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# HEAVY WATER ORGANIC COOLED REACTOR

## INVESTIGATION OF TUNGSTEN AS A DIFFUSION BARRIER FOR HWO CR SAP - STEEL PRESSURE TUBE TRANSITION JOINTS

By  
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## CONTENTS

	Page
Abstract . . . . .	5
I. Introduction . . . . .	7
II. Experimental Procedure . . . . .	11
A. Preparation of Diffusion Couples . . . . .	11
B. Diffusion Annealing . . . . .	13
C. Growth and Identification of Diffusion Layers . . . . .	14
III. Results . . . . .	17
A. Growth and Characterization of Diffusion Layers . . . . .	17
B. Data Analysis . . . . .	23
1. SAP 895 - Tungsten Data . . . . .	23
2. SAP 895 - Type 304 SS Data . . . . .	26
IV. Discussion . . . . .	27
V. Conclusions . . . . .	29

## TABLES

1. Summary of Diffusion Data for SAP 895 - W and SAP 895 - Type 304 SS Systems . . . . .	16
2. Analysis of Diffusion Data for SAP 895 - W and SAP 895 - Type 304 SS Systems . . . . .	24

## FIGURES

1. HWO CR Pressure Tube Transition Joint . . . . .	8
2. SAP 895 - Tungsten Diffusion Couple . . . . .	10
3. Diffusion Couple Bonding Apparatus . . . . .	12
4. Diffusion Couple . . . . .	13
5. Microstructure of Couple No. 26 Showing Diffusion Zone Between SAP 895 and Tungsten After 768 hr at 980° F . . . . .	18
6. Microstructure of Couple No. 47 Showing Diffusion Zone Between SAP 895 and Tungsten After 745 hr at 960° F . . . . .	18
7. Microstructure of Couple No. 20 Showing Diffusion Zone Between SAP 895 and Tungsten After 24 hr at 1075° F . . . . .	19

## FIGURES

	Page
8. Microstructure of Couple No. 25 Showing Formation of Nodules After 96 hr at 1000°F . . . . .	20
9. Microstructures of Diffusion Zones in Couple No. 10 After 4841 hr at 940°F . . . . .	21
10. Microstructure of Couple No. 41 Showing Penetration of Defective Tungsten Coating by Aluminum and Resultant Reaction with Nickel After 2756 hr at 920°F . . . . .	22
11. Microstructure of Couple No. 38 Showing Diffusion Zone Between SAP 895 and Type 304 SS After 1177 hr at 980°F . . . . .	22
12. Arrhenius Plot of SAP 895 - Tungsten Diffusion Data . . . . .	25
13. Arrhenius Plot of SAP 895 - Type 304 SS Diffusion Data . . . . .	26

## ABSTRACT

The Heavy Water Moderated Organic Cooled Reactor requires a transition joint between the SAP 895\* pressure tube and the out-of-core carbon steel piping. An intermediate section of stainless steel is metallurgically bonded to the SAP tube and welded to the carbon steel. A tungsten barrier layer (applied by chemical vapor deposition) is used at the SAP-stainless steel joint interface to prevent the formation of brittle intermetallic compounds by diffusion between stainless steel and aluminum during the 30-year design life of the pressure tube at temperatures to 765°F. This study was conducted to determine the extent of diffusion that could be expected between the constituents of the bond system, particularly between SAP 895 and tungsten, due to the time-temperature conditions of reactor service.

Diffusion couples were prepared which simulated the metallurgical bonding obtained in the pressure tube transition joint. The couples were diffusion annealed in the temperature range of 920° to 1125°F for periods of from 4 to 4841 hr. The design of the couple provided diffusion data for SAP 895 - Type 304 SS as well as for SAP 895 - W. Data analysis indicated a relationship in the SAP 895 - W system between total diffusion layer thickness  $x$  (in.), time  $t$  (hr) and temperature  $T$  (°K) to be:

$$\frac{x^{0.90}}{t} = 3.27 \times 10^{17} \exp\left(\frac{-82,500}{RT}\right) .$$

A similar relationship in the SAP 895 - Type 304 SS system is:

$$\frac{x^2}{t} = 1.35 \times 10^4 \exp\left(\frac{-40,800}{RT}\right)$$

It was concluded that the maximum diffusion layer that could be expected between SAP 895 and tungsten after 30 years at 765°F is 0.000108 in., which is not considered detrimental to the transition joint tensile strength.

\*Sintered aluminum powder product consisting of a nominal 10% aluminum oxide dispersed in aluminum.



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## I. INTRODUCTION

The Heavy Water Moderated Organic Cooled Reactor (HWOCR) core design requires SAP 895 pressure tubes to contain the fuel elements and to constitute a portion of the organic coolant circuit. Carbon steel is used to complete the out-of-core coolant piping. An intermediate section of stainless steel is hot pressed bonded to the SAP tube and welded to the carbon steel, as shown in Figure 1. The SAP-stainless steel transition joint is required to maintain high strength and leak-tight integrity at a maximum service temperature of about 765°F for a design life of 30 years.

Past experience<sup>\*</sup> has shown that a diffusion barrier is required at the SAP-stainless steel joint interface to prevent the formation of brittle intermetallic compounds by diffusion in reactor service. A thin tungsten coating, applied by chemical vapor deposition to the stainless steel component prior to bonding, has been effectively utilized<sup>†</sup> as a diffusion barrier to aluminum.

The purpose of this investigation was to determine the extent of diffusion that could be expected between SAP 895 and vapor-deposited tungsten in 30 years at 765°F. By annealing appropriate diffusion couples at temperatures above 765°F, the diffusion process is greatly accelerated. A primary objective was to define an expected relationship of the type

$$x^n = k_o t \exp (-Q/RT) \quad \dots (1)$$

where

$x$  = thickness of diffusion layer (in.)

$t$  = time (hr)

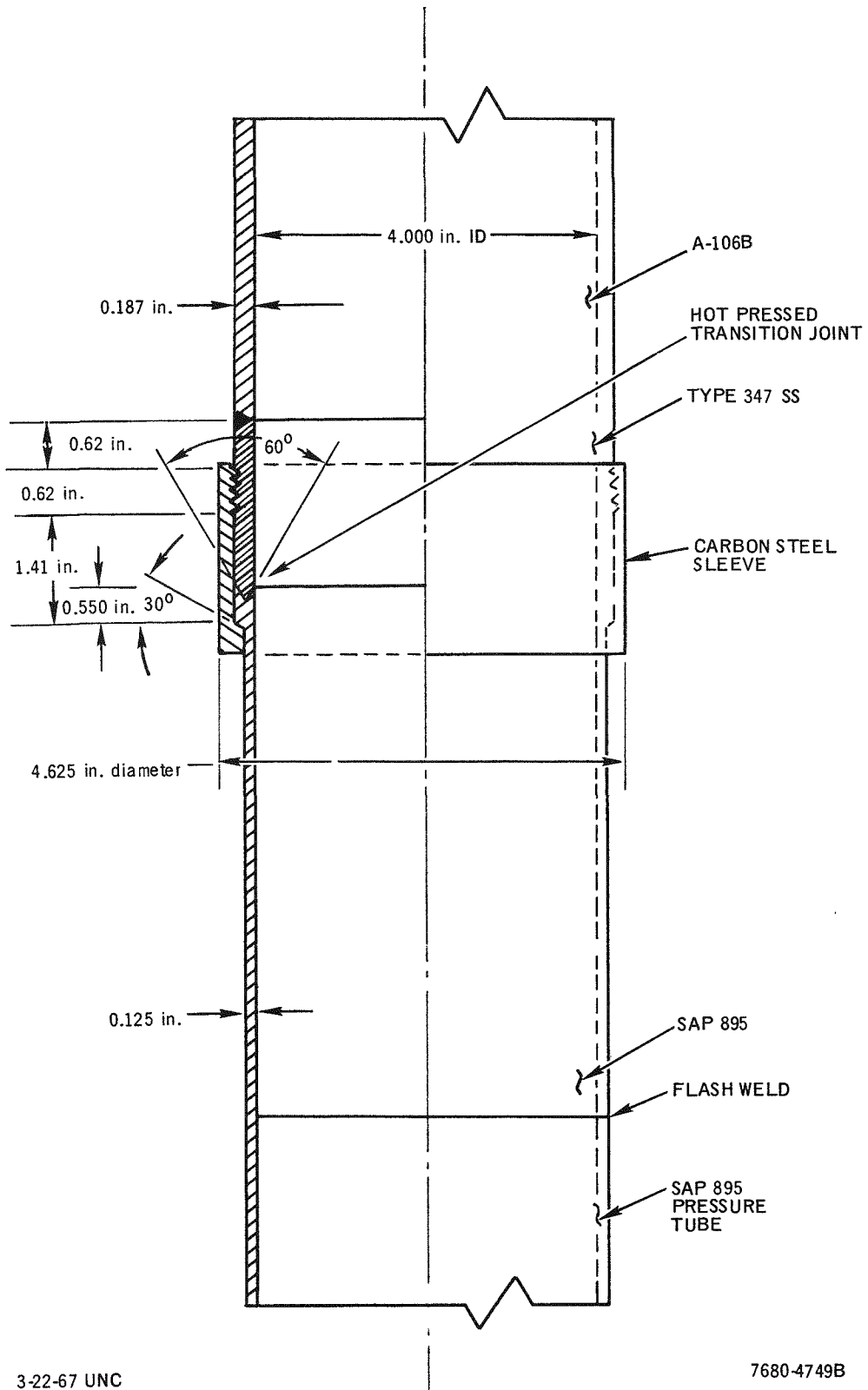
$Q$  = activation energy (cal/mol)

