	16
0.040	2	2500	100	40
"	2	2700	100	42
"	2	2600	1000	43
"	2	2400	1000	38
"	2	---	1000	41
0.040	3	2500	1000	30
"	3	2600	1000	32
"	3	2700	1000	31
"	3	--	5000	26
"	3	2500	5000	27
"	3	2600	5000	28
"	3	2700	5000	29



* (1) Low heat input GTA weld, 9730 Joules/in
(2) High heat input GTA weld, 18,900 Joules/in
(3) Electron beam weld, 4190 Joules/in

TABLE 4 - Aging Responses of Welds in 0.035 Inch T-111 Sheet (Reference Heat)

Material Sheet Tk. (in.)	Weld Type*	1 Hour Anneal (°F)	Hours Age at 2100°F	Weld Spec. No.	1t Bend Transition Temperature, °F
0.035	1	--	100	59-2 60-1	
"	1	--	1000	47	
"	1	--	5000	48	
"	1	2500	1000	49	
"	1	2600	1000	50	
"	2	2400	1000	58-1 5' -2	
"	2	2600	1000	54	
"	2	2700	1000	55	
"	2	3000	1000	56	
"	3	2400	1000	52	
"	3	2200	5000	51	
"	3	2600	5000	53	



* (1) Low heat input GTA weld, 9730 Joules/in
(2) High heat input GTA weld, 18,900 Joules/in
(3) Electron beam weld, 4190 Joules/in

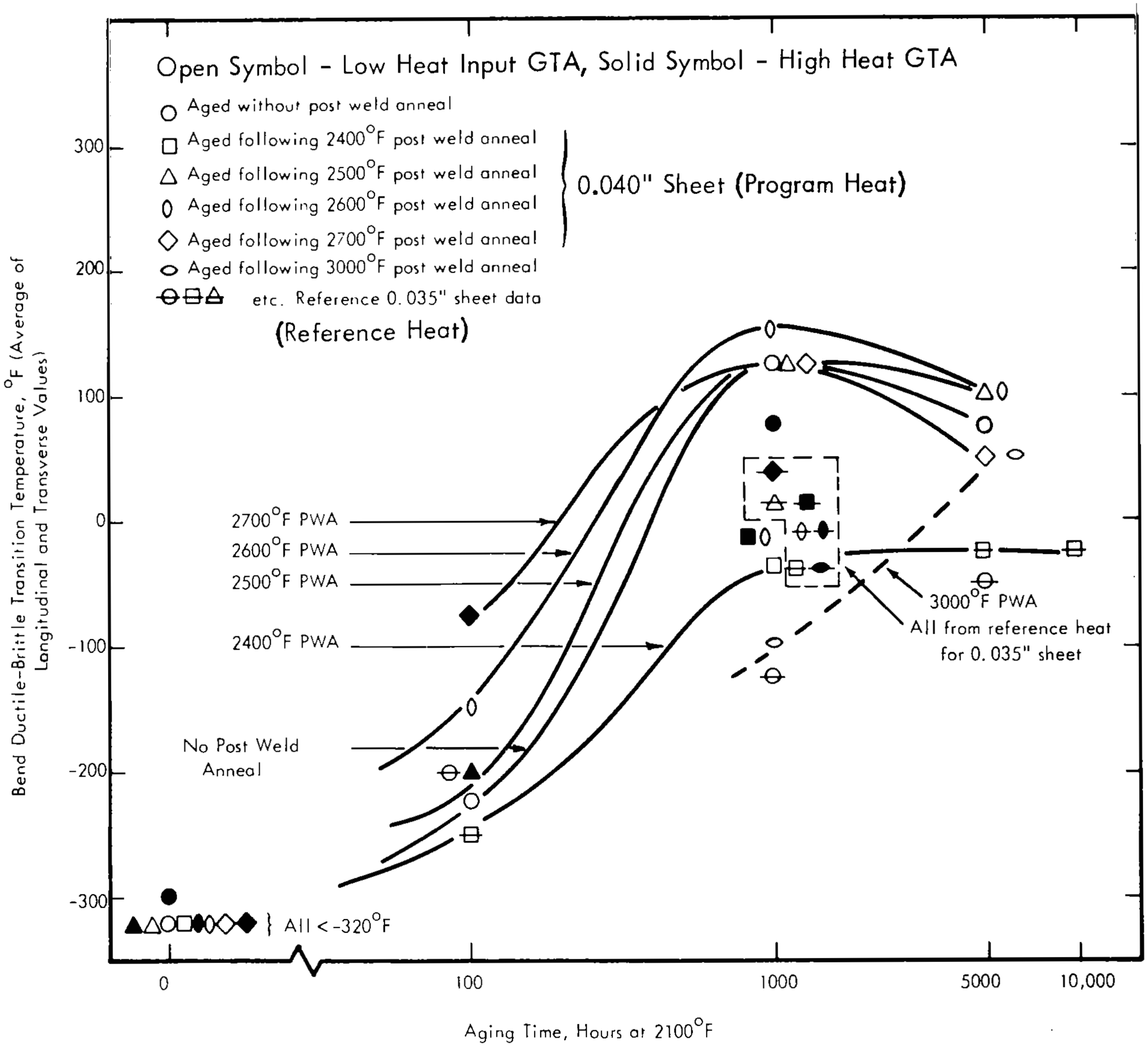


FIGURE 5 - Effect of Post Weld Annealing on Aging of T-111 GTA Welds

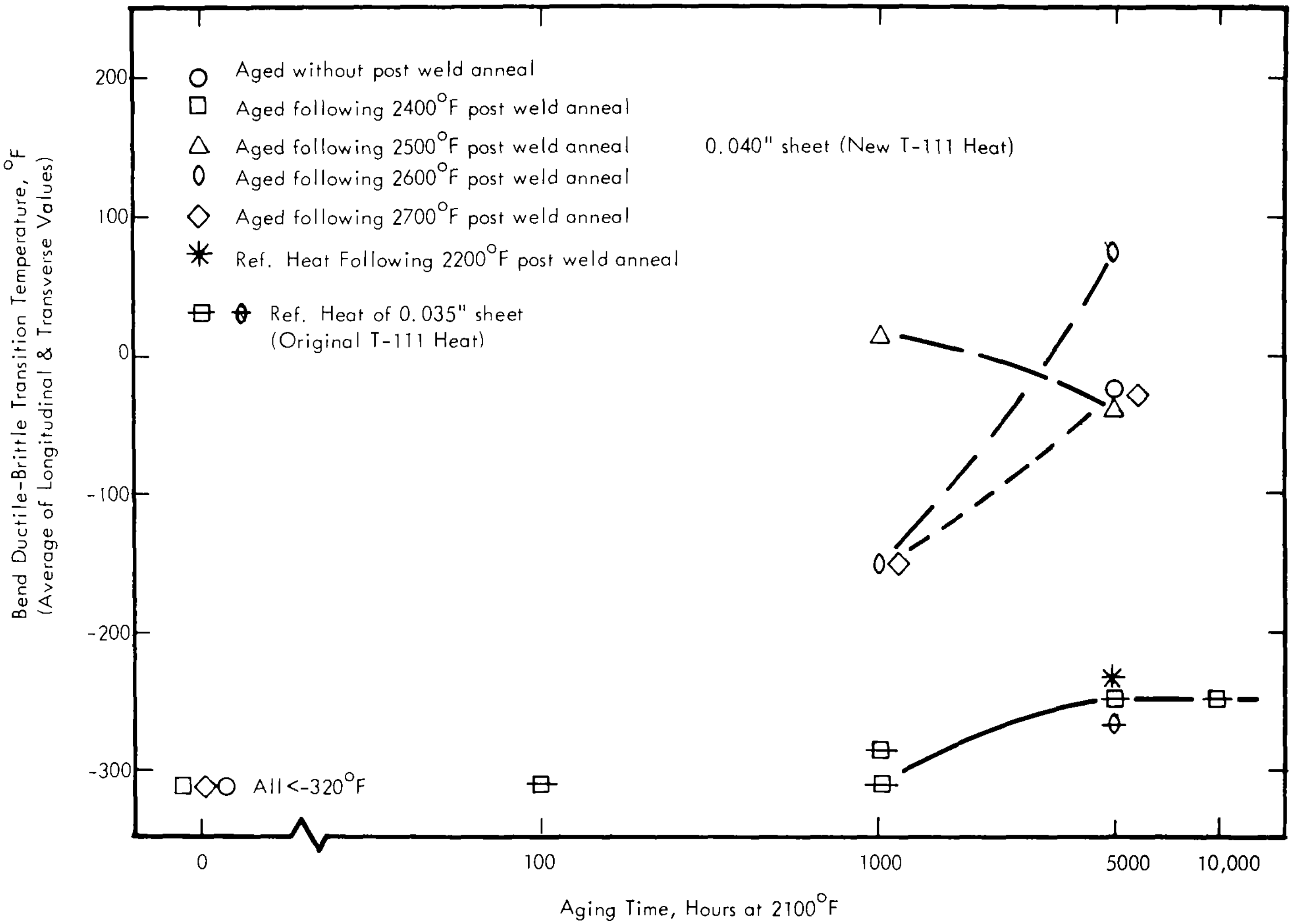


FIGURE 6 - Effect of Post Weld Annealing on Aging of T-111 EB Welds

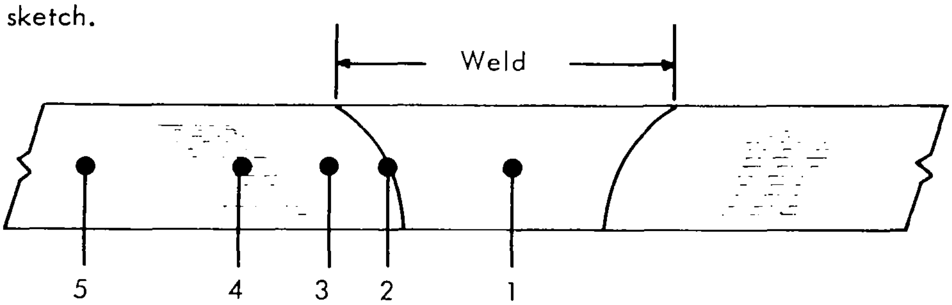
Generally the transverse bend tests gave the higher bend transition temperatures and fractures tended to follow the weld centerline. The centerline fractures were ascribed to the combined failure mode (grain boundary tearing) coupled with the greater structural mismatch typical along weld centerlines. This centerline weakness is most noticeable in transverse testing as indicated by the higher transverse bend transition temperatures.

TENSILE RESPONSES

Results of longitudinal GTA weld tensile tests at 0°C (32°F) demonstrated that in a conventional engineering sense neither weld ductility nor strength were impaired by aging. This is shown in Figure 7 for ultimate strength and yield strength for aging up to 5000 hours. Figure 8 shows the effect of aging on total and uniform elongation and a reduction in area. Reduction in area was modestly lower following 5000 hour aging while elongation remained unchanged. Total elongation was approximately 25% for all combinations of annealing and aging. Interestingly, this is less than the 33-1/3% outer fiber strain produced in a 1t bend. This supports the Go-No Go interpretation applied to T-111 bend test results in this study.

STRUCTURAL RESPONSES

The typical microstructure of as-welded T-111 is shown in Figures 9 through 13. These are shown as general reference for all zones of the weld from base metal to weld center. Five general areas fully characterize a typical weld in T-111. These are indicated in the following sketch.



1. Weld Metal, Weld Center
2. Weld Edge
3. Heat Affected Zone
4. Edge of Heat Affected Zone
5. Base Metal

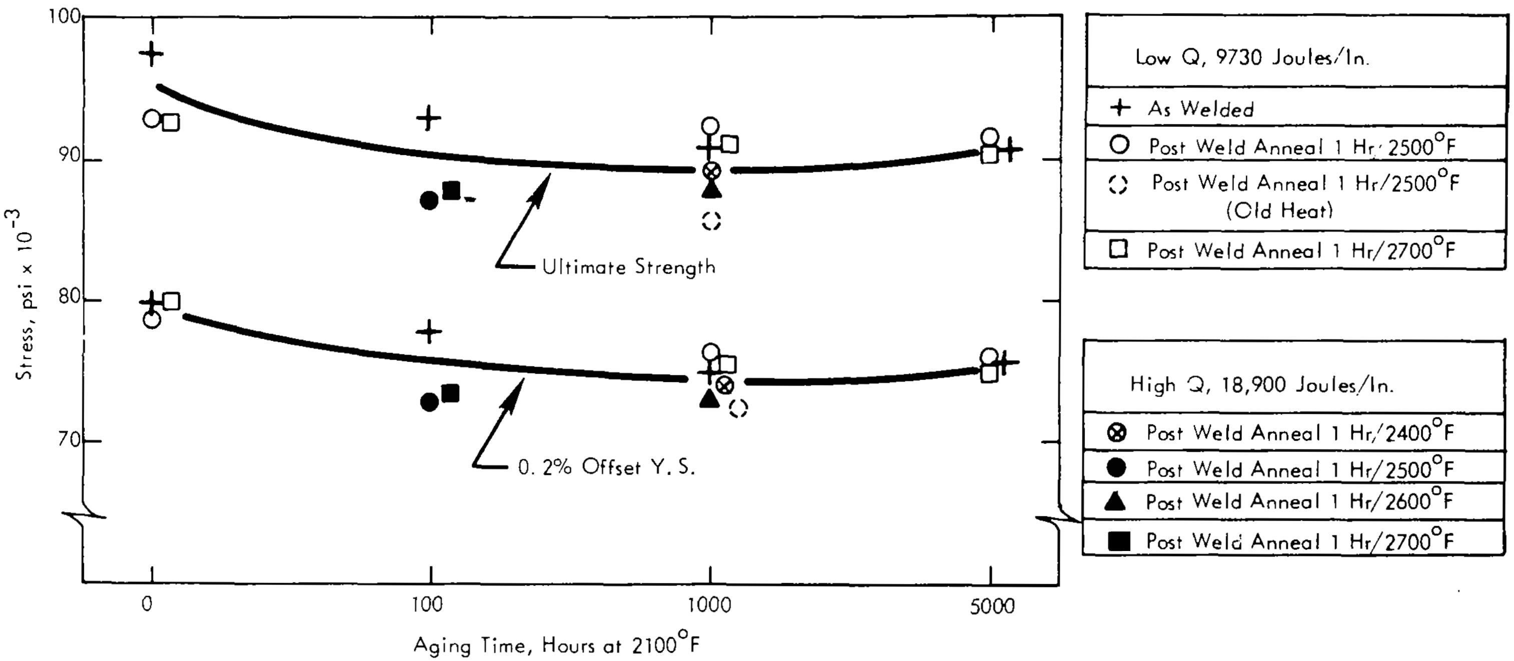


FIGURE 7 - Effect of Aging on T-111 Weld Strength as Measured by Longitudinal Tensile Tests of GTA Welds at 0°C (32°F)