$R$  = gas constant

$T$  = absolute temperature (°K)

\*E. C. Supan and W. H. Friske, "Development of APM-Stainless Steel Process Tube Transition Joints for the 30-Mwe DON Prototype Power Reactor," AI-65-114 (May 24, 1965)

†E. C. Supan, "Development of SAP-Steel Pressure Tube Transition Joints for the HWOCR," AI-CE-46 (August 15, 1967)



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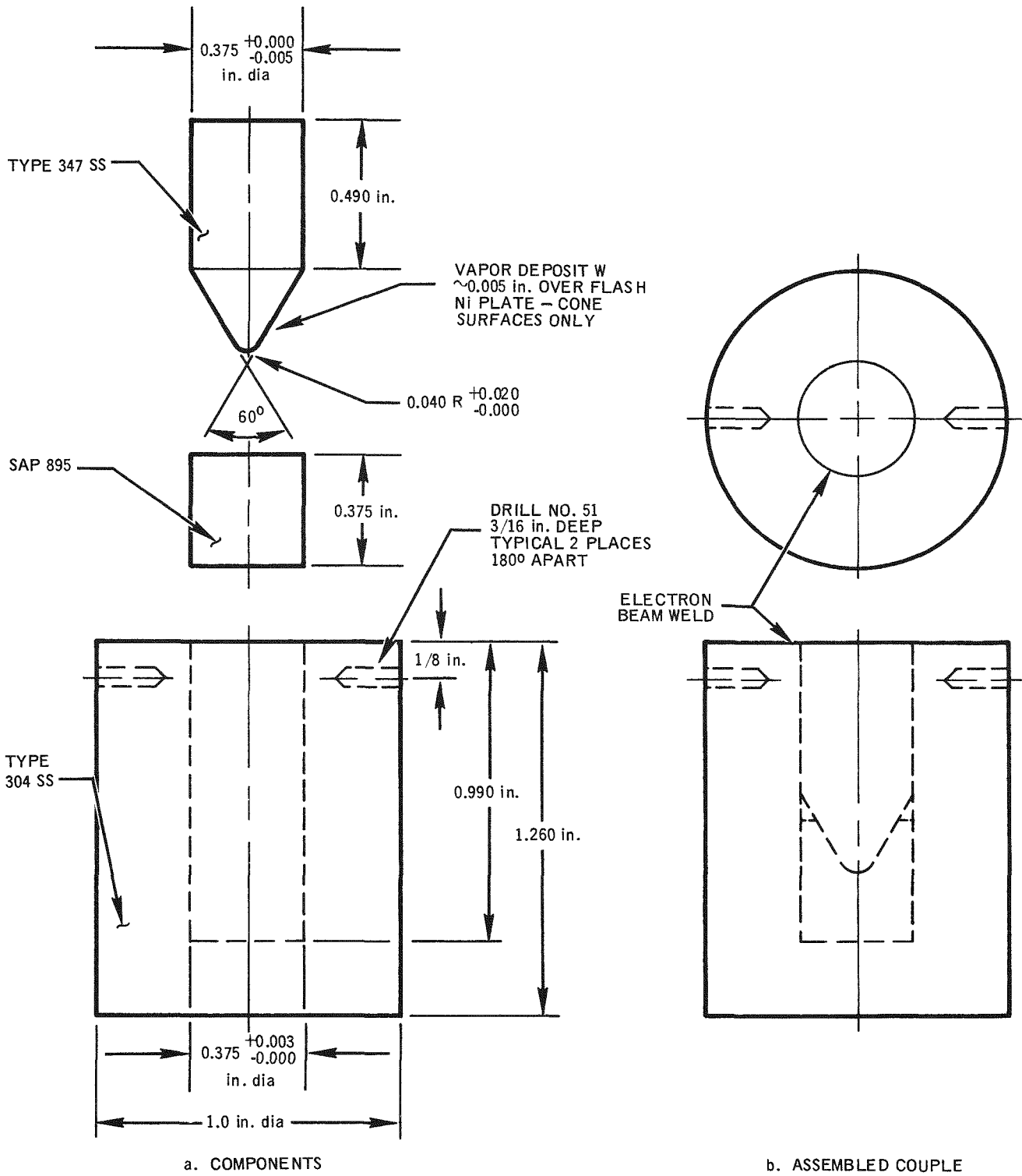
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Figure 1. HWOCR Pressure Tube Transition Joint

The determination of the activation energy is particularly important. From Equation 1, it can be shown that the required time ( $t_2$ ) at a higher temperature ( $T_2$ ) to give an equivalent diffusion thickness in 30 years ( $t_1$ ) at 765°F ( $T_1 = 680^\circ\text{K}$ ) is related to  $Q$  by the equation:

$$\ln\left(\frac{t_2}{t_1}\right) = \frac{Q}{R}\left(\frac{1}{T_2} - \frac{1}{T_1}\right) \quad \dots (2)$$

where the same diffusion constants must be valid at the two temperatures ( $T_1$  and  $T_2$ ). Therefore, the activation energy value provides a basis for selecting time-temperature combinations to simulate 30-year service diffusion in accelerated tests. Such tests are useful in predicting the effect of service life diffusion on the physical and mechanical properties of transition joints.



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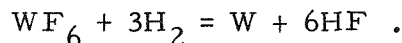
Figure 2. SAP 895 - Tungsten Diffusion Couple

## II. EXPERIMENTAL PROCEDURE

### A. PREPARATION OF DIFFUSION COUPLES

The diffusion couple selected for this study simulates the metallurgical bonding process which is characteristic of the pressure tube transition joint shown in Figure 1. The design of the diffusion couple is illustrated in Figure 2. In a manner analogous to transition joint fabrication, the application of heat and pressure causes the SAP to flow over the cone-shaped tungsten-coated component, resulting in solid-state bonding.

The cone-shaped stainless steel components were prepared for bonding by electroplating with nickel, followed by chemical vapor deposition of tungsten. The nickel coating provides an oxide-free substrate for the tungsten coating operation. Nickel coating thicknesses of about 0.2 to 0.5 mil were electro-deposited from a Wood's\* nickel strike bath at room temperature for 5 min at 4 volts. Tungsten was chemically vapor deposited by hydrogen reduction of tungsten hexafluoride:

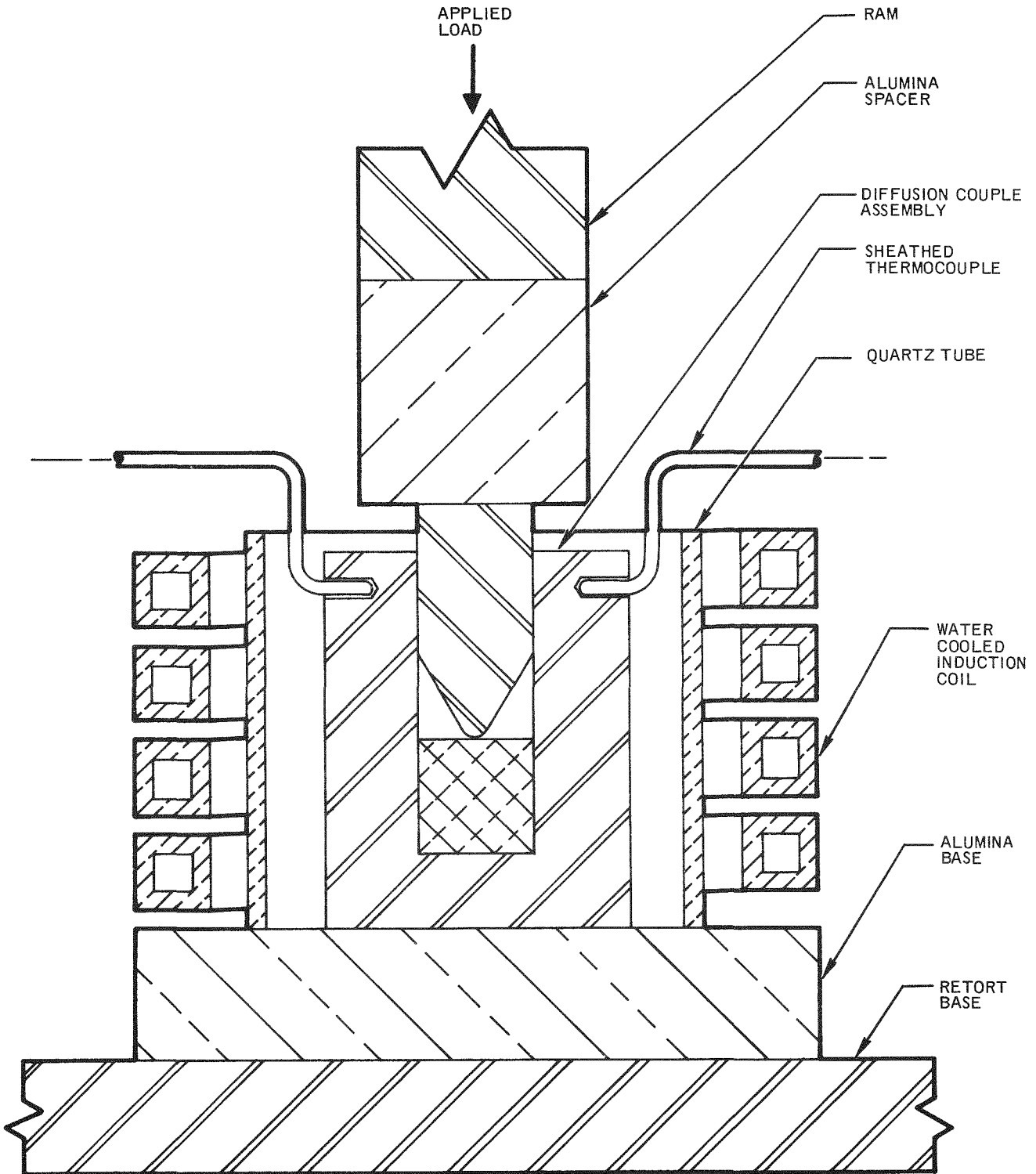


The reaction takes place on the heated substrate (nickel-plated cone). The parts were locally heated by RF induction in a Vycor tube evacuated to  $<5 \times 10^{-5}$  torr until outgassing essentially ceased ( $\sim 2$  to 3 min). Hydrogen and  $\text{WF}_6$  were introduced into the Vycor chamber under a partial vacuum of 28.5 in. of Hg for 8 min. The parts were then allowed to cool in vacuum. The resulting tungsten coating was approximately 5 mils thick.

The apparatus used for bonding of the diffusion couples is shown schematically in Figure 3. The diffusion couple components were positioned in a stainless steel vacuum-tight retort enclosure. The retort (not shown) was then evacuated and backflushed with argon three times and finally evacuated and maintained at a pressure of  $<10^{-2}$  torr. The diffusion couple components were heated by RF induction to a temperature of 1000°F. The temperature was continuously monitored on a two-pen recorder by the output of Chromel-Alumel sheathed

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\*Nickel chloride 28 to 34 oz/gal, HCl 14 to 18 oz/gal.



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Figure 3. Diffusion Couple Bonding Apparatus

thermocouples located in the holes provided in the diffusion couple housing. A small hydraulic press was used to apply a load of 10,000 lb to the tungsten-coated component, causing it to penetrate the SAP component and its top to become flush with the housing. The RF heat input was immediately removed and the bonded diffusion couple was allowed to cool under load to about 600°F before removal from the retort. The diffusion couples were sealed by electron beam welding. Since the welding was performed in vacuum, the internal voids of the couple remain evacuated as a result of the welding operation. Figure 4 shows a completed diffusion couple and one which has been sectioned by electrical discharge machining.

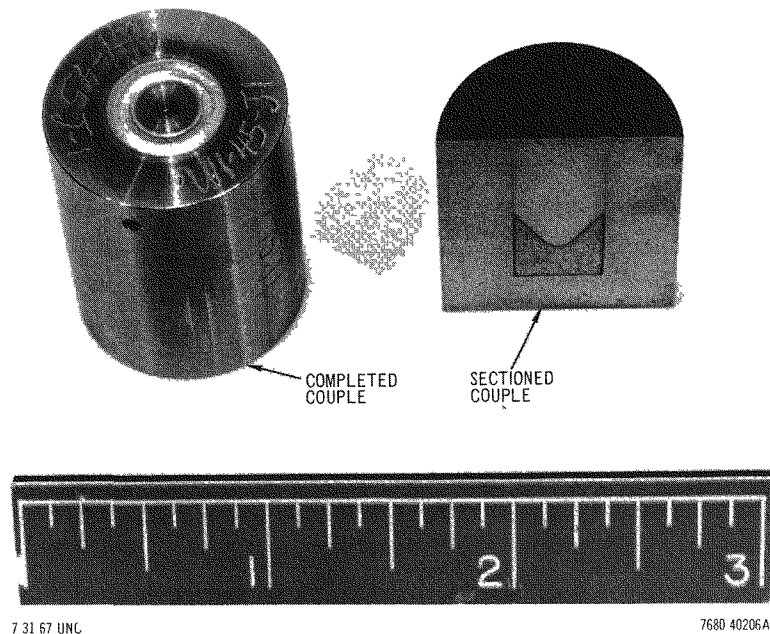


Figure 4. Diffusion Couple

#### B. DIFFUSION ANNEALING

The diffusion couples were annealed for various times at temperatures of 1000° to 1125°F in 25°F increments, and 920° to 980°F in 20°F increments. The couples annealed at 1000°F and above were heated in lead baths, and the couples annealed at temperatures below 1000°F were heated in salt baths. The salt baths consisted of a ternary eutectic mixture having a composition of 30% LiNO<sub>3</sub>, 53% KNO<sub>3</sub>, and 17% NaNO<sub>3</sub> (melting point: ≈245°F). The lead and salt baths were used because of their good temperature uniformity.

During the course of this study, the higher temperature couples were examined first because of their shorter annealing time requirements. It was found that the diffusion rates were highly temperature dependent. For this reason, the temperature intervals were decreased from 25°F to 20°F for annealing temperatures below 1000°F.

Each diffusion couple was instrumented with a calibrated 1/16-in.-diameter Chromel-Alumel sheathed thermocouple positioned in one of the two holes provided in the diffusion couple housing. Temperatures were monitored by strip-chart recorders and by potentiometer measurements of the thermocouple outputs. Corrections to the nominal annealing temperatures were based on the potentiometer measurements and individual thermocouple calibrations. Annealing times were generally selected to obtain a diffusion layer thickness between SAP 895 and tungsten of about 1 to 5 mils.

### C. GROWTH AND IDENTIFICATION OF DIFFUSION LAYERS

The annealed diffusion couples were sectioned either with an abrasive cut-off wheel or by electrical discharge machining. Sectioning was done in such a manner that the flat surface of the final polished specimen passed very nearly through the centerline of the couple to minimize any errors in measurement of diffusion layer thicknesses due to oblique cuts.