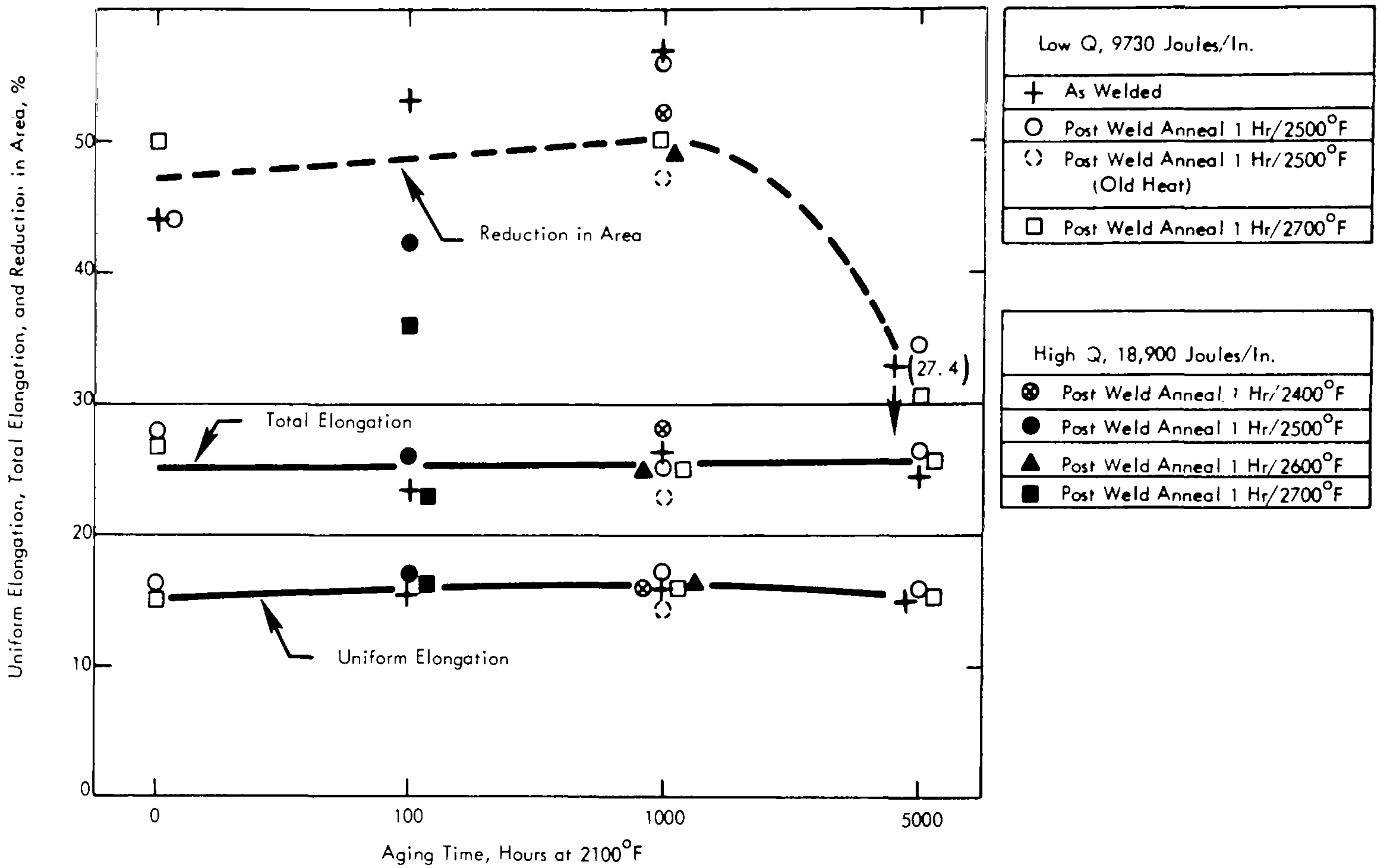


FIGURE 8 - Effect of Aging on T-111 Weld Ductility as Measured by Longitudinal Tensile Tests of GTA Welds at 0°C (32°F)

Zone 4 displays a response to welding which is somewhat unique, Figure 12. A precipitation occurs along what appear to be flow lines observed as a ghost structure in T-111. This ghost structure is visible in the 200X base metal photomicrograph, Figure 13. No explanation has been developed for this ghost structure although it is believed to be flow lines resulting from incomplete homogenization. This structure is usually observed in Ta based alloys and frequently in Cb based alloys. The precipitation appears then to be a relatively low temperature response associated with constitutional segregation. This is an interesting observation particularly since T-111 aging occurs at relatively low temperatures and, as will be shown shortly, the aging is accomplished by weld and HAZ precipitation in patterns controlled by constitutional segregation. The weld zone is single phase as-welded, Figure 9. This is likewise the case at the weld interface for weld and HAZ, Figure 10, and in the HAZ proper, Figure 11, and in the base metal, Figure 13. A very modest amount of precipitate is observed along the ghost structure in the base metal. This appears to be the precipitate which develops more fully during welding at the edge of the HAZ as shown in Figure 13.

The general response of T-111 weld structure to aging as observed in Task III is shown in Figure 14. GTA weld structures are shown since these represent welds of maximum response to aging. Although structural thermal responses are shown irrespective of where they occur in the weldment, it is important to recall that bend test failures always occurred by ductile tearing along weld metal grain boundaries and that no failures occurred in either the HAZ or base metal.

The 1 hour-2400°F post weld anneal, Figure 14a, has resulted in a weld zone relatively precipitate-free much like the as-welded structure. In the HAZ the only evidence of precipitates is along the grain boundaries. The bend DBTT of this structure was below -320°F.

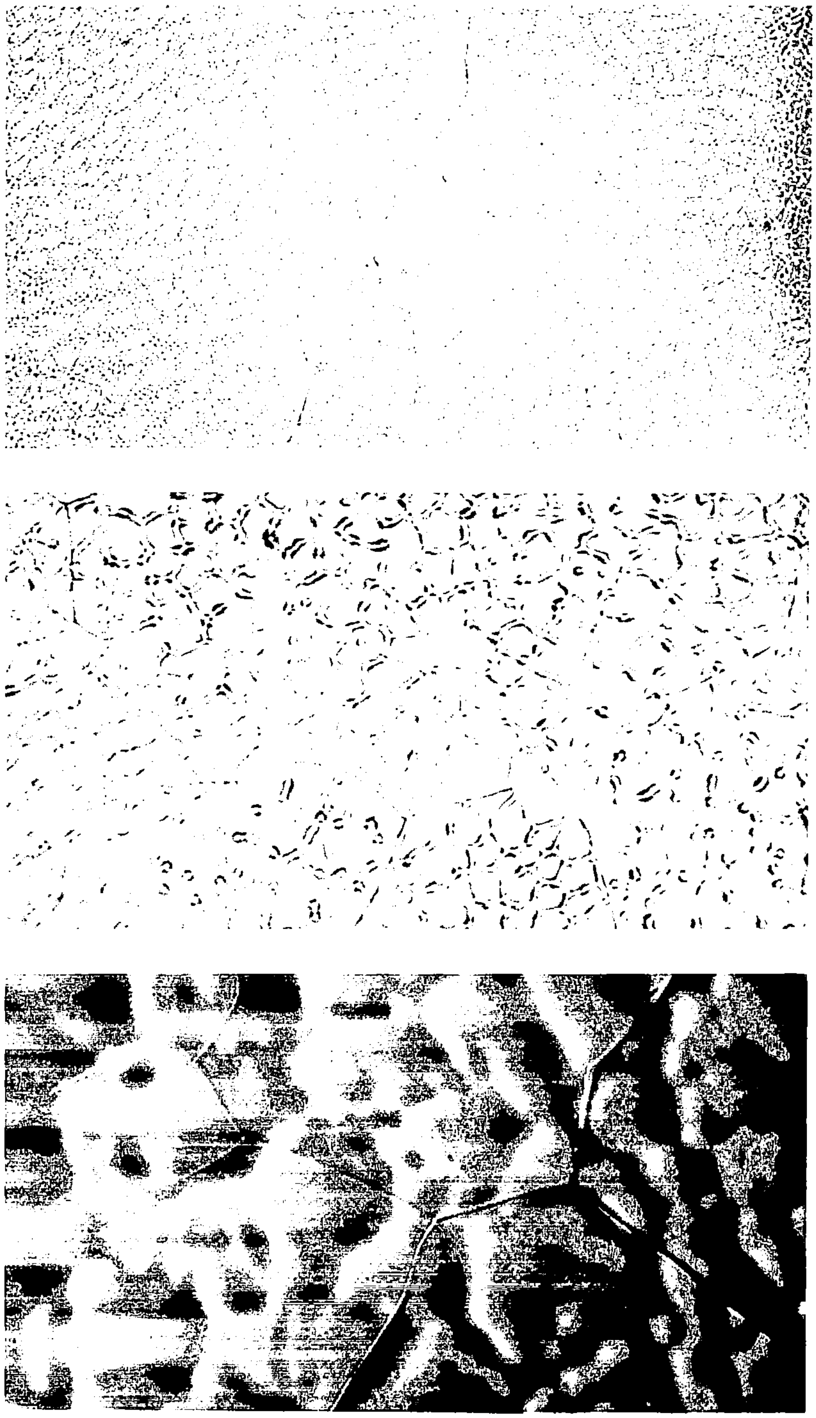


FIGURE 9 - Microstructure of As-Welded T-111 at Center of GTA Weld. Top, 200X. Middle, 500X. Bottom 1500X. Met. 16,697.

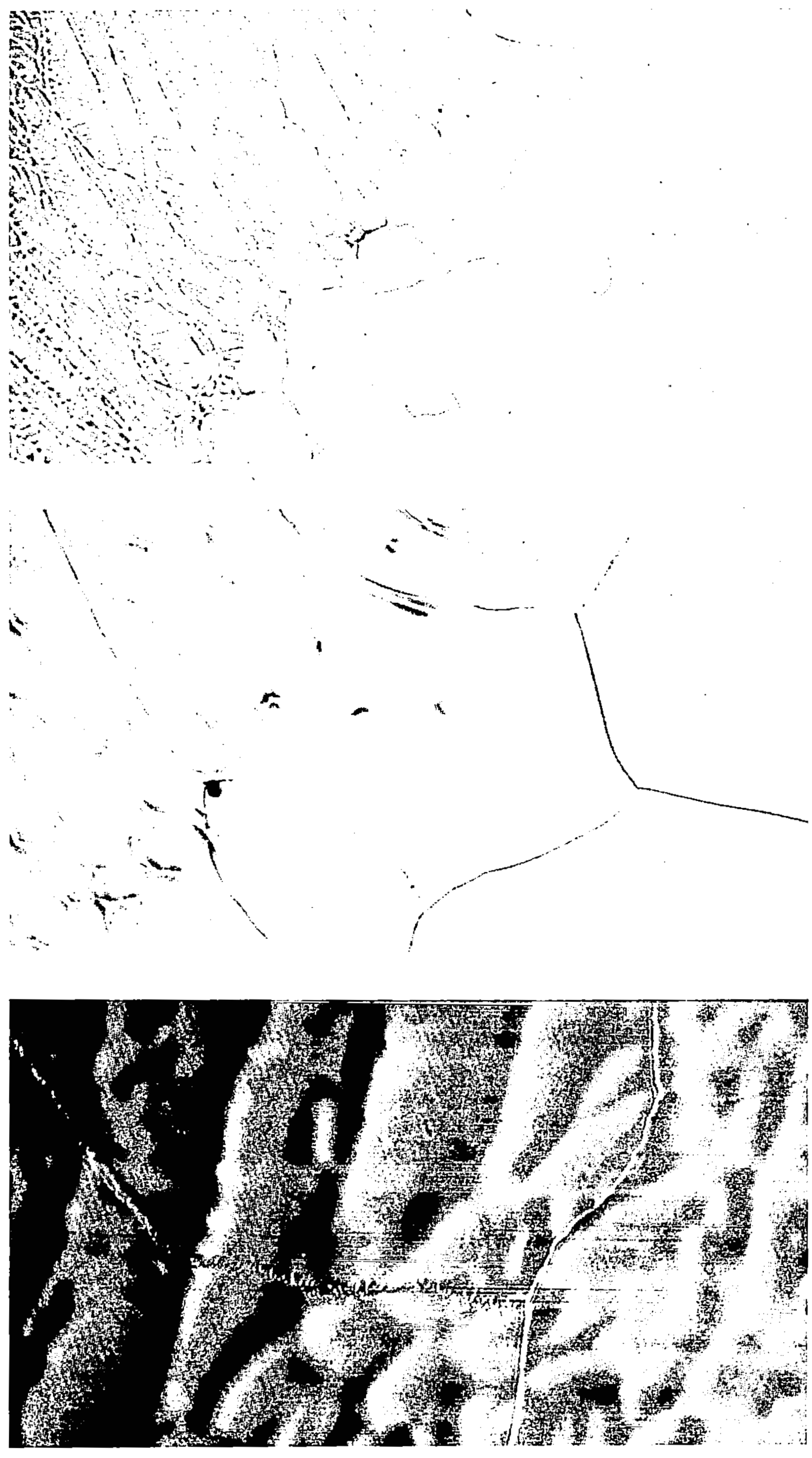


FIGURE 10 - Microstructure of As-Welded T-111 at Weld Edge. Top, 200X. Middle, 500X. Bottom, 1500X. Met. 16,697.

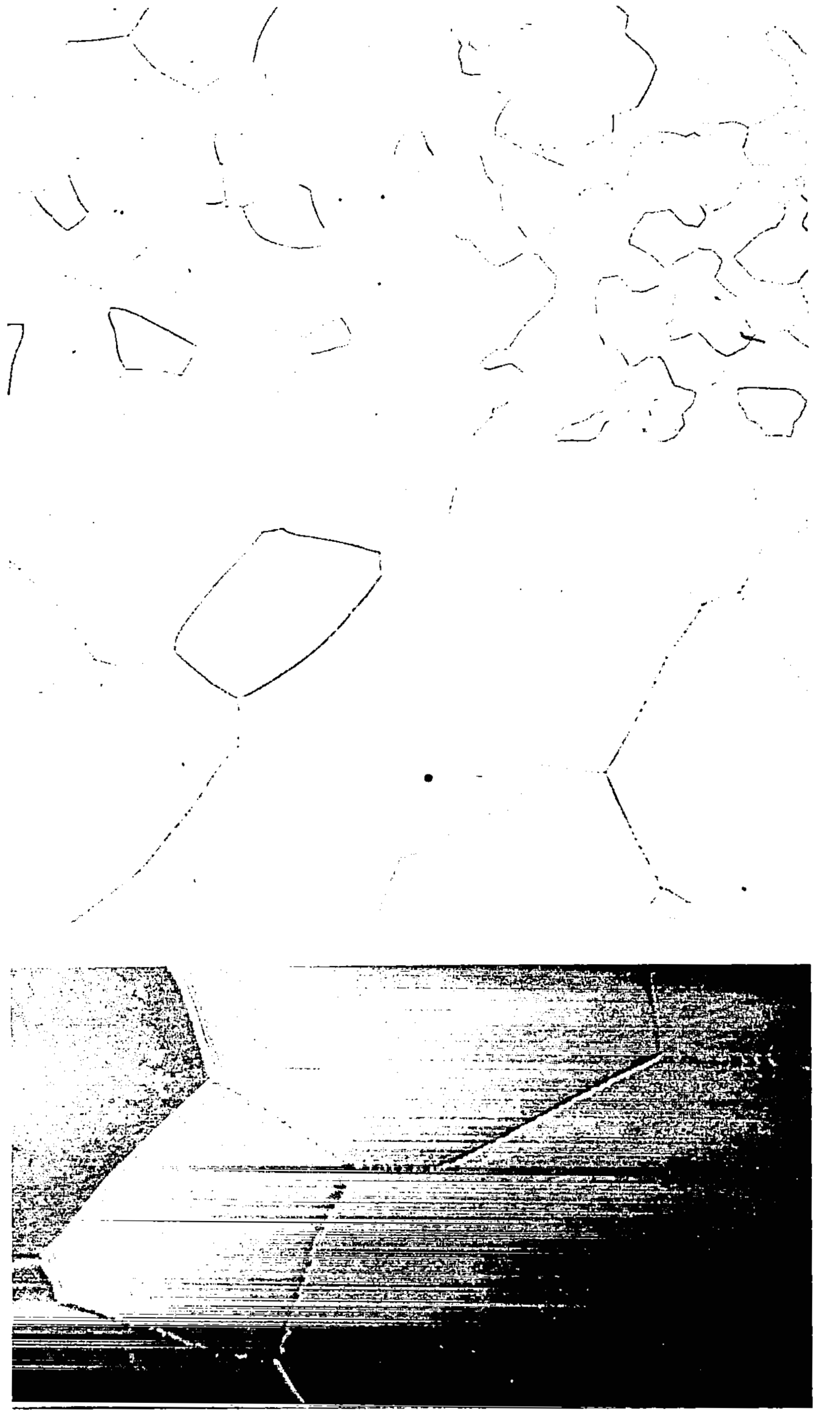


FIGURE 11 - Microstructure of As-Welded T-111 in the Heat Affected Zone.
Top, 200X. Middle, 500X. Bottom, 1500X. Met. 16,697.

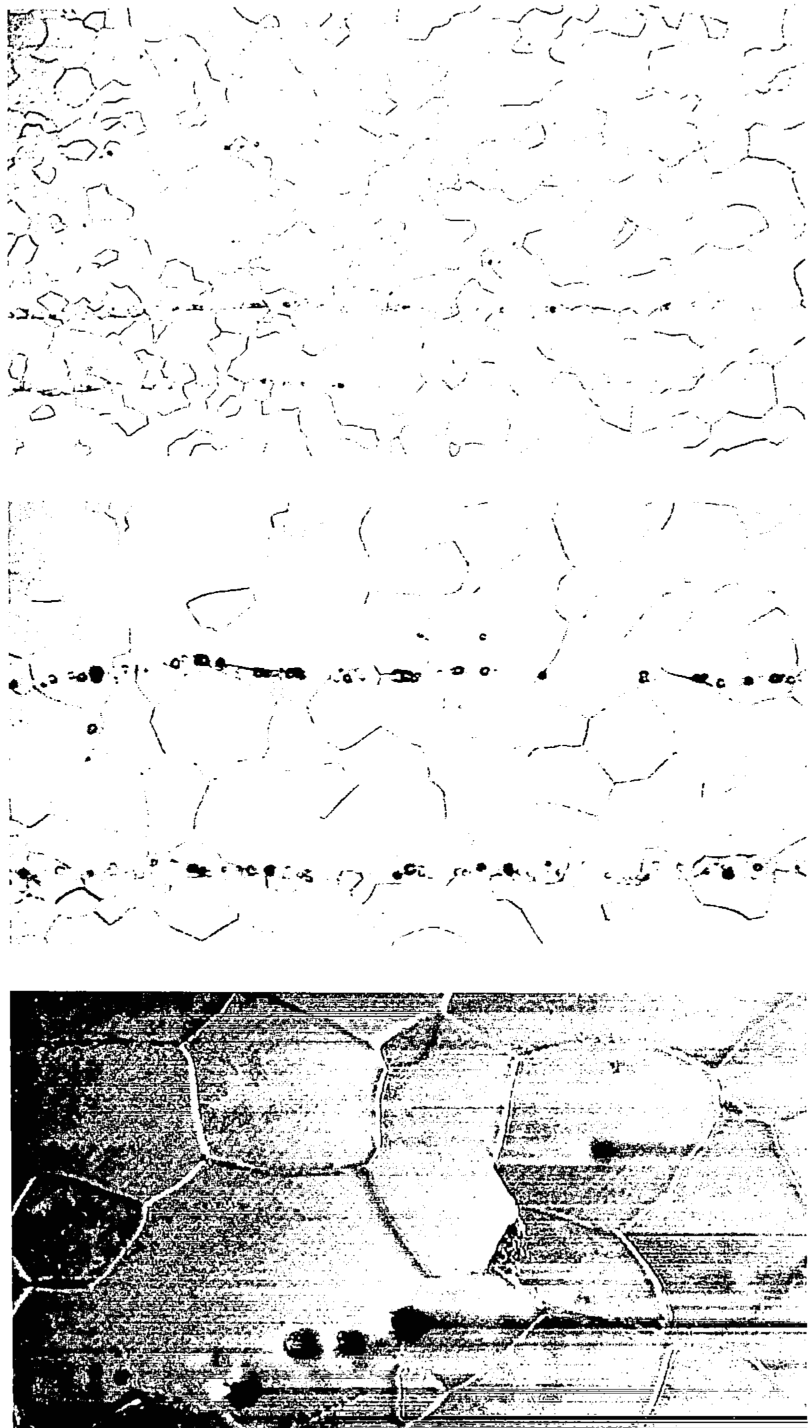


FIGURE 12 - Microstructure of As-Welded T-111 at the Edge of the HAZ. Top, 200X. Middle, 500X. Bottom, 1500X. Met. 16,697.

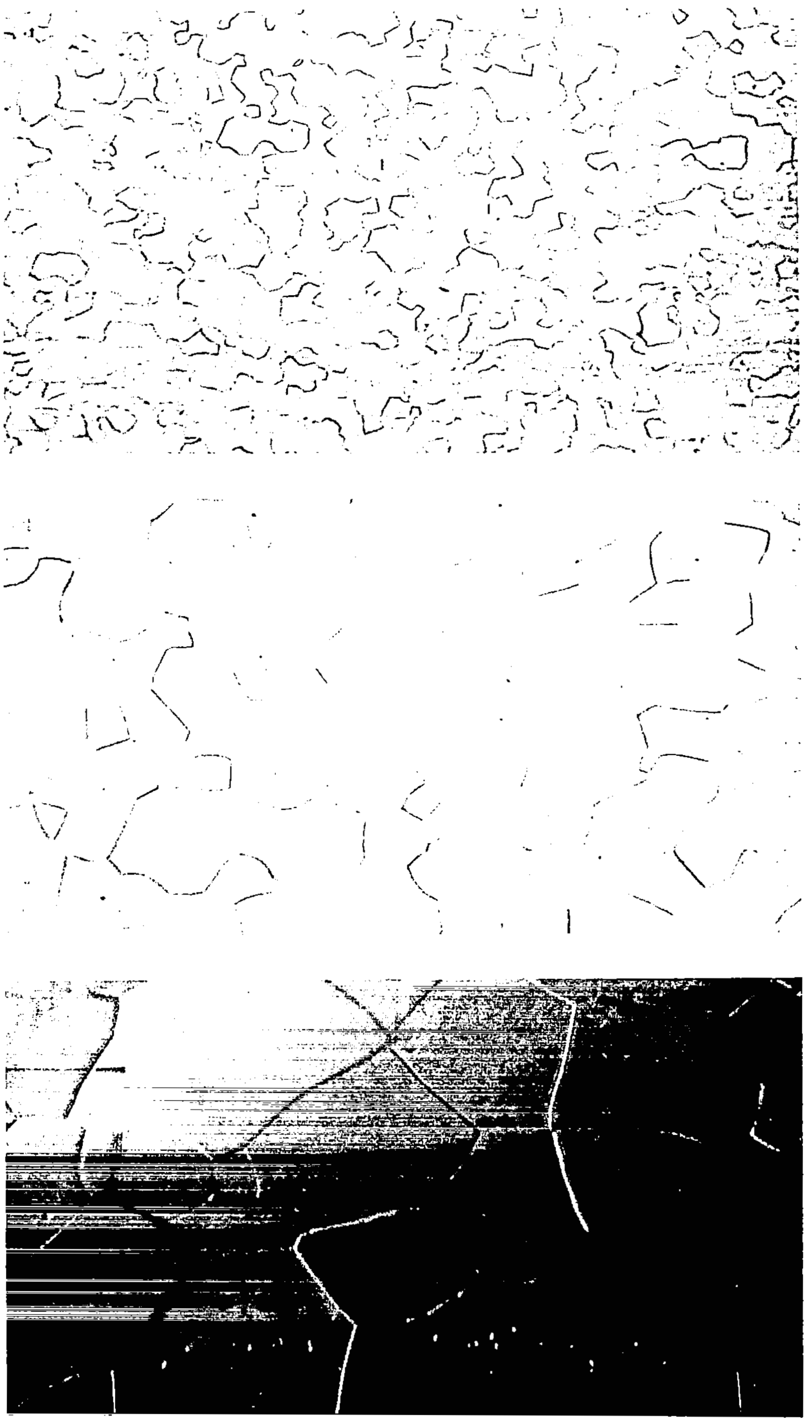
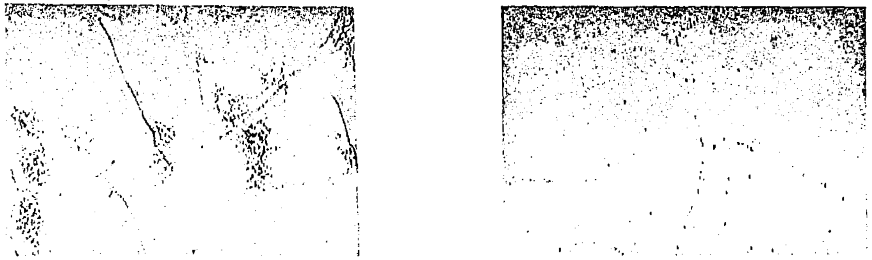
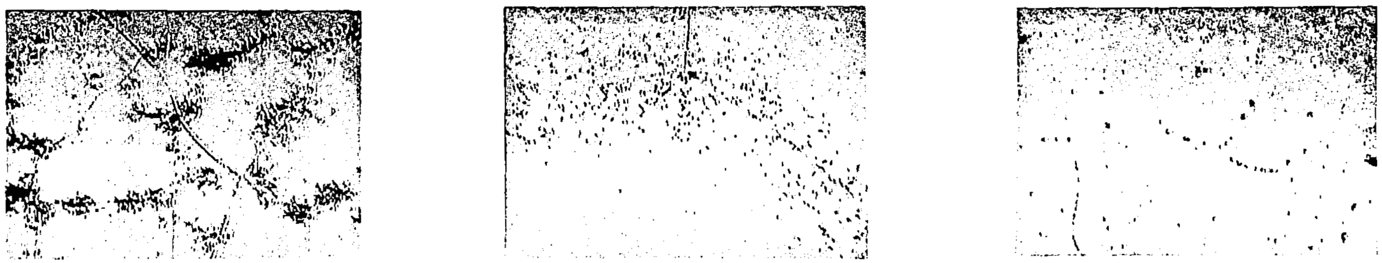


FIGURE 13 - Microstructure of T-111 Base Metal, As-Received. Top, 200X. Middle, 500X. Bottom 1500X. Met. 16,697.

WELD ZONE | HAZ | BASE METAL



(a) As-Welded + 1 Hour - 2400°F Post Weld Anneal Bend DBTT < -320°F



(b) Welded, PWA 1 Hr./2400°F. Aged 10,000 Hours/1800°F Bend DBTT +125°F

(c) Welded, PWA 1 Hr./2400°F. Aged 5000 Hours/2100°F Bend DBTT -25°F

FIGURE 14 - Microstructures of Aged T-111 GTA Welds

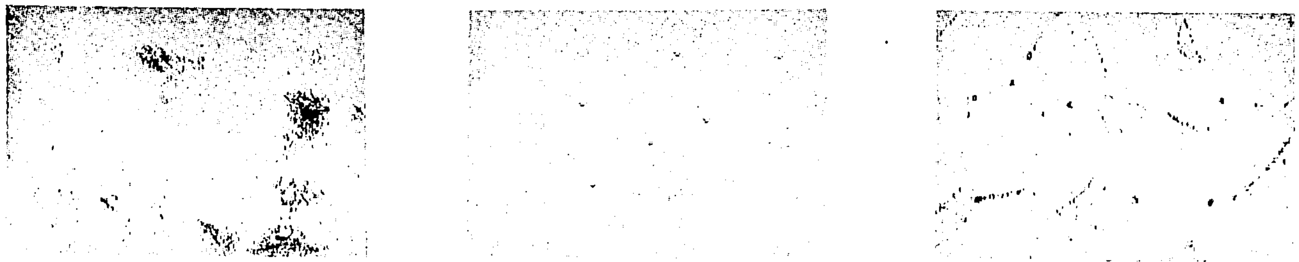
After aging for 10,000 hours at 1800°F the microstructure is altered from that of Figure 14a to that shown in Figure 14b. Weld zone precipitates can be observed in the aged structure, mainly along the interdendritic boundaries of the original cored weld structure. This suggests that solute enrichment associated with the cored structure provides the driving force for this precipitation. There is, in addition, a nearly continuous precipitate phase located at the grain boundaries of the fusion zone. Fine, dispersed precipitates are apparent in the HAZ while the base metal appears to be quite clean except for the presence of coarse precipitates along the grain boundaries. The bend DBTT of this specimen was +125°F.

The effect of aging T-111 at 2100°F for 5000 hours can be seen in Figure 14c. The amount of interdendritic precipitate is less while the amount of grain boundary precipitate in the fusion zone is about the same. Little difference is noted in the HAZ except that possibly a little more precipitation is present. The base metal, shown at 500X, is similar to that seen in Figure 14a. The bend DBTT is -25°F.

The DBTT remained below -320°F when aged at 2400°F. After 10,000 hours complete homogenization had occurred and, except for isolated, coarse precipitates at the grain boundaries, the weldment was single phase. Further, a single phase structure could essentially be restored in specimens aged 10,000 hours at 1800°F by post age annealing at 2400°F. This is shown in Figure 15. Comparison with Figure 14b indicates post age annealing effected a reduction in the amount of weld zone precipitates at both interdendritic and grain boundary areas and the nearly complete elimination of precipitates in the HAZ while having virtually no effect on the base metal. Hence, the aging reaction appears to be reversible. The post age annealed structure is nearly identical to the 1 hour-2400°F post weld annealed structure of Figure 14a.

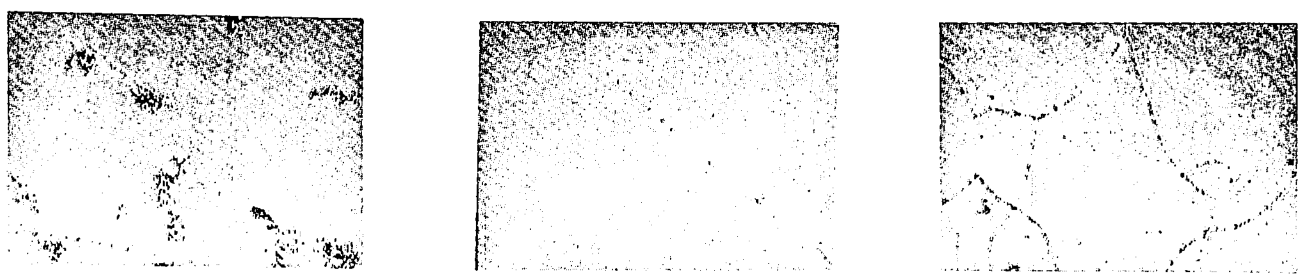
There appears to be a correlation between the bend transition temperature and the amount of interdendritic and grain boundary precipitates in the weld fusion zone. Since the transition temperature decreases with a decrease in the amount of precipitate it seems likely

WELD ZONE HAZ BASE METAL



20,500 1500X 20,500 1500X 20,500 1500X

(a) Welded, PWA 1 Hr./2400°F. Aged 10,000 Hrs./1800°F. Post-Age Annealed 1 Hr./2400°F



20,530 1500X 20,530 1500X 20,530 1500X

(b) Welded, PWA 1 Hr./2400°F. Aged 10,000 Hrs./1800°F. Post-Age Annealed 16 Hrs./2400°F