The sectioned diffusion couples were mounted in epoxy resin. Rough polishing was done progressively on silicon carbide abrasive papers, 180 through 600 grit sizes. Further polishing was done using 30- $\mu$  and 6- $\mu$  diamond compounds. Final polishing was accomplished with a slurry of Linde B compound in water.

The polished specimens were etched at room temperature in two steps: (1) electrolytic etching in 10% oxalic acid solution for 10 to 20 sec at 5 volts, and (2) swab etching with Murakami's Reagent\* for 1 to 5 sec.

The total aluminum-tungsten diffusion layer thickness was measured on a Tukon microhardness tester in filar units and converted to inches. The diffusion layer thicknesses were highly irregular. Because of this irregularity, the maximum thickness was measured at five different locations on the sides of the tungsten-coated cone interface with the SAP.

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\*Potassium ferricyanide 10 g, potassium hydroxide 10 g, water 100 ml.



No diffusion layers between nickel and tungsten were observed.

Although it was not a primary objective of this study to determine the diffusion between SAP 895 and Type 304 stainless steel, the data were of interest, and were readily available from the annealed couples. Therefore, the total layer thicknesses between the side of the SAP 895 component and the couple housing were measured. The total diffusion layer was characteristically quite uniform. The maximum layer thickness was measured with a microscope having a micrometer eyepiece calibrated in inches. In a few cases, the diffusion layers were extensively damaged as a result of sectioning and polishing so that a representative measurement could not be made.

Identification of aluminum-tungsten intermetallic compounds in the total diffusion layers of selected couples was determined by X-ray powder diffraction techniques and by electron microprobe analyses. Small samples of the diffusion layer were obtained for the X-ray diffraction analyses by cutting and breaking apart the unmounted half of the diffusion couple after sectioning. The mounted specimens were used for microprobe scans across the diffusion zones to determine relative percentages of tungsten and aluminum. No attempt was made to identify the constituents of diffusion layers between SAP 895 and Type 304 SS.

TABLE 1  
 SUMMARY OF DIFFUSION DATA FOR SAP 895 - W  
 AND SAP 895 - TYPE 304 SS SYSTEMS

Nominal Annealing Temperature (°F)	Diffusion Couple No.	Corrected Annealing Temperature (°F)	Annealing Time (hr)	SAP-W Diffusion Layer Maximum Thickness (mils)		SAP - 304 SS Diffusion Layer Thickness (mils)
				Average	Range	
1125	36	1124	4	1.47	1.35 - 1.70	2.4
1100	35	1099	8	2.04	1.87 - 2.19	2.0
1075	34	1075	4	0.421	0.290 - 0.554	*
	15	1068	8	1.07	0.91 - 1.19	2.0
	16	1072	16	2.68	2.49 - 2.79	2.8
	20	1076	24	3.01	2.86 - 3.15	3.2
1050	17	1046	16	0.900	0.74 - 1.16	*
	31	1048	24	1.19	1.04 - 1.29	2.4
	18	1047	30	1.34	1.13 - 1.52	2.8
	19	1048	48	3.00	2.91 - 3.11	3.6
1025	33	1022	24	0.475	0.322 - 0.554	2.6
	23	1032	48	1.26	1.07 - 1.49	2.8
	37	1024	96	2.41	2.03 - 2.99	5.2
1000	25	997	96	0.787	0.540 - 1.05	3.6
	7	996	170	2.74	2.57 - 2.93	4.4
	8	998	290	5.76	5.40 - 6.12	6.0
980	27	979	429	1.66	1.39 - 2.06	6.4
	26	981	768	3.01	2.82 - 3.27	7.6
	38	983	1177	4.43	4.22 - 4.70	12.0
960	39	958	455	0.725	0.570 - 0.936	4.8
	47	960	745	1.17	1.13 - 1.24	6.4
	48	959	1028	1.71	1.58 - 1.86	8.0
	11	959	1770	4.98	4.01 - 5.93	10.0
940	9	939	2009	0.114	0.096 - 0.129	9.6
	40	938	2761	3.20	†	11.2
	10	941	4841	8.70	†	15.6
920	41	919	2756	2.0	†	10.0
	28	920	3117	0.135	0.106 - 0.152	10.4

\*Cracking too extensive for measurements.

†Single maximum value measured.

## III. RESULTS

## A. GROWTH AND CHARACTERIZATION OF DIFFUSION LAYERS

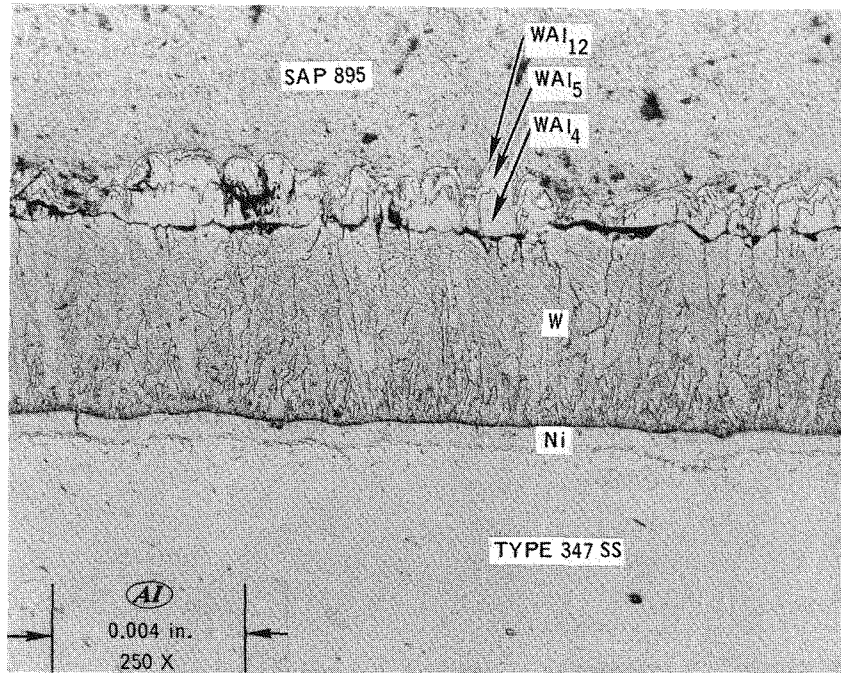
The growth of diffusion layers between SAP 895 and tungsten and between SAP 895 and Type 304 SS is summarized in Table 1. The extremes of annealing times ranged from 4 hr at 1125°F to 4841 hr at 940°F.

Typical microstructures of aluminum-tungsten diffusion zones for various annealing treatments are shown in Figures 5, 6, and 7. The tungsten coating resulting from chemical vapor deposition has a characteristic dendritic structure. The diffusion zones show a phase having "teeth" which coincide with the grain boundaries of the tungsten. The growth of the total diffusion layers is principally toward the SAP side of the original interface and is very irregular in thickness. The evidence of prior grain boundaries of tungsten in the diffusion layer microstructures indicates that the direction of diffusion is toward the tungsten (i. e. , aluminum diffuses into the tungsten).

The phase diagram\* for the aluminum-tungsten system shows the existence of three stable intermetallic phases corresponding to formulas of  $WAl_{12}$ ,  $WAl_5$ , and  $WAl_4$ . Two of these intermetallic compounds,  $WAl_{12}$  and  $WAl_5$ , were identified by X-ray powder diffraction patterns in couples annealed at 960° and 1000°F. Insufficient reference powder data were available from the literature to identify the presence of  $WAl_4$ . Electron microprobe scans across the diffusion layers of several couples annealed in the 940° to 1025°F temperature range revealed that the three layers in the typical microstructure had relative percentages of Al and W corresponding to the formulas  $WAl_{12}$ ,  $WAl_5$ , and  $WAl_4$ . As shown in Figure 5, the very thin layer (~0.1 mil thick) adjacent to the SAP is  $WAl_{12}$ . The phase associated with tungsten grain boundary penetration is  $WAl_5$  and the remaining phase is  $WAl_4$ .