FIGURE 15 - Microstructure of T-111 GTA Welds Following Post Age Annealing

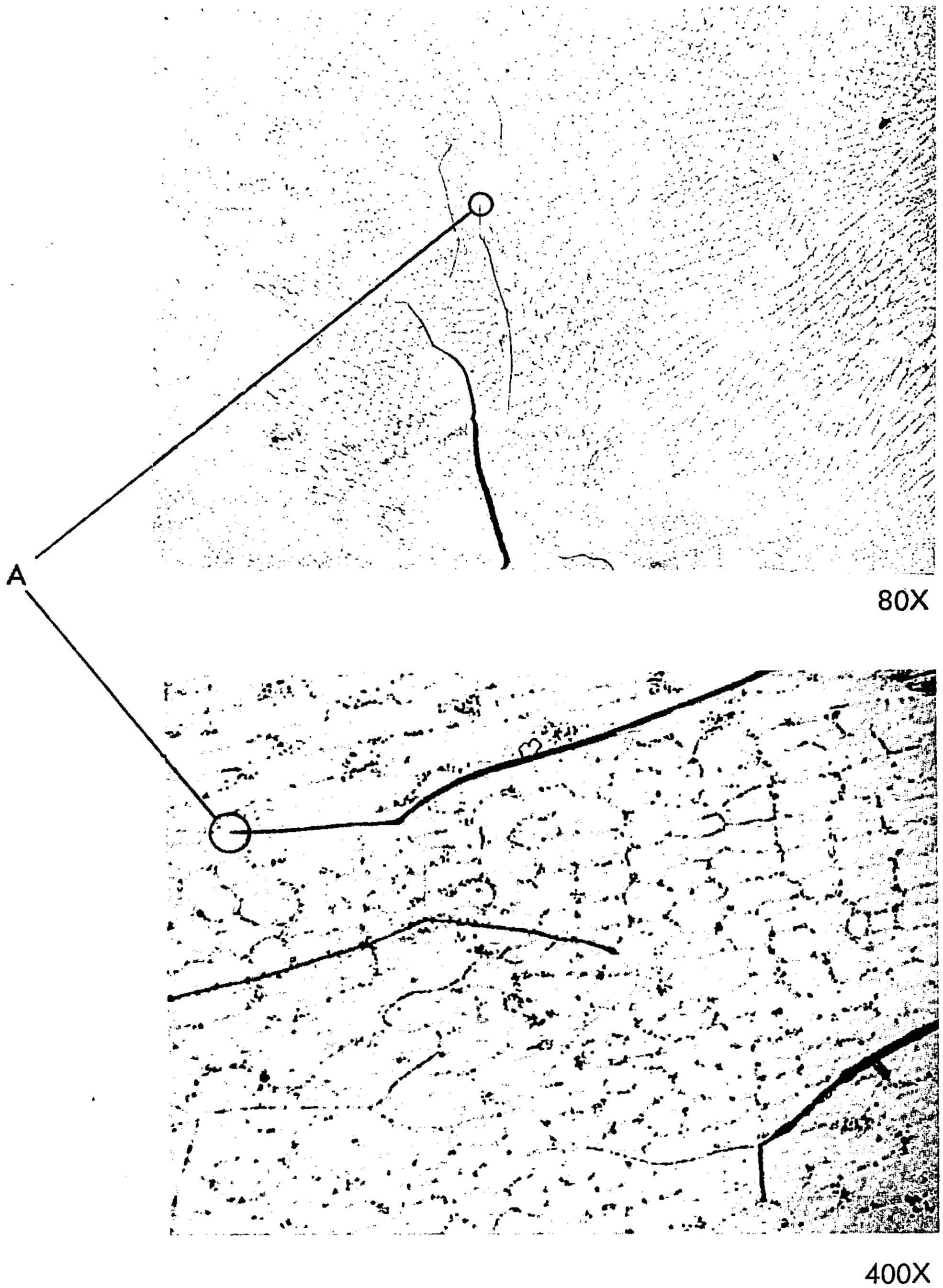


FIGURE 16 - Crack Termination in T-111 GTA Weld Bend Tested Following Post Weld Annealing (1 Hour at 3000°F) and Aging 5000 Hours at 2400°F Met. 18,267

that a cause-effect relationship exists between them. This relationship is discussed in detail in the Task III final and is not repeated here. However, the mechanism of the T-111 aging response as postulated in the Task III final is included in Section V of this report. Evidence of a relationship between bend test results and structural responses, particularly in the case of the grain boundary precipitates, is the fact that weld fractures during bend testing were invariably intergranular. A crack tip in an aged T-111 weld bend specimen is shown in Figure 16. Cracks have propagated typically along grain and interdendritic boundaries in this specimen.

In this evaluation, as in the aging of Task III, weld structure correlates with bend test results. Hence, just as the ductility of aged welds failed to respond to post weld annealing, structure also failed to respond to annealing prior to aging. Weld structure like weld bend ductility was not stabilized by post weld annealing. This effect is summarized in Figures 17 thru 21. Quite clearly a general correlation exists between the amount of weld precipitate and the bend-ductile-brittle-transition-temperature, and generally post weld annealing had little, if any, influence on the aging reaction. Even the 3000°F anneal had no effect although this is the standard recrystallization treatment for T-111. The structures summarized in Figures 17 thru 21 correspond almost exactly to the more detailed structures shown previously. The only exception is the continuous weld grain boundary precipitates in the weld structure aged 1000 hours at 2100°F , Figure 19. This was an isolated structure, even in this specimen. Hence, these grain boundaries may actually be separated rather than decorated by a precipitate.

Hardness traverses were also taken on aged specimens. Like tensile testing, these were not sensitive to the aging reaction. Hence, these are included only in the appendix to this report.

Further, in-depth investigations of the aging response in T-111 were reported for Task III. These included phase identification studies by chemical and mechanical extraction of precipitates as well as microprobe scanning to identify the extent of micro-and macro-segregation and its possible effect as the driving force of the aging reaction. Details of this

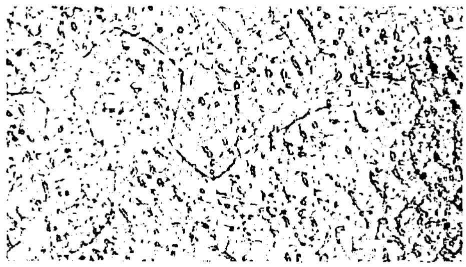
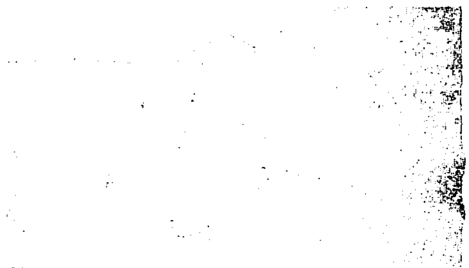
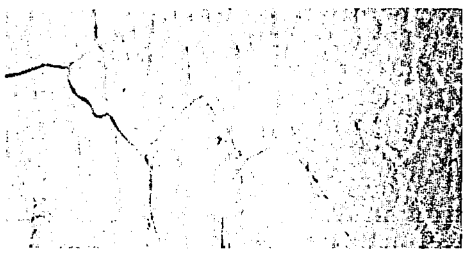
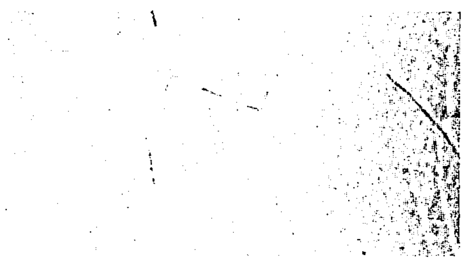
NO POST WELD ANNEAL		Weld 200X BDBTT <– 320°F (8679)
PWA 1 Hour - 2400°F	 	HAZ 400X (16,698) BDBTT <– 320°F Weld 400X (9519)
PWA 1 Hour - 2600°F	BDBTT <– 320°F (16,699 and 16,708)	
PWA 1 Hour - 3000°F		Weld 400X BDBTT <– 320°F (9521)

FIGURE 17 - Microstructure of Unaged T-111. Post Weld Annealed as Indicated.

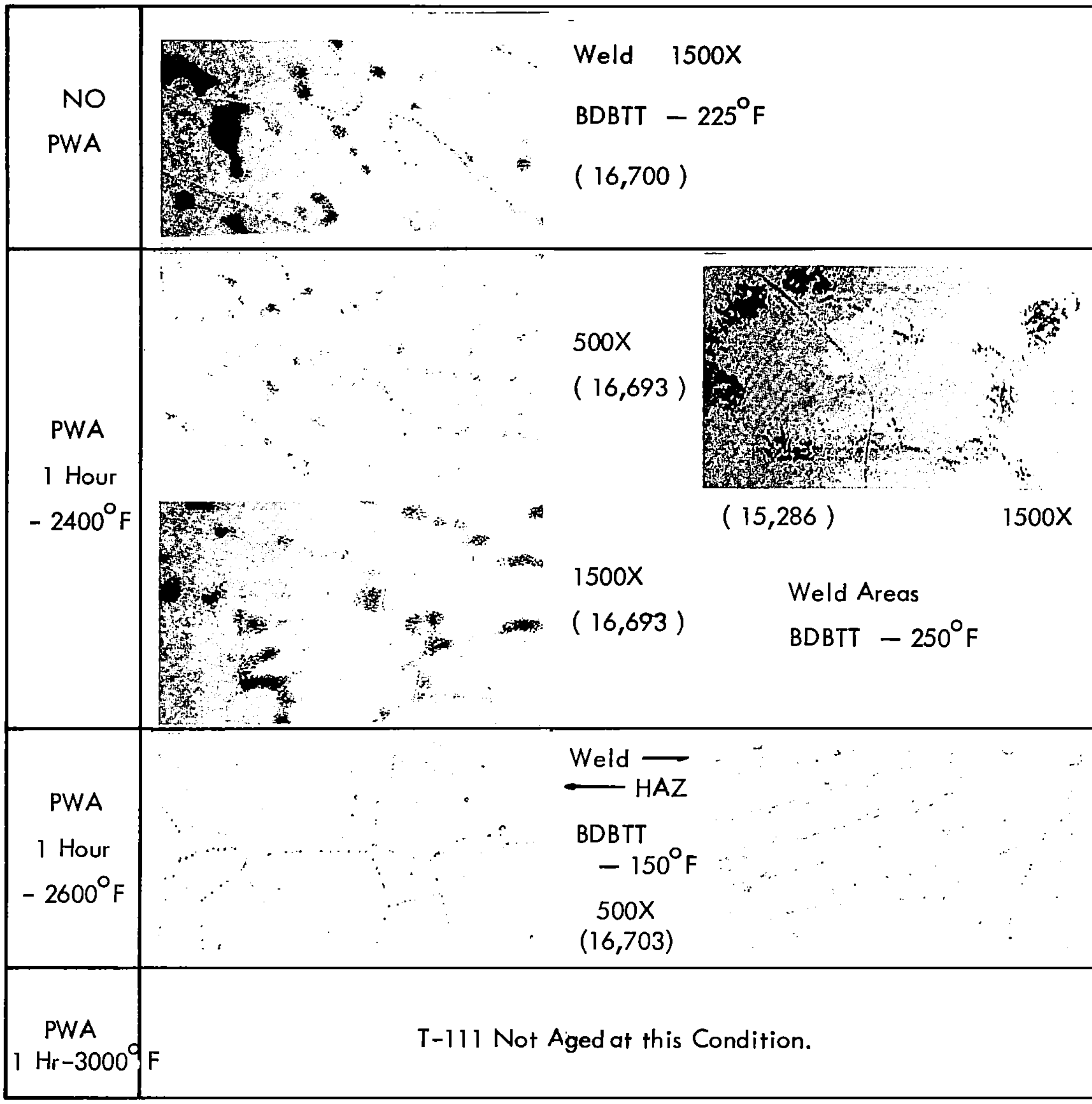


FIGURE 18 - Microstructure of T-111 Aged 100 Hours at 2100°F. Post Weld Annealed as Indicated.

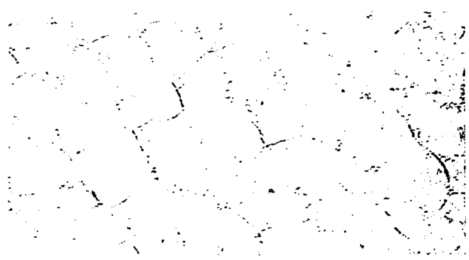
NO PWA	 Weld 500X (16,701) BDBTT + 125°F  HAZ 500X (16,706)
PWA 1 Hr. - 2400°F	BDBTT — 50°F to + 25°F (16,702 and 16,707)
PWA 1 Hr. - 2600°F	BDBTT + 150°F (16,704 and 16, 709)
PWA 1 Hr. - 3000°F	 Weld 500X BDBTT — 100°F (16,705)

FIGURE 19 - Microstructure of T-111 Aged 1000 Hours at 2100°F. Post Weld Annealed as Indicated.

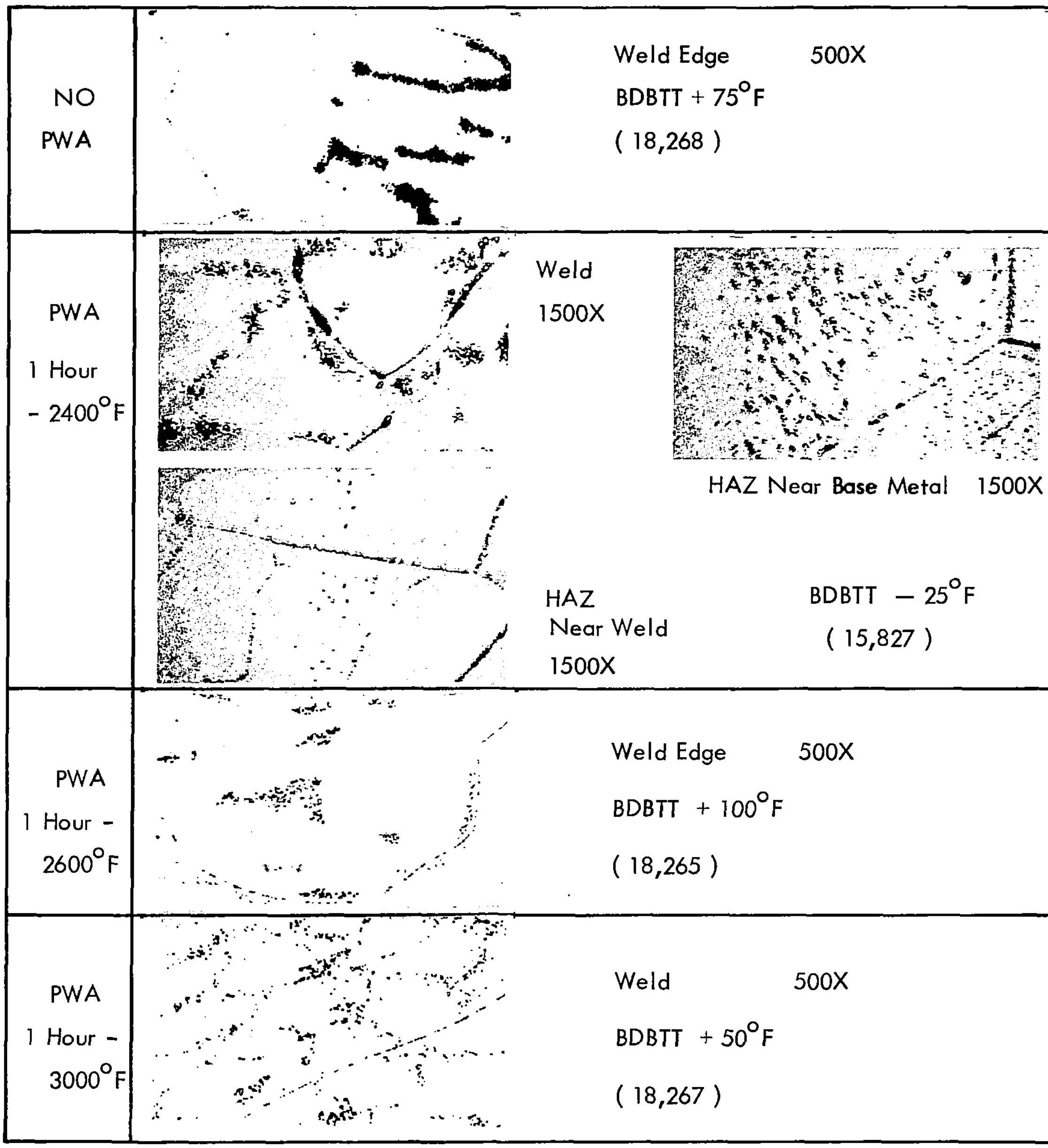


FIGURE 20 - Microstructure of T-111 Aged 5000 Hours at 2100°F. Post Weld Annealed as Indicated.

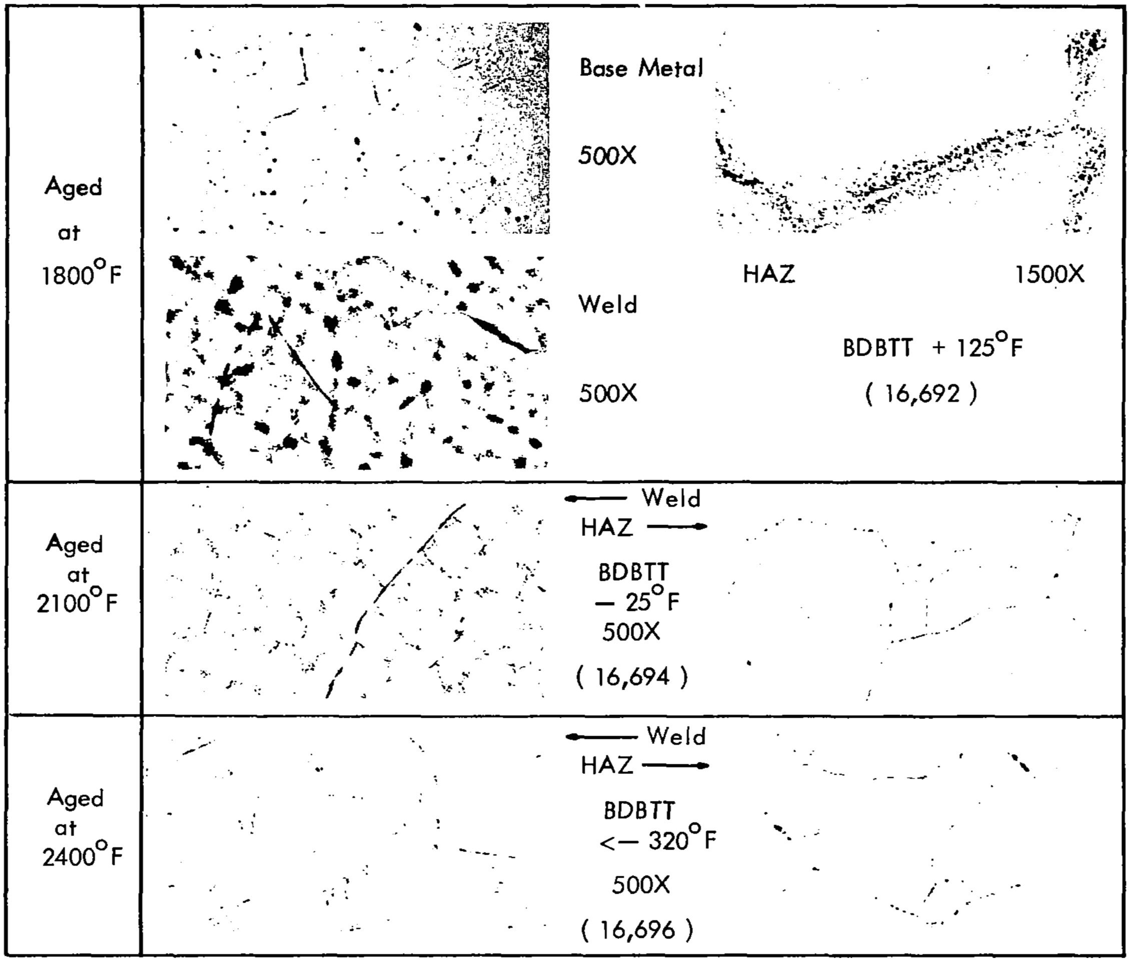


FIGURE 21 – Microstructures of T-111 Aged 10,000 Hours at Indicated Temperatures. All Specimens Post Weld Annealed 1 Hour-2400°F Prior to Aging.

effort are not repeated in this report, and, because of the similarity of responses following post weld annealing no further work of this general type was conducted in this task.

IV. SUMMARY AND CONCLUSIONS

1. The weld structure of T-111 cannot be stabilized with respect to aging by post weld annealing for 1 hour at temperatures to 3000°F.

2. The aging response observed was limited to an observed shift in the bend transition temperature with aging at 2100°F (above 1500°F and below 2400°F). This was accompanied by weld precipitation associated with cored areas of solute enrichment.

3. Neither tensile properties nor hardness traverses responded to aging. Weld tensile ductility remained excellent irrespective of thermal history. Hence, the engineering properties of this alloy appear unaffected by aging.

4. The aging response appears to be peculiar to the 1t bend test which in this case represents a temperature sensitive test for accommodation of an outer fiber strain of 33-1/3%. This agrees with the tensile ductility of 25% which although excellent was less than that required for 1t bends.

5. All bend test fractures occurred by intergranular ductile tearing rather than by brittle cleavage. Hence, the bend-ductile-brittle-transition temperature is a misnomer for the effect observed in this program. Again, this shows that the observed aging response has no detrimental effect on the performance of this alloy.

6. Weld structure correlates with bend ductility in that a precipitation reaction occurs primarily at weld and interdendritic boundaries. The postulated mechanism of the bend ductility response was developed in Task III taking into consideration results of this Task. The postulated mechanism is illustrated in Figure 22. Upon aging a grain boundary precipitate typical in behavior to Ta_2C forms simultaneous with interdendritic precipitation of an intermetallic compound typified by W_2Hf. The grain boundary precipitate exerts the most pronounced influence since the fractures were intergranular. The interdendritic precipitate probably has an indirect influence on the aging response. Its substructure-like arrangement leads to strengthening within the weld grains and progressively to a greater differential between grain boundary and matrix strength with aging. This forces the grain boundaries to accommodate an increasingly larger share of the total strain with aging and enhances the tendency for grain boundary failures, a failure mode already promoted in welds by their large grain size (and hence, low total grain boundary area).

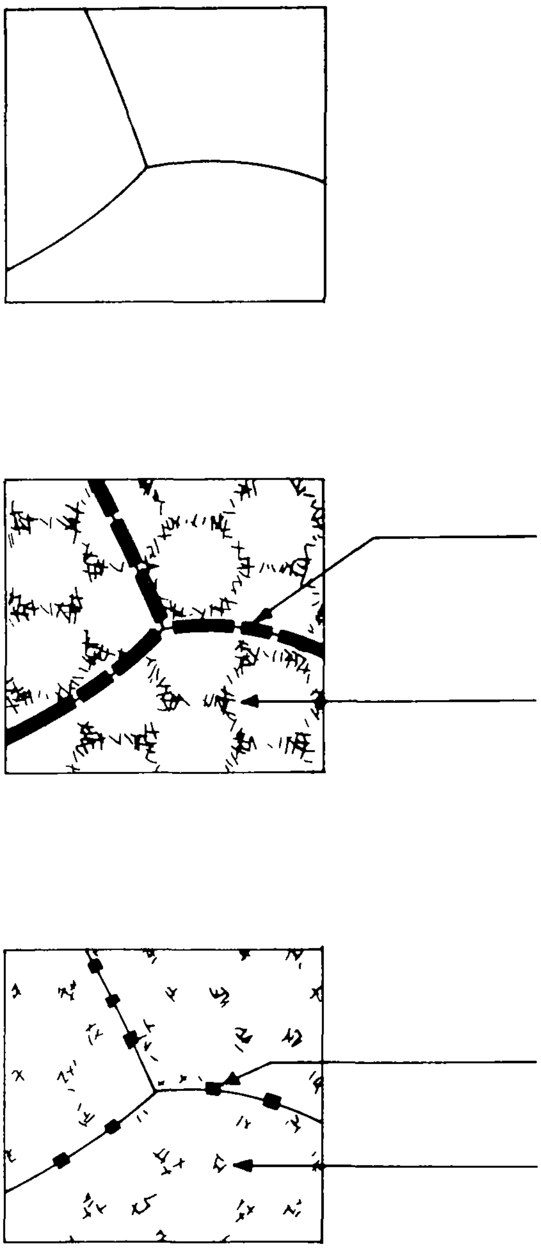


FIGURE 22 - Schematic Representation of Response of T-111 Weld Structure to Aging

The evidence suggesting the grain boundary precipitate to be an interstitial compound such as Ta_2C is as follows:

(i) Located preferentially at grain boundaries.

(ii) Stable within the approximate aging temperature range.

(iii) Unstable at or about 2400°F (may transform and go into solution at this temperature).

(iv) Primarily Ta-base (no W or Hf) as observed by x-ray spectrometry of the fracture surface compound.

The evidence suggesting the presence of an intermetallic phase (such as W_2Hf) at the interdendritic boundaries is as follows:

(i) The observed sluggishness in formation is typical of an intermetallic compound.

(ii) The large volume fraction of interdendritic precipitate seems to preclude the possibility it is an interstitial compound.

(iii) Solute redistribution to dendrite boundaries, as determined by electron beam microprobe analysis, results in W and Hf concentrations nearly stoichiometric with W_2Hf.

The compromising evidence for this hypothesis includes:

(i) Neither the grain boundary phase nor the interdendritic phase was positively identified. Ta_2C was detected by x-ray diffraction of bulk extraction residues but could not be definitely associated with the grain boundaries. Extraction of the intermetallic would be particularly difficult.

(ii) Phase relationships for W_2Hf in the ternary system are not known.

Other possibilities which were considered but seem to be precluded by the experimental evidence include:

(i) That the grain boundary precipitate is the MC phase (or some analogous phase), since this phase invariably is Hf-rich, a possibility refuted by the results of the x-ray spectrometry. Also, MC exhibits no preference for grain boundary precipitation.

(ii) That the interdendritic phase is a complex Ta-Hf compound forming due to the $\beta' - \beta''$ miscibility gap known for this alloy system. For such a reaction to occur an enormous local concentration of Hf (>30%) would be required.

(iii) That either the interdendritic or the grain boundary phase is HfO_2 since this compound, both in the monoclinic and cubic forms, is easily and routinely extracted and its concentration determined using bulk diffraction techniques. The HfO_2 concentration did not vary with location or aging conditions.

APPENDIX - PROGRAM TEST DATA

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BEND TEST RESULTS (Continued)

Weld No.	Figure No.	Page No.
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31	A15	59
32	A15	59
35	A11	55
36	A11	55
37	A11	55
38	A13	57
40	A12	56
41	A13	57
42	A12	56
43	A13	57
44	A4	48
47	A9	53
48	A10	54
49	A9	53
50	A9	53
51	A17	61
52	A16	60
53	A17	61
54	A14	58
55	A14	58
56	A14	58
58	A14	58
59	A7	51
60	A7	51

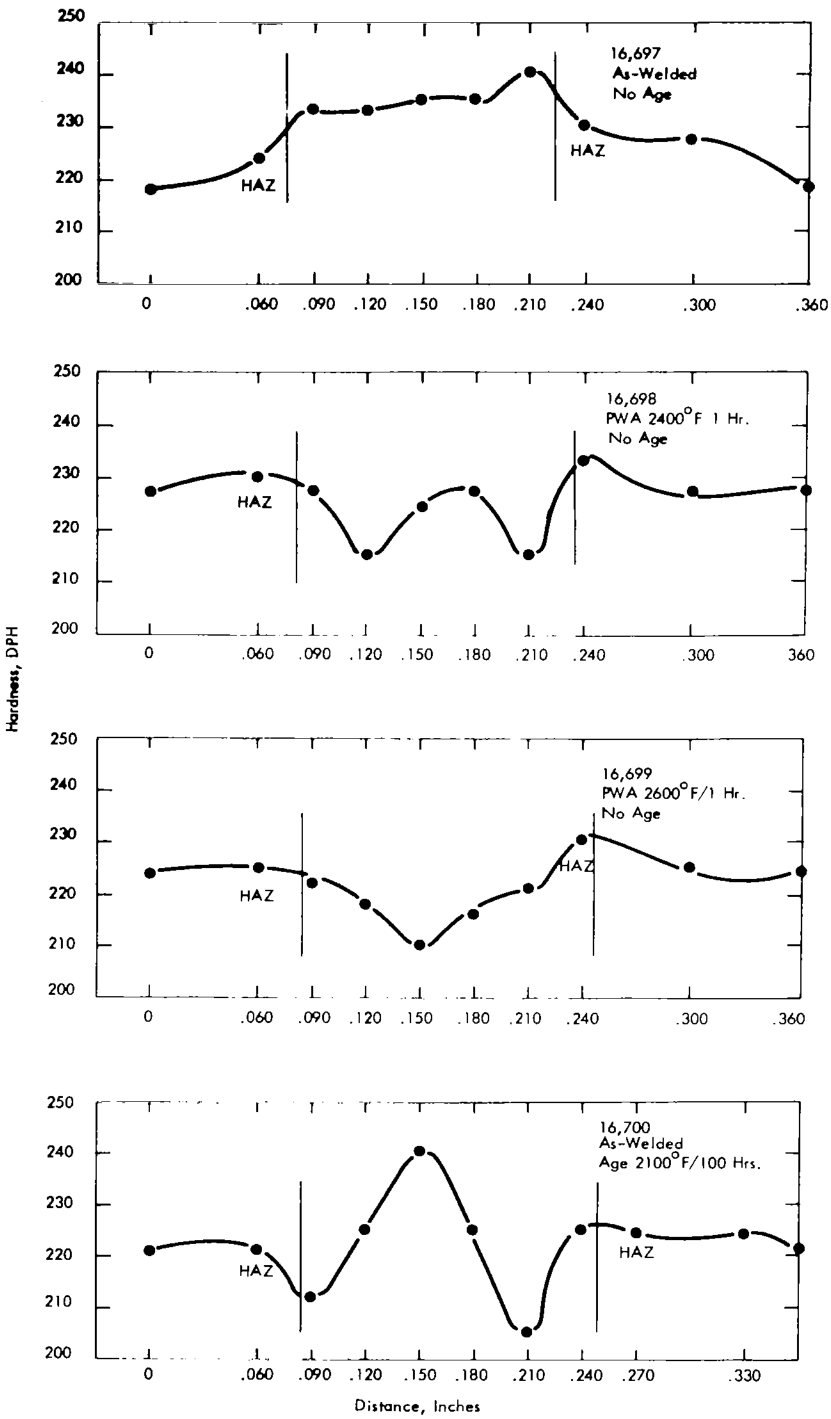


FIGURE A1 - T-111 GTA Weld Hardness Traverses. Low Heat Input Weld Parameters.

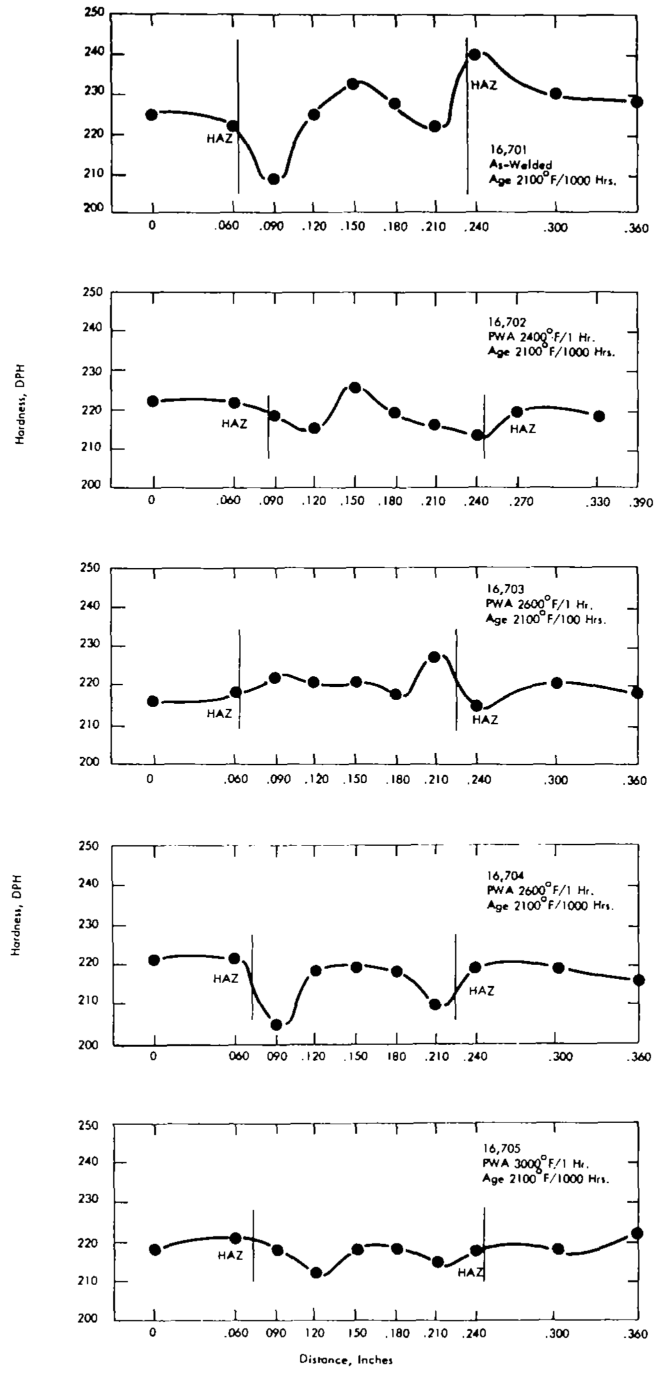


FIGURE A2 - T-111 GTA Weld Hardness Traverses. Low Heat Input Weld Parameters.