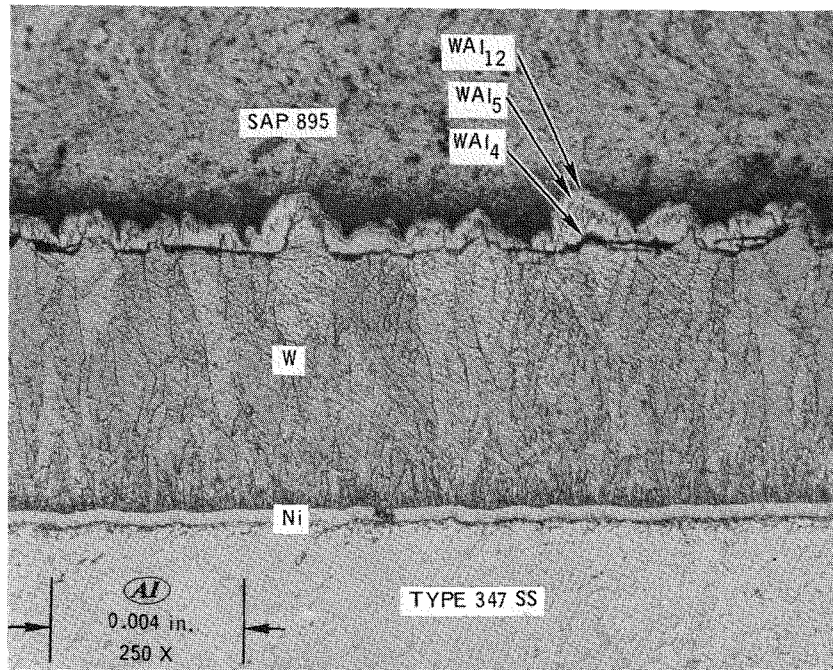
The formation of the initial  $WAl_{12}$  phase occurs at a relatively slow rate and is highly temperature dependent. The thickness of this layer appears to reach a maximum of about 0.1 to 0.2 mils over the entire range of annealing temperatures (920° to 1125°F). The  $WAl_5$  and  $WAl_4$  phases do not appear until after

\*M. Hansen, Constitution of Binary Alloys (2nd Ed. ; McGraw-Hill Book Company, Inc. , New York, 1958)



7567-1-1

Figure 5. Microstructure of Couple No. 26 Showing Diffusion Zone Between SAP 895 and Tungsten After 768 hr at 980°F



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Figure 6. Microstructure of Couple No. 47 Showing Diffusion Zone Between SAP 895 and Tungsten After 745 hr at 960°F

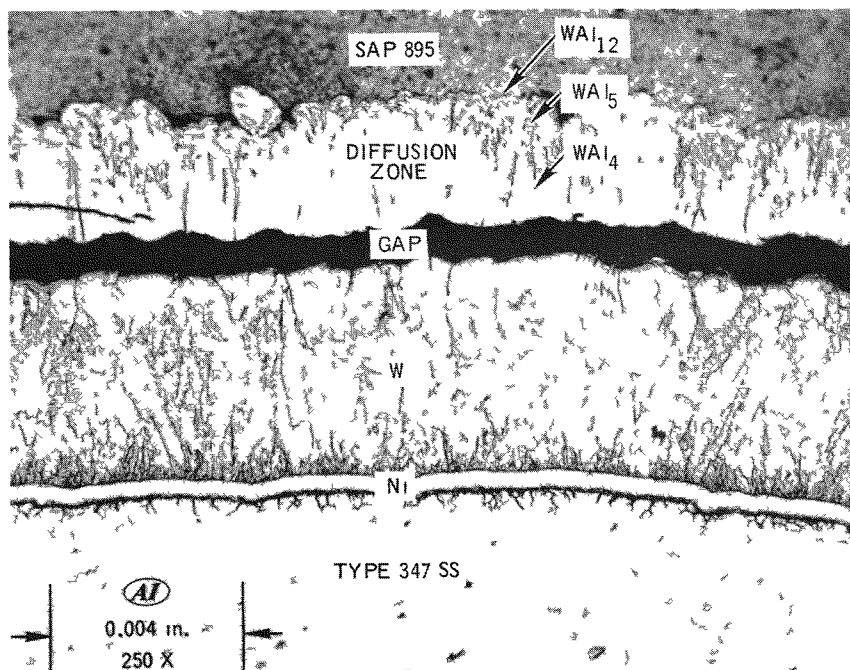


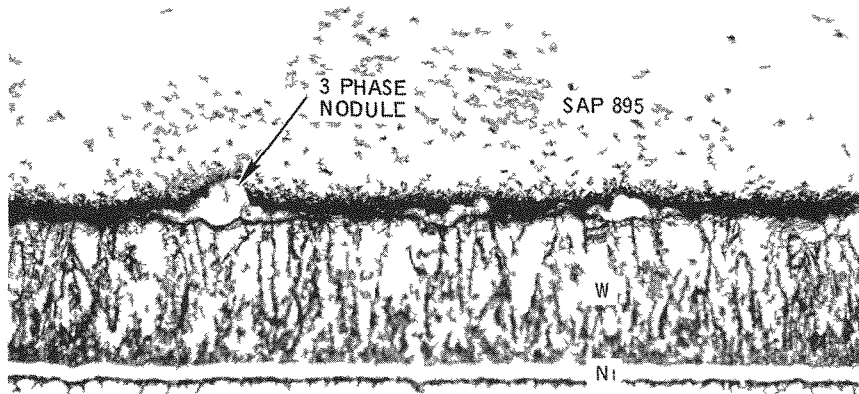
Figure 7. Microstructure of Couple No. 20 Showing Diffusion Zone Between SAP 895 and Tungsten After 24 hr at 1075°F

the initial  $WA1_{12}$  layer formation. These phases first appear as nodules, as shown in Figure 8, and their growth rate is considerably faster than for the initial  $WA1_{12}$  layer formation. Figure 9 shows a couple (no. 10) annealed 4841 hr at 940°F where the initial  $WA1_{12}$  layer is still forming in one location while in another location the three-phase diffusion structure is quite extensive. This irregularity was observed to be increasingly severe with decreasing annealing temperatures.

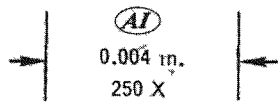
Aluminum penetrated the tungsten coating in many couples and attacked the underlying nickel coating, as shown in Figure 10 (couple no. 41). It was concluded that the penetration was due to defects in the tungsten coating (such as cracks or porosity) which allowed aluminum to migrate through the tungsten layer by surface diffusion. Defects of this type are associated with process control in the chemical vapor deposition of tungsten. The reaction product was identified by X-ray diffraction as  $Al_3Ni_2$ .

The photomicrograph in Figure 11 is typical of the diffusion layers formed between SAP 895 and Type 304 SS. The diffusion zone thicknesses were quite uniform compared to the SAP 895 - W diffusion zones.

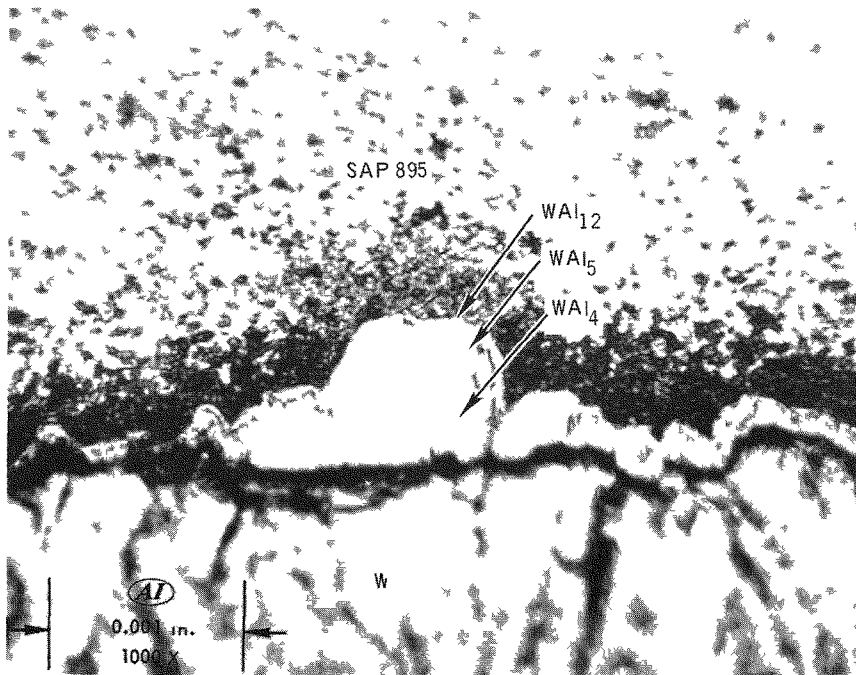
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TYPE 347 SS