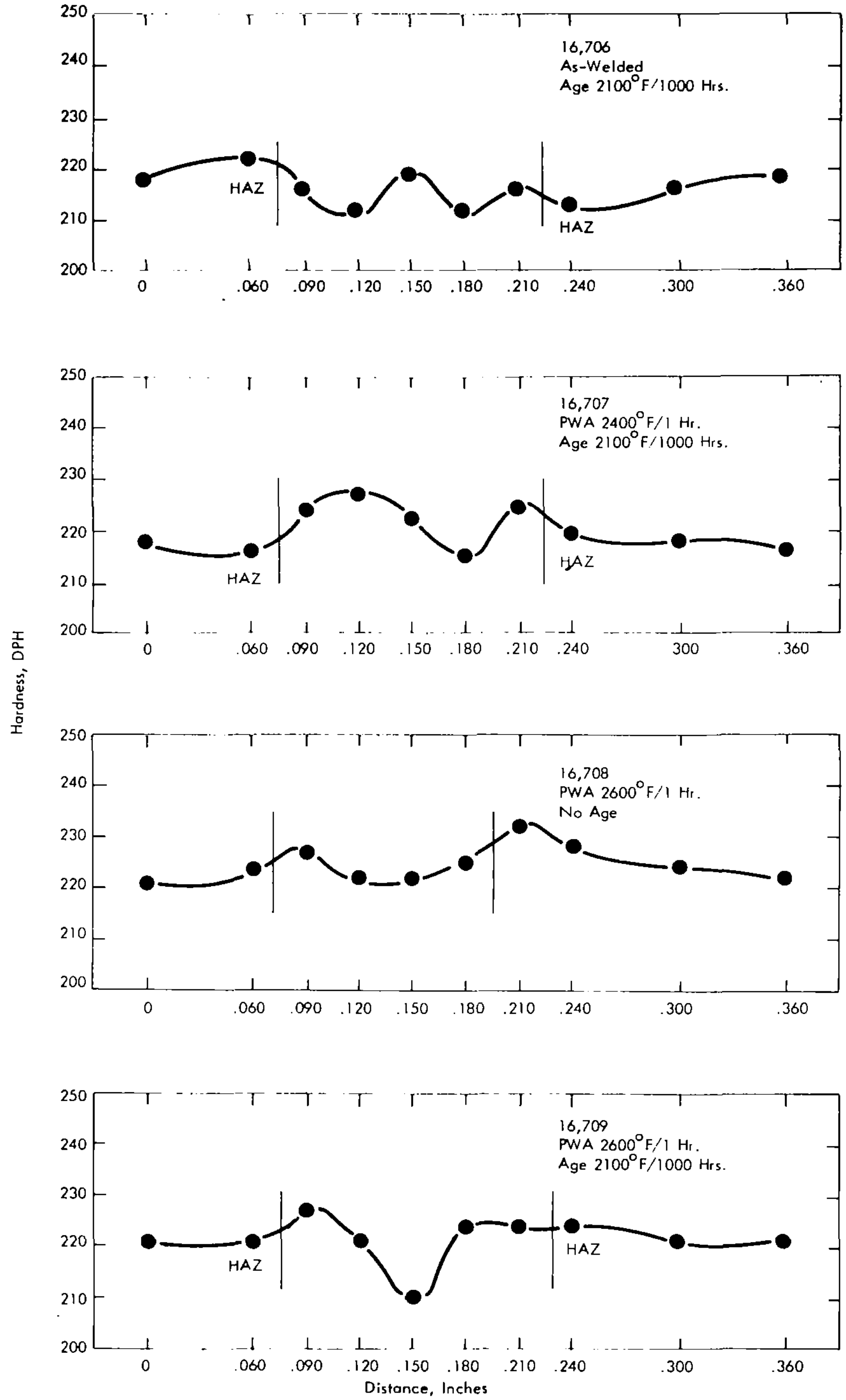
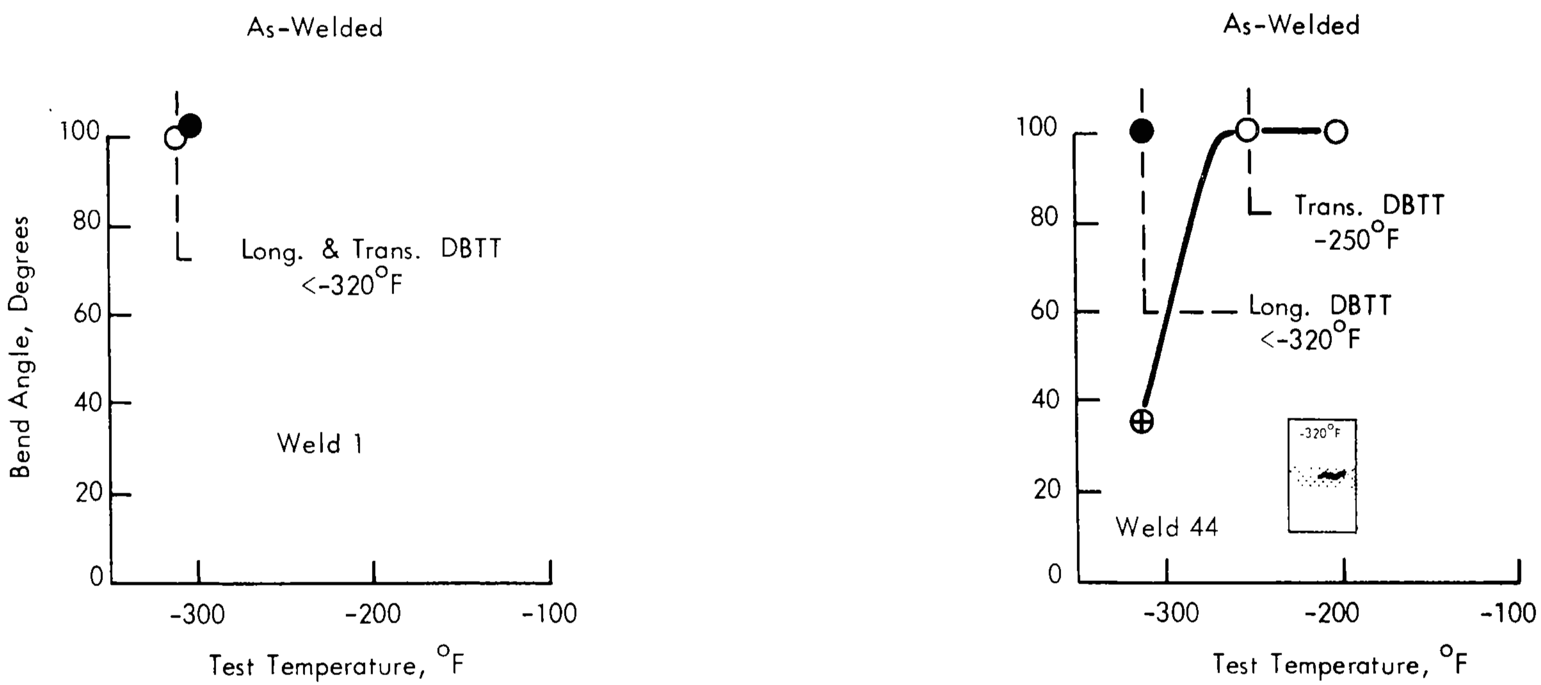


FIGURE A3 - T-111 GTA Weld Hardness Traverses. High Heat Input Weld Parameters.



Alloy: T-111
Aging: None
Welding: Low Heat Input GTA

Alloy: T-111
Aging: None
Welding: High Heat Input GTA

FIGURE A4 - Bend Test Results for Welds 1 and 44.

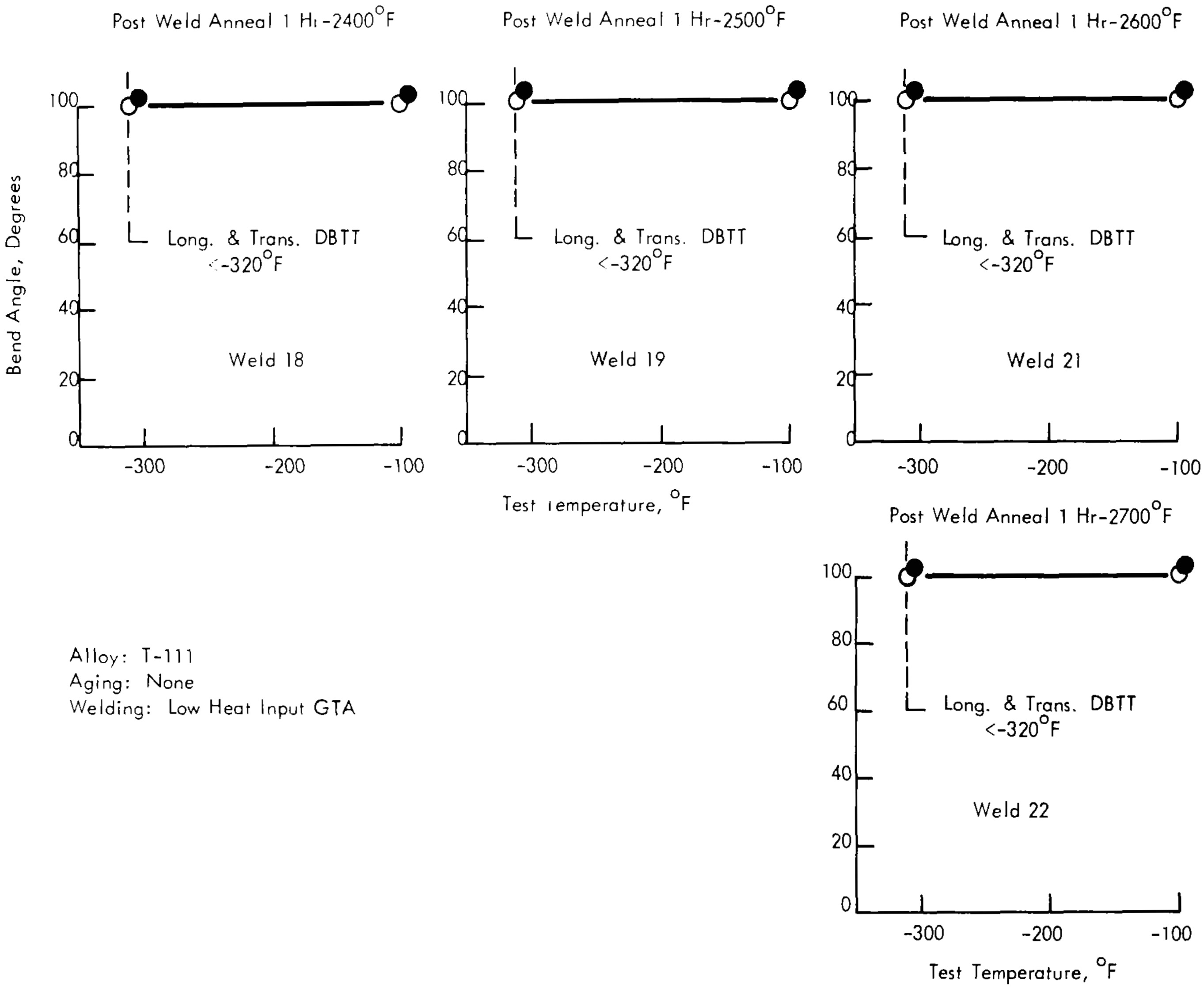
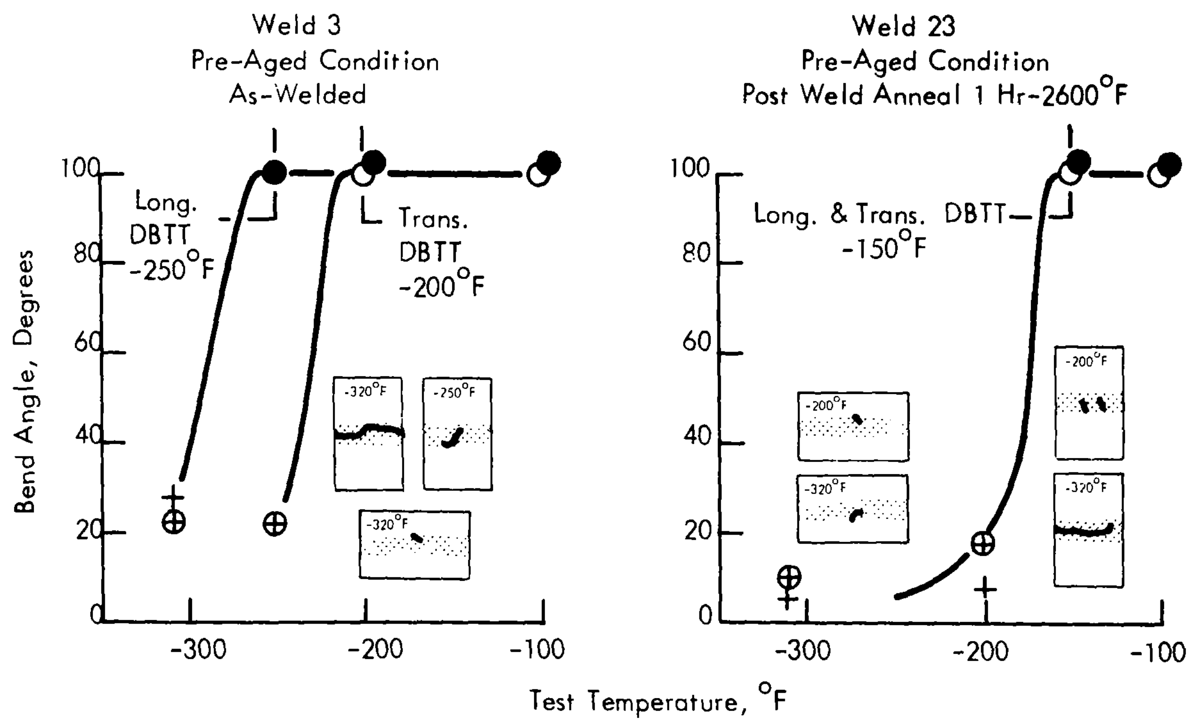
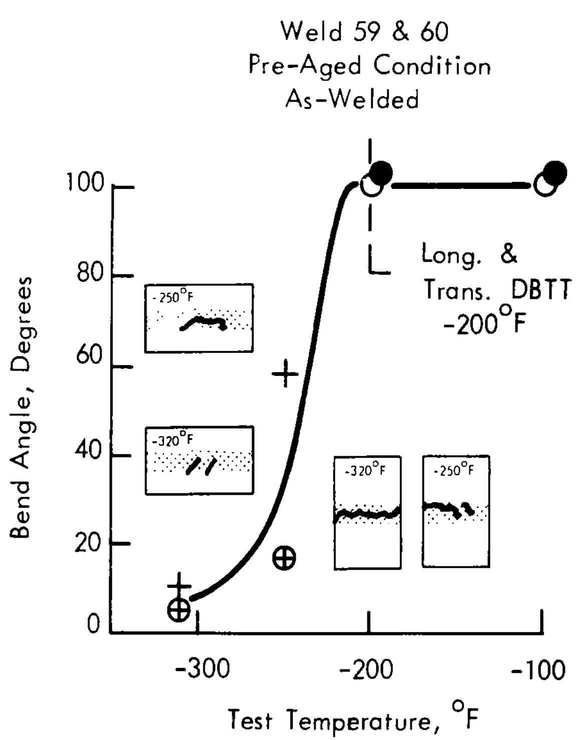


FIGURE A5 - Bend Test Results for Welds 18, 19, 21 and 22.



Alloy: T-111
Aging Temp.: 2100°F
Aging Time: 100 Hrs.
Welding: Low Heat Input GTA

FIGURE A6 - Bend Test Results for Welds 3 and 23.



Alloy: T-111
Aging Temp. : 2100°F
Aging Time: 100 Hrs.
Welding: Low Heat Input GTA

FIGURE A7 - Bend Test Results for Welds 59 and 60.

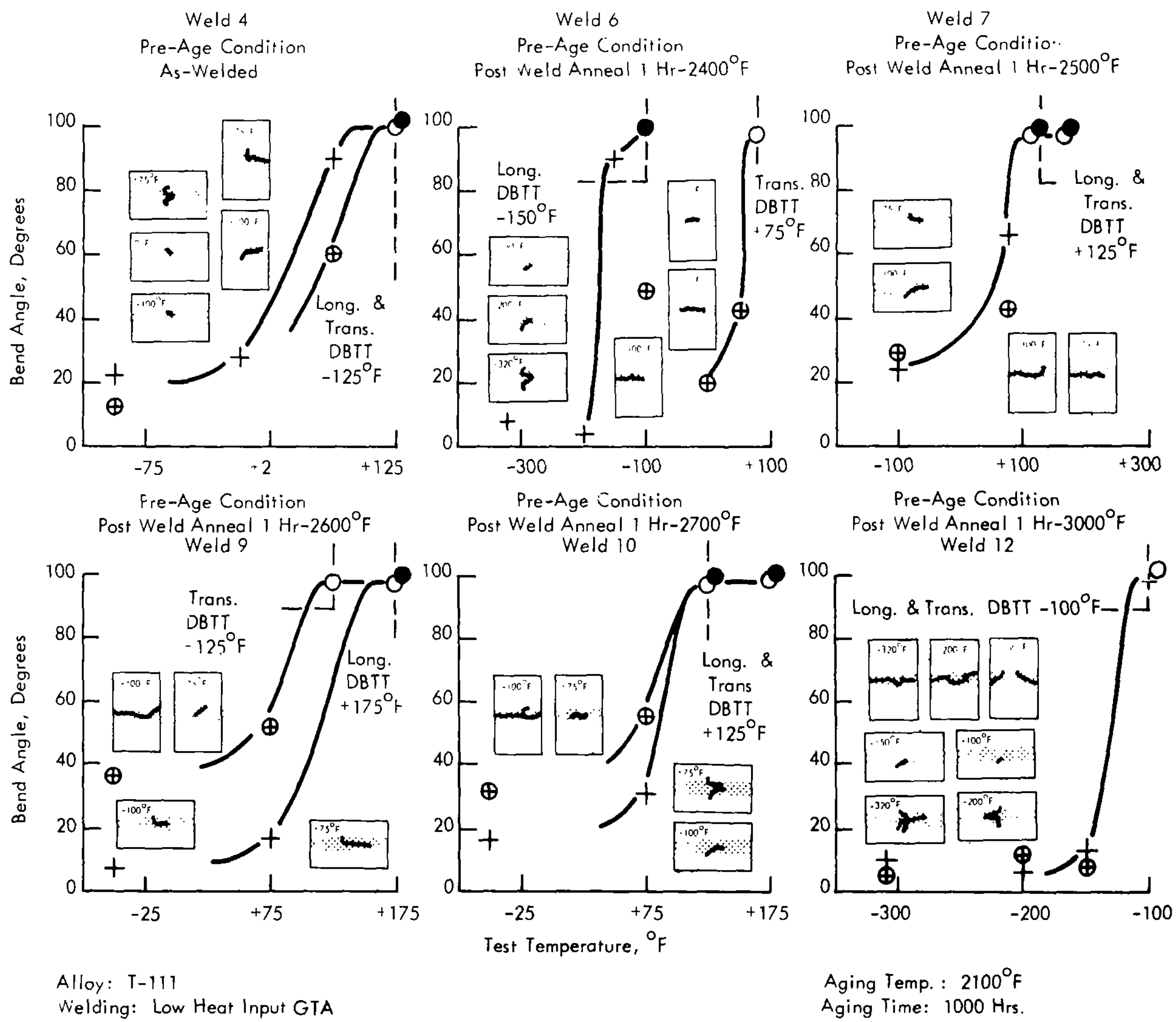
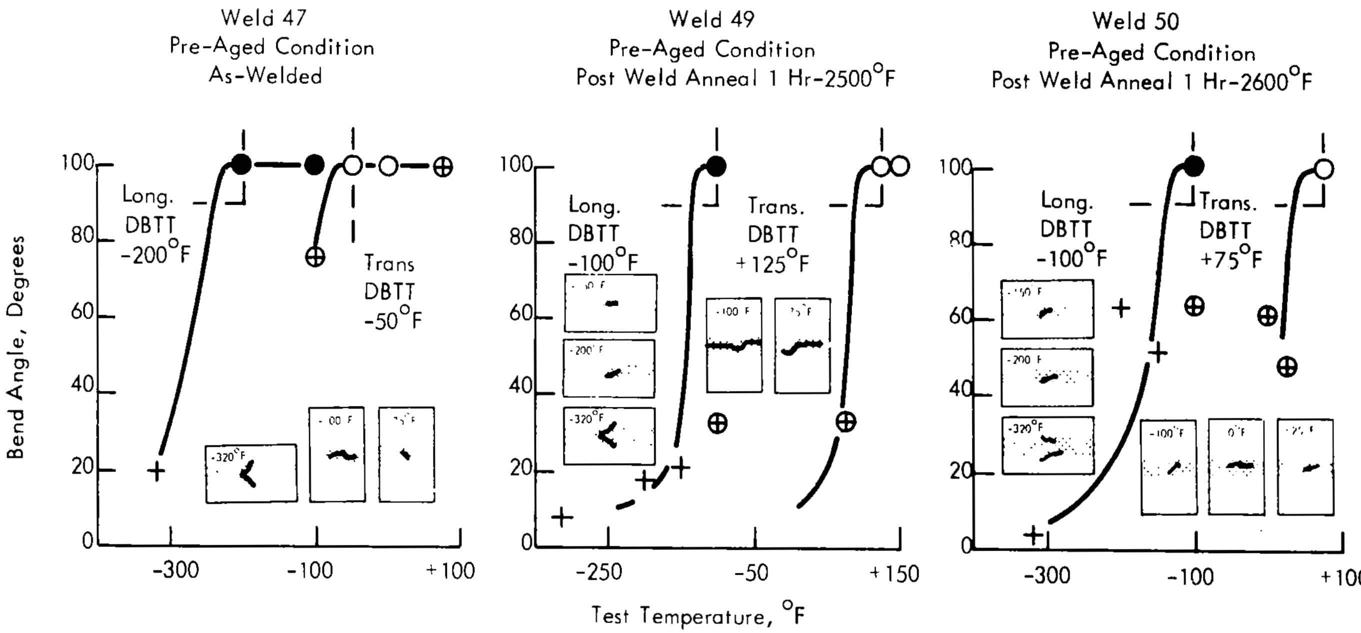
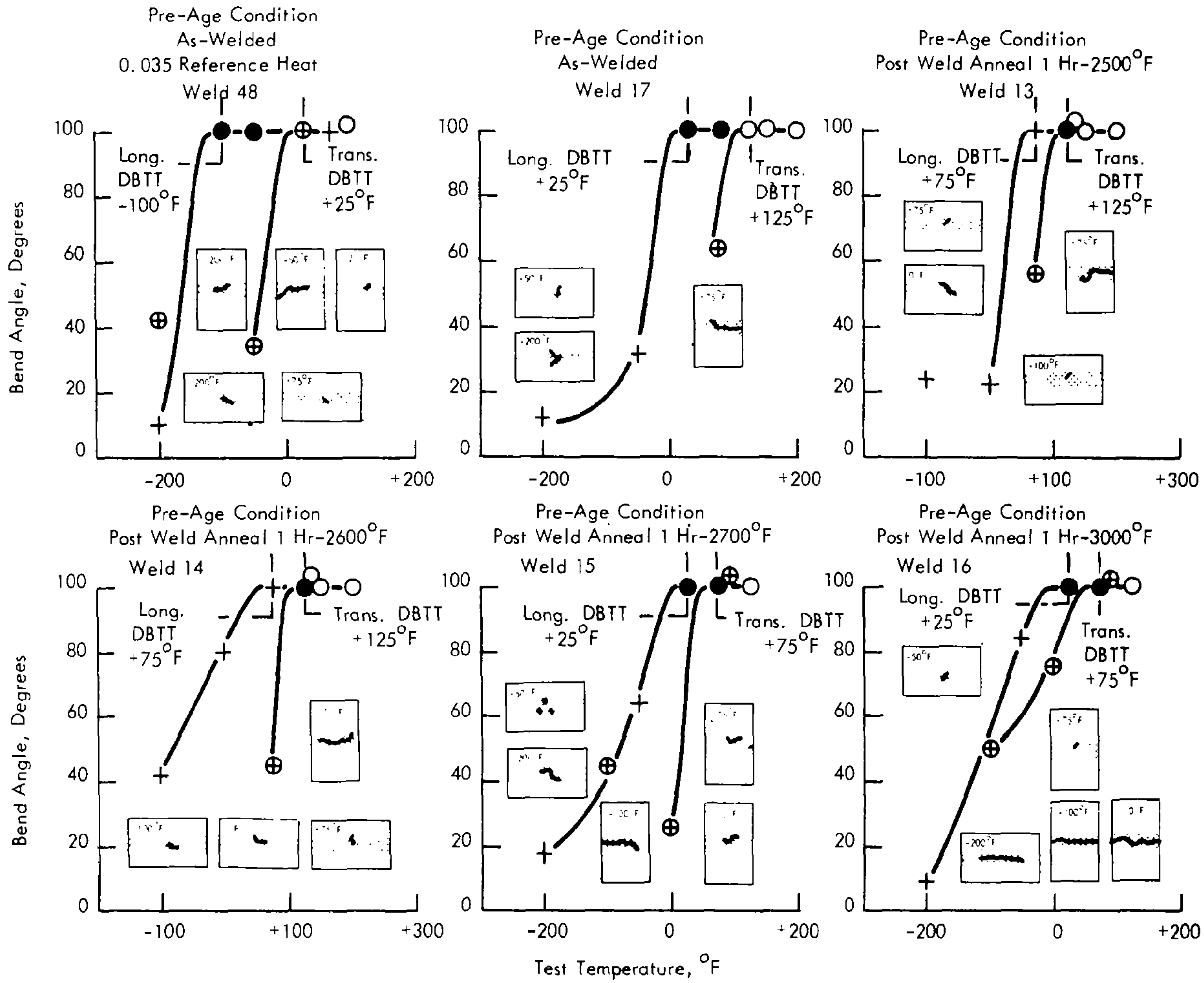


FIGURE A8 - Bend Test Results for Welds 4, 6, 7, 9, 10 and 12.



Alloy: T-111
Aging Temp.: 2100°F
Aging Time: 1000 Hrs.
Welding: Low Heat Input GTA

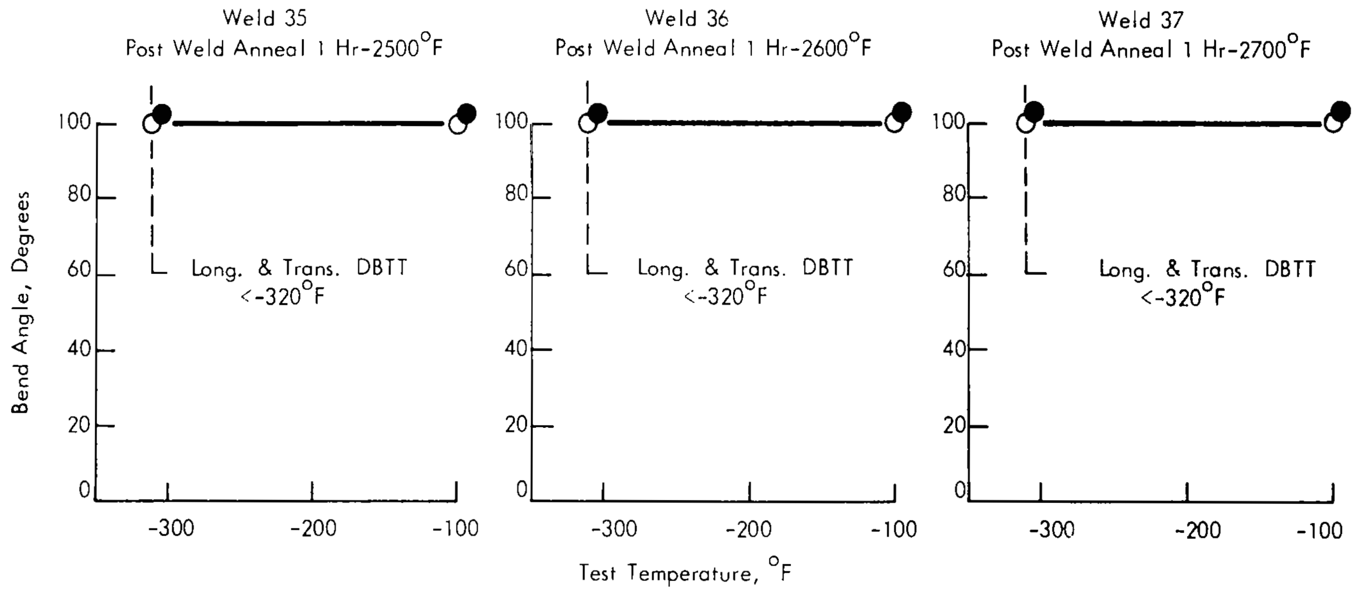
FIGURE A9 - Bend Test Results for Welds 47, 49 and 50.



Alloy: T-111
Welding: Low Heat Input GTA

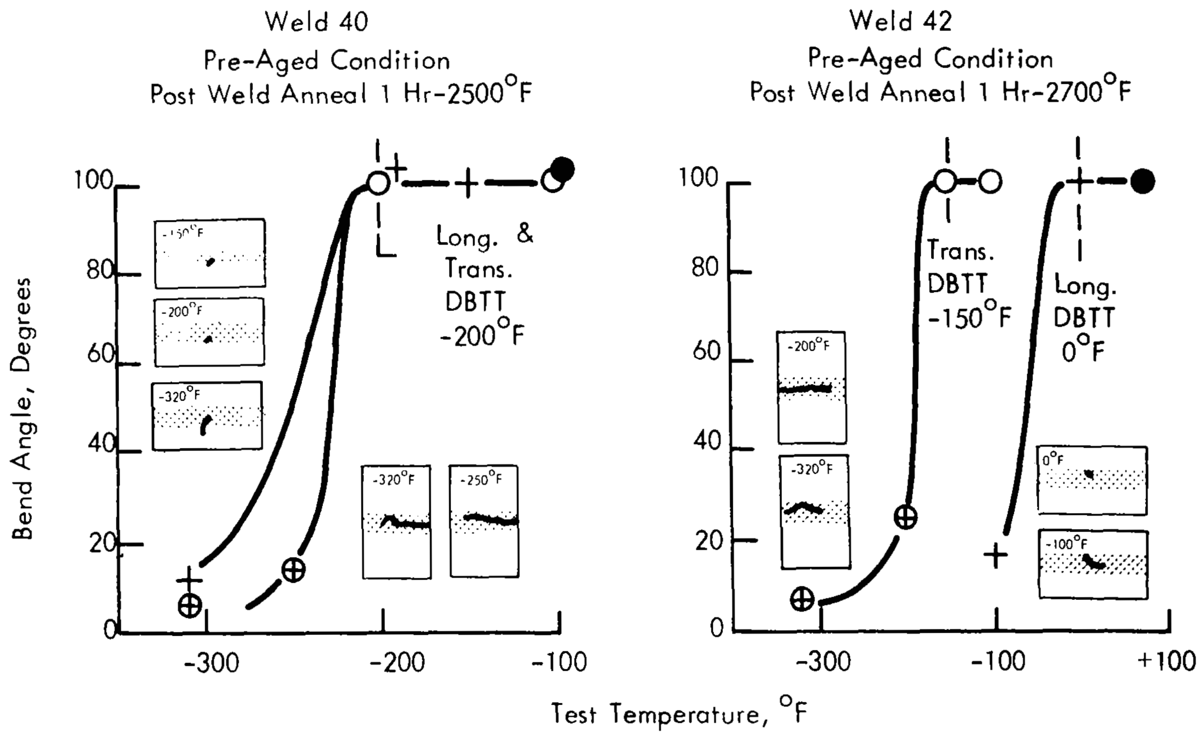
Aging Temp.: 2100°F
Aging Time: 5000 Hrs.