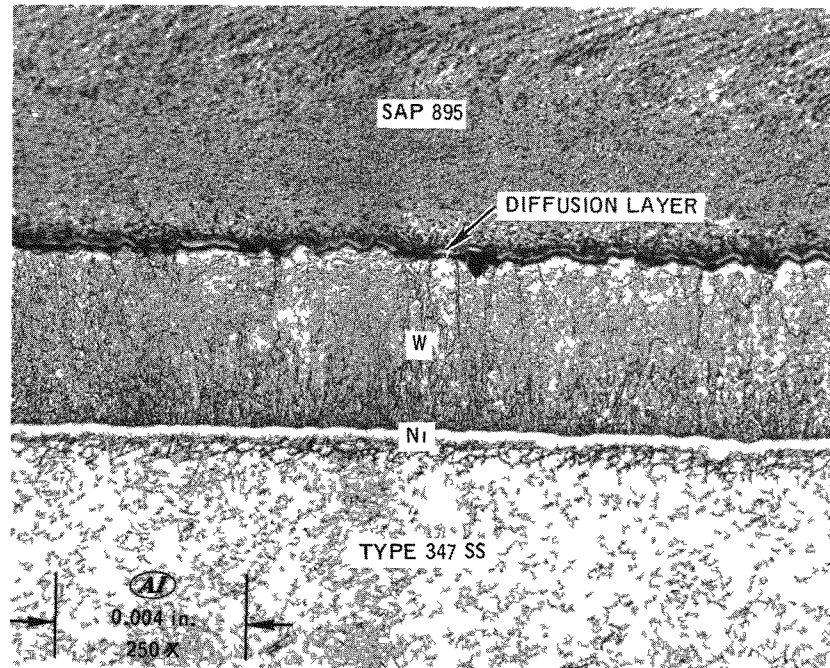


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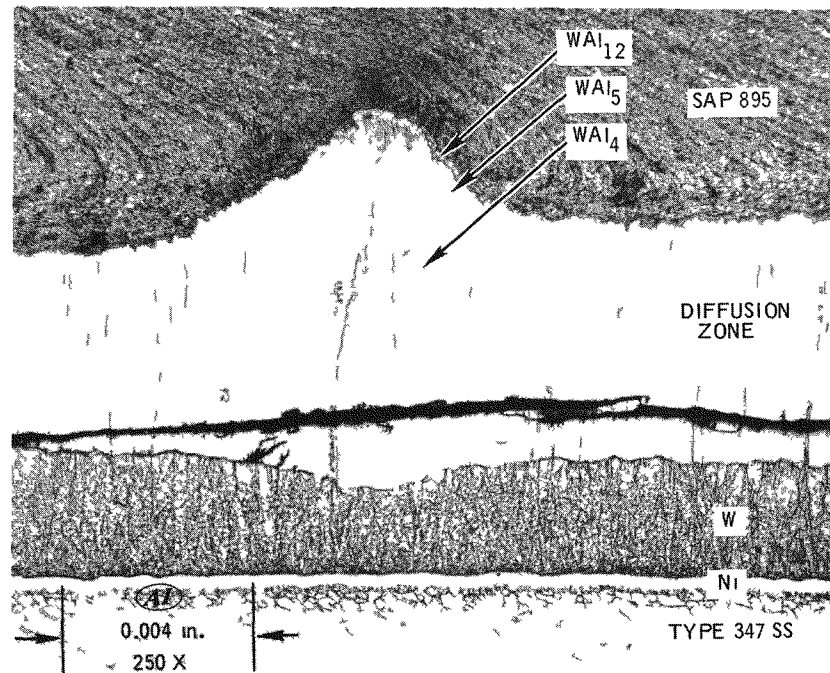
7830 2 4

Figure 8. Microstructure of Couple No. 25 Showing Formation of Nodules After 96 hr at 1000°F



7772 1 2

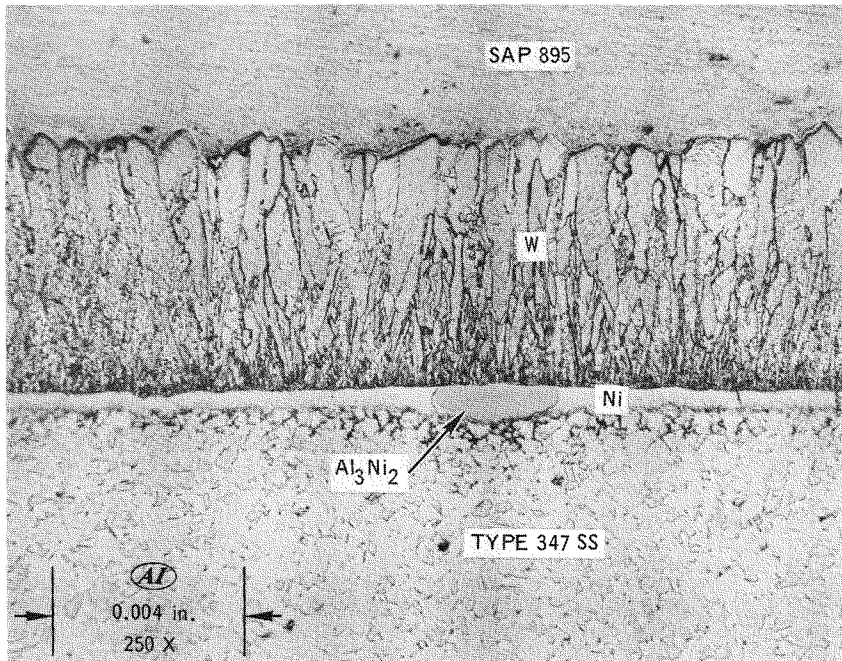
a. Area of Couple Showing Initial Diffusion Layer Formation



7772 1 1

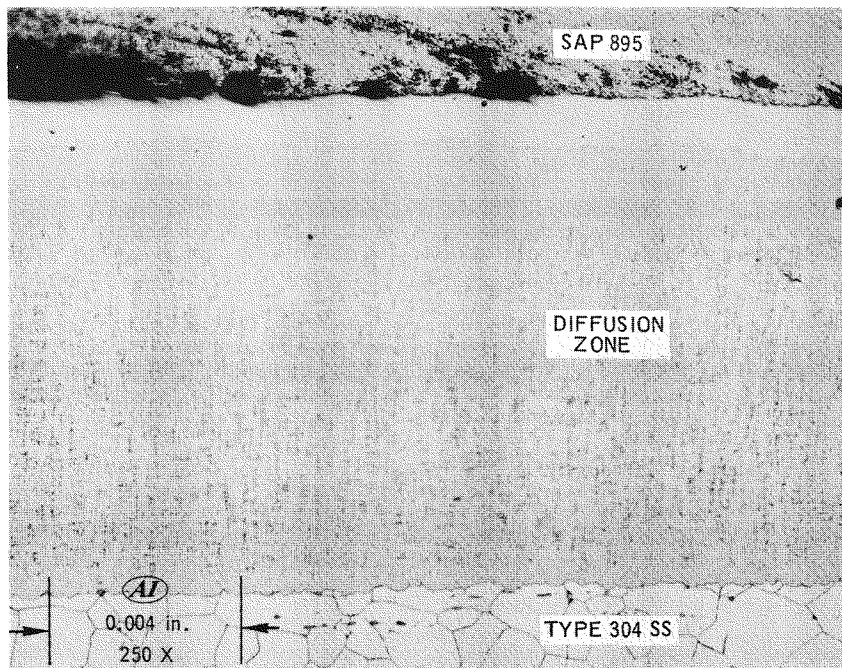
b. Area of Couple Showing Extensive Three-Phase Diffusion Layer Formation

Figure 9. Microstructures of Diffusion Zones in Couple No. 10 After 4841 hr at 940°F



7772-5-1

Figure 10. Microstructure of Couple No. 41 Showing Penetration of Defective Tungsten Coating by Aluminum and Resultant Reaction with Nickel After 2756 hr at 920°F



7830-3-2

Figure 11. Microstructure of Couple No. 38 Showing Diffusion Zone Between SAP 895 and Type 304 SS After 1177 hr at 980°F



## B. DATA ANALYSIS

The analysis of data was directed toward defining the relationship between the total diffusion layer thickness,  $x$  (in.), the annealing time,  $t$  (hr), and the annealing temperature,  $T$  ( $^{\circ}$ K). At constant temperature, a relationship was sought of the type

$$x^n = kt \quad \dots (3)$$

In the ideal case, the value of  $n$  is constant and equal to 2, giving a parabolic growth rate of the diffusion layer thickness. The temperature dependency of  $k$  is given as

$$k = k_o \exp (-Q/RT) \quad \dots (4)$$

1. SAP 895 - Tungsten Data

The SAP 895 - W diffusion data were examined at each of the nominal annealing temperatures in an effort to define the value of  $n$  and to determine if it could be considered constant. Poor correlation was found with a parabolic diffusion rate relationship. Log  $x$  vs log  $t$  plots at each of the nominal annealing temperatures generally gave a value of the slope  $n \approx 0.8$  to 1.0. Because of the high temperature dependency of the diffusion rate, even small deviations from the assumed constant nominal annealing temperature can lead to error in the determination of  $n$ . Therefore, a second approach was to select a value of  $n$  which would give the best straight line fit in a plot of log  $k$  vs  $1/T$  (Arrhenius plot), using corrected temperature values. It was found that if  $n$  is considered constant and equal to 0.90, there is good agreement of the data points for three-phase layer thicknesses over the entire annealing temperature range in an Arrhenius plot. Diffusion couples showing only the initial  $WA_{12}$  layer were omitted because they fell well below the Arrhenius plot. The calculated values of  $k$  for  $n = 0.90$  are summarized in Table 2. The Arrhenius plot of the data is shown in Figure 12, where the straight line fit was obtained by the method of least squares. The analysis yielded calculated values of

$$Q = 82,500 \text{ cal/mol} \quad \text{and} \quad k_o = 3.27 \times 10^{17} \text{ in.}^{0.90}/\text{hr.}$$

TABLE 2  
ANALYSIS OF DIFFUSION DATA FOR SAP 895 - W AND  
SAP 895 - TYPE 304 SS SYSTEMS

Nominal Annealing Temperature (°F)	Diffusion Couple No.	t (hr)	1000/T (1/°K)	SAP - W	SAP - 304 SS
				$K \cdot 10^6$ (in. <sup>0.90</sup> /hr)	$K \cdot 10^8$ (in. <sup>2</sup> /hr)
1125	36	4	1.1364	705	144
1100	35	8	1.1547	474	50.0
1075	34	4	1.1737	229	-
	15	8	1.1779	265	50.0
	16	16	1.1751	303	49.0
	20	24	1.1723	223	42.7
1050	17	16	1.1962	113	-
	31	24	1.1940	97.1	24.0
	18	30	1.1947	85.3	26.1
	19	48	1.1940	112	27.0
1025	33	24	1.2151	42.5	28.2
	23	48	1.2063	51.3	16.3
	37	96	1.2136	45.8	28.2
1000	25	96	1.2353	16.8	13.5
	7	170	1.2361	29.1	11.4
	8	290	1.2346	33.3	12.4
980	27	429	1.2516	7.34	9.55
	26	768	1.2500	6.97	7.52
	38	1177	1.2484	6.47	12.2
960	39	455	1.2706	3.27	5.06
	47	745	1.2674	3.09	5.50
	48	1028	1.2690	3.14	6.23
	11	1770	1.2690	4.79	5.65
940	9	2009	1.2870	-	4.59
	40	2761	1.2887	2.06	4.54
	10	4841	1.2853	2.88	5.03
920	41	2756	1.3055	1.35	3.63
	28	3117	1.3055	-	3.47

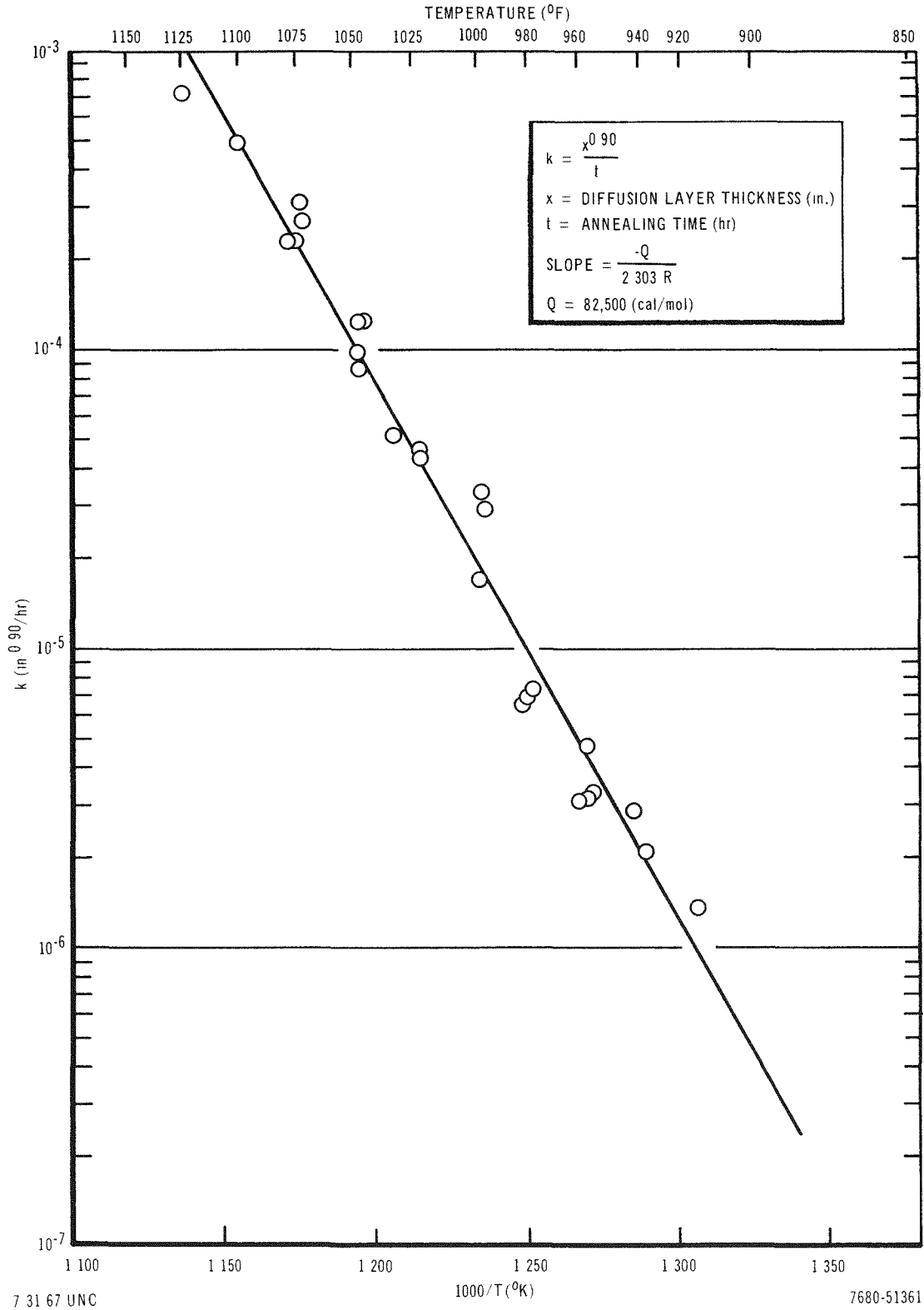


Figure 12. Arrhenius Plot of SAP 895 - Tungsten Diffusion Data

2. SAP 895 - Type 304 SS Data

The data for SAP 895 - Type 304 SS diffusion have shown a parabolic growth rate of the diffusion layer thickness  $x$  at constant (nominal) annealing temperatures. Table 2 summarizes the calculated values of  $k$  for  $n = 2$ . A straight line fit based on a least squares analysis of the data is shown in the Arrhenius plot of Figure 13. The analysis yielded calculated values of

$$Q = 40,800 \text{ cal/mol} \quad \text{and} \quad k_0 = 1.35 \times 10^4 \text{ in.}^2/\text{hr} .$$