FIGURE A10 - Bend Test Results for Welds 48, 17, 13, 14, 15 and 16.



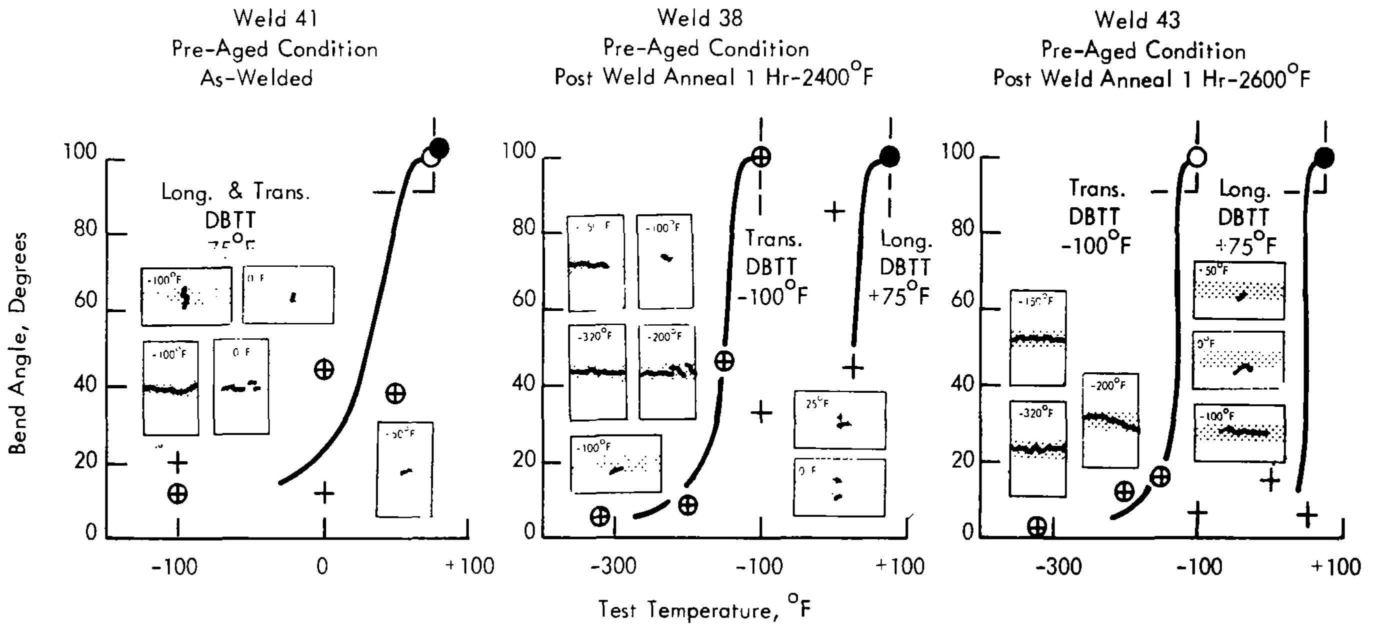
Alloy: T-111
Aging: None
Welding: High Heat Input GTA

FIGURE A11 - Bend Test Results for Welds 35, 36 and 37.



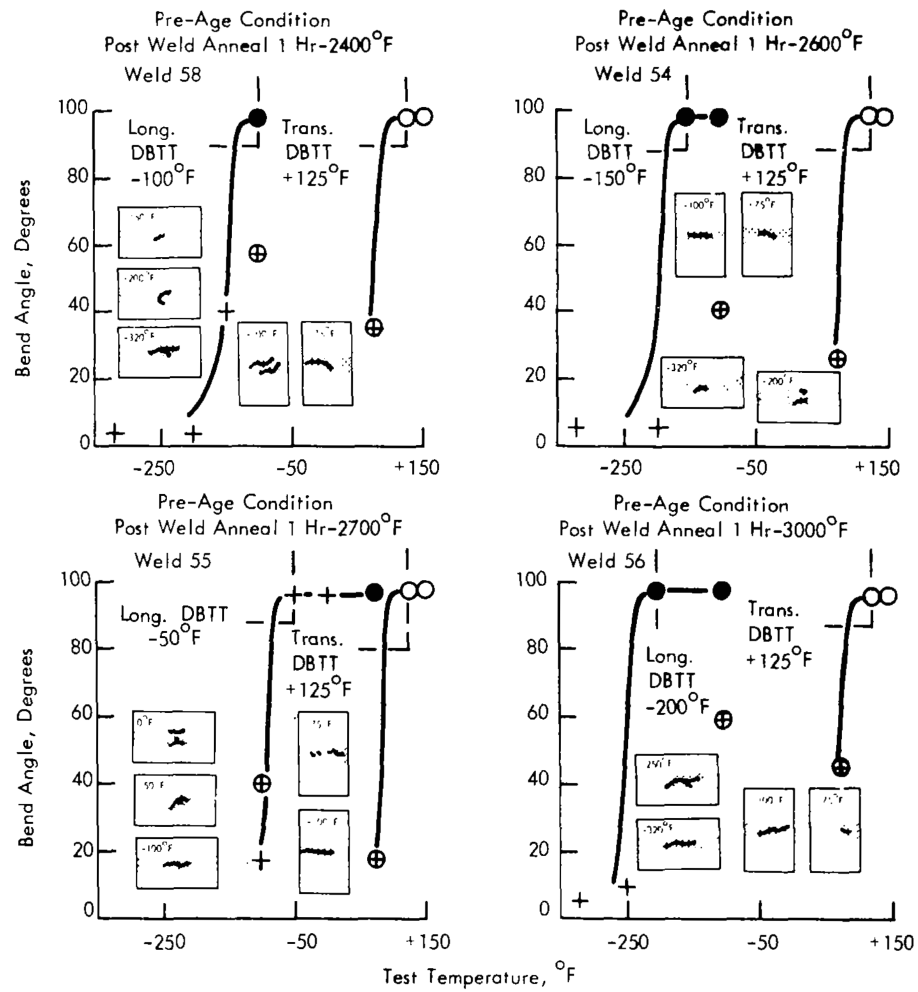
Alloy: T-111
Aging Temp.: 2100°F
Aging Time: 100 Hrs.
Welding: High Heat Input GTA

FIGURE A12 - Bend Test Results for Welds 40 and 42.



Alloy: T-111
Aging Temp.: 2100°F
Aging Time: 1000 Hrs.
Welding: High Heat Input GTA

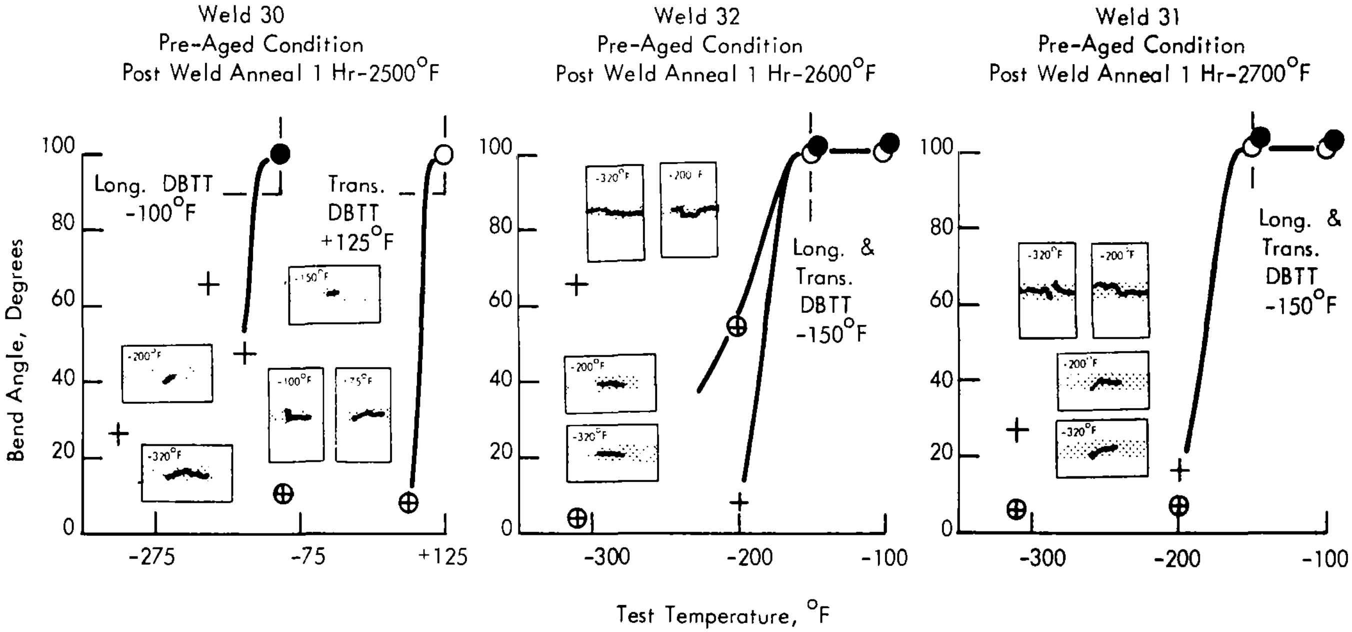
FIGURE A13 - Bend Test Results for Welds 41, 38 and 43.



Alloy: T-111
Welding: High Heat Input GTA

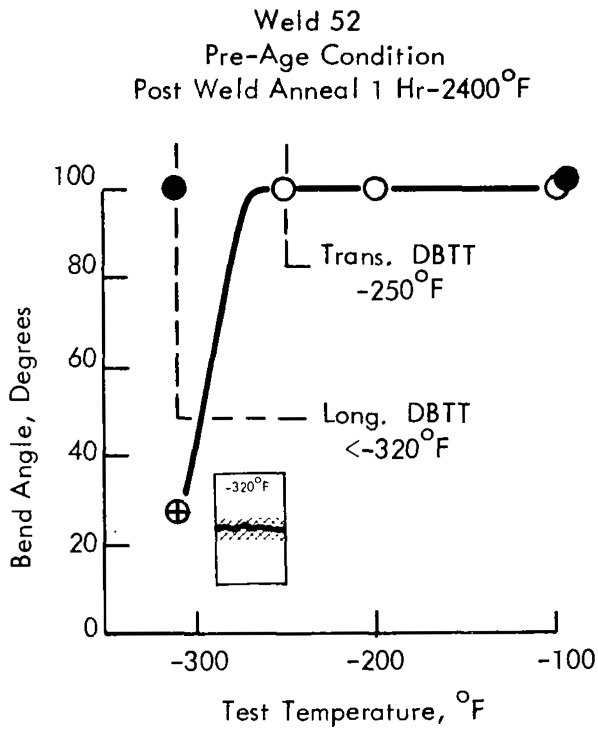
Aging Temp.: 2100°F
Aging Time: 1000 Hrs.

FIGURE A14 - Bend Test Results for Welds 58, 54, 55 and 56.



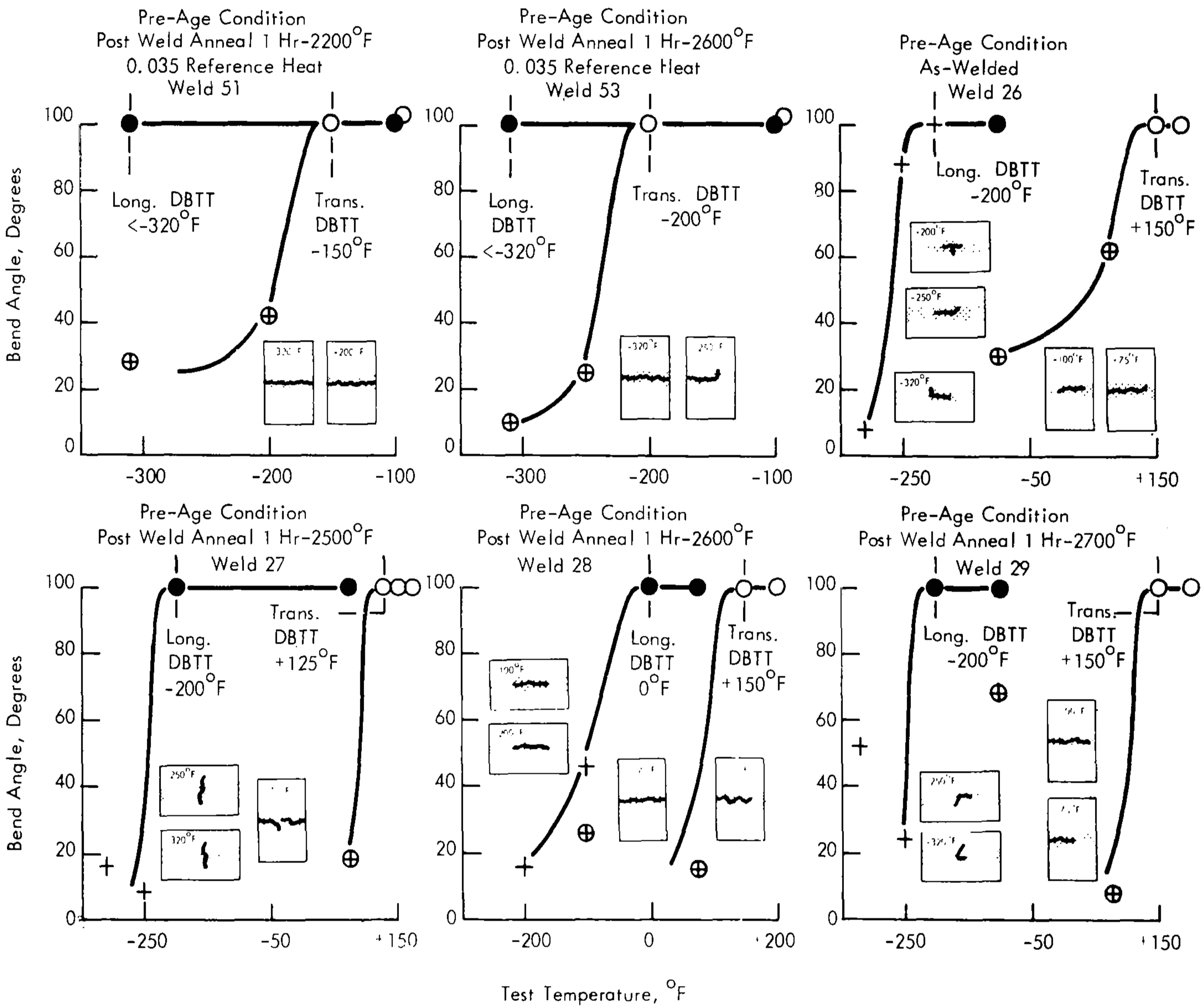
Alloy: T-111
Aging Temp.: 2100°F
Aging Time: 1000 Hrs.
Welding: EB

FIGURE A15 - Bend Test Results for Welds 30, 32 and 31.



Alloy: T-111
Aging Temp.: 2100°F
Aging Time: 1000 Hrs.
Welding: EB

FIGURE A16 - Bend Test Results for Weld 52



Alloy: T-111
Welding: EB

Aging Temp.: 2100°F
Aging Time: 5000 Hrs.

FIGURE A17 - Bend Test Results for Welds 51, 53, 26, 27, 28 and 29.