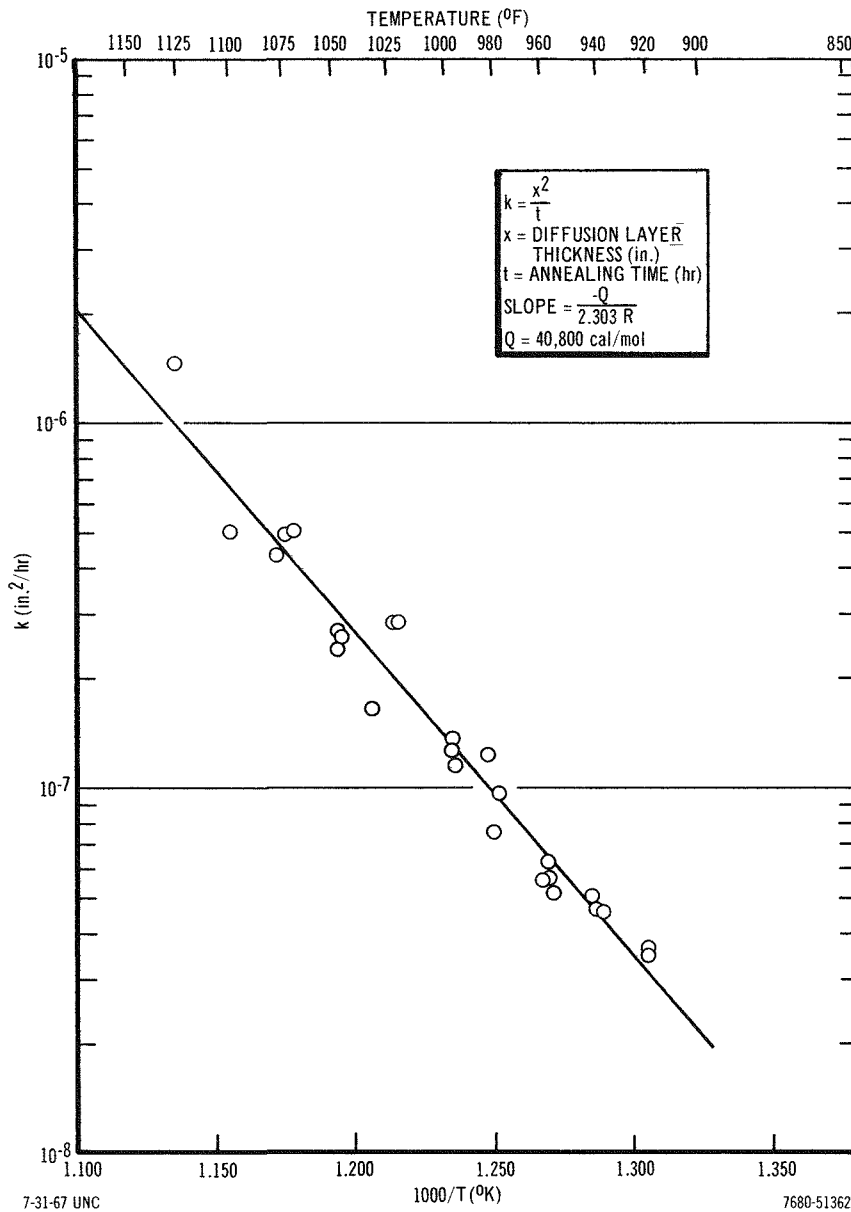


Figure 13. Arrhenius Plot of SAP 895 - Type 304 SS Diffusion Data

## IV. DISCUSSION

The diffusion of aluminum from SAP 895 into chemically vapor deposited tungsten appears to be a complex, structure-sensitive process involving both lattice and grain boundary diffusion. The data suggest that the initial layer formation of the  $WAl_{12}$  phase is rate-controlling by virtue of its apparent constant (steady-state) thickness and by the near-linear growth rate of the combined  $WAl_5$  and  $WAl_4$  phases. The relatively high growth rate of the latter two phases, compared to the initial layer of  $WAl_{12}$ , suggests that the tungsten grain boundaries have become weakened by the initial layer formation. The direction of diffusion in forming the initial  $WAl_{12}$  layer is not known. However, in the latter stages of the diffusion process, the evidence of prior tungsten grain boundaries associated with the  $WAl_5$  phase indicates that the aluminum diffuses into the tungsten.

The annealing times required for the initial layer formation cannot be accurately defined from the data obtained. Rough limits can be assessed by knowing that the time for initial layer formation is less than the minimum time at a given temperature where the three-phase layer was observed (e.g., <96 hr at 1000°F, >2009 hr and <2761 hr at 940°F).

Restating the objective of this study, it was desired to know the extent of diffusion that could be expected between SAP 895 and chemically vapor deposited tungsten after 30 years at 765°F. Extrapolation of the straight line plot of Figure 12 to 680°K (= 765°F) leads to a value of  $k = 1.025 \times 10^{-9} \text{ in.}^{0.90}/\text{hr}$ . After 30 years ( $t = 2.628 \times 10^5 \text{ hr}$ ) at 765°F the diffusion layer thickness  $x$  equals 0.000108 in. This value of the thickness corresponds in magnitude to the initial single-phase layer formation.

SAP-steel transition joints have in the past\* been annealed at 950°F for various periods of time to determine the effect of diffusion on bond strength. Using Equation 2 and the determined activation energy value of 82,500 cal/mol, the required time at 950°F to simulate 30-year diffusion at 765°F is about 86 hr. Transition joints annealed at 950°F for up to 1000 hr have shown only the initial

\*Ibid., AI-CE-46

diffusion layer formation ( $\sim 0.1$  mil) by metallographic examination. After 504 hr annealing time the tensile strength was typical of as-bonded joints. An annealing time of 1000 hr at  $950^{\circ}\text{F}$  resulted in a loss of about 20% in tensile strength. From these observations, it appears that the initial diffusion layer formation does not grossly affect the strength of the transition joint. Also, the time required to form the initial diffusion layer in transition joints at  $950^{\circ}\text{F}$  indicates that the expected diffusion layer thickness calculated for 30 years at  $765^{\circ}\text{F}$  is conservative.

The effectiveness of the tungsten diffusion barrier is demonstrated by comparing the SAP 895 - W data with the SAP 895 - Type 304 SS data. A SAP 895 - Type 304 SS transition joint having no diffusion barrier would develop a diffusion layer thickness of 0.0164 in. after 30 years at  $765^{\circ}\text{F}$  compared to a thickness of only 0.000108 in. with a tungsten barrier.

## V. CONCLUSIONS

The results of this study have shown that the diffusion of aluminum from SAP 895 into chemically vapor deposited tungsten is a complex process involving both lattice and grain boundary penetration. The structure of the diffusion zone is characterized by a thin layer ( $\sim 0.1$  mil) of  $WAl_{12}$  phase adjacent to the SAP, a  $WAl_5$  phase which penetrates the W grain boundaries, and a  $WAl_4$  phase. The  $WAl_{12}$  phase forms before initiation and growth of the other two phases and is believed to be rate-controlling. The growth rate of the total diffusion layer thickness  $x$  (in.) with respect to time  $t$  (hr) in the temperature range studied ( $920^\circ$  to  $1125^\circ$  F) may be described by the equation

$$\frac{x^{0.90}}{t} = 3.27 \times 10^{17} \exp\left(\frac{-82,500}{RT}\right)$$

The growth rate of the diffusion layer thickness between SAP 895 and Type 304 SS was described by the equation

$$\frac{x^2}{t} = 1.35 \times 10^4 \exp\left(\frac{-40,800}{RT}\right)$$

It was concluded that the total maximum diffusion layer thickness that could be expected in SAP-steel transition joints, utilizing a W diffusion barrier, after 30 years at  $765^\circ$  F is about 0.000108 in. The diffusion would probably involve only the initial  $WAl_{12}$  layer formation, which has been shown to be generally nondetrimental to the tensile strength of transition joints in accelerated tests.

The effectiveness of tungsten as a diffusion barrier was demonstrated by comparing diffusion data of SAP 895 - W with SAP 895 - Type 304 SS. The resultant diffusion layer thickness without the barrier would be greater by more than two orders of magnitude in 30 years at  $765^\circ$  F.