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FUNDAMENTALS OF DIFFUSIONAL
BONDING — II

Second Annual Progress Report, June 30, 1956
to June 30, 1957

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L. S. Castleman

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Research Laboratories
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ABSTRACT

The study of the kinetics of layer growth in the aluminum-nickel system and the effect thereon of applied pressure has been continued. Additional information has been obtained about the growth of the beta phase, and a method was developed whereby the kinetics of growth of the beta phase could be investigated. The implications of Vasileff's equations which provide the solution for interdiffusion in a three-phase two-component system have been examined semi-analytically, and quantitative insight has been gained into the kinetics of interface movement. Finally, an investigation of the kinetics of interface movement and layer growth has been begun on systems which have less complicated phase equilibrium relationships than those existing in the aluminum-nickel system.

I. INTRODUCTION

Investigations of bond formation in the cladding of fuel elements by pressure welding have shown that the size and nature of the diffusion zone are significantly affected by variations in applied pressure^(1,2,3,4,5). Increasing pressure alters the thicknesses of intermediate phase layers in the diffusion zone and changes the strength of the bond. Since this effect may be important from both a practical and a theoretical point of view, a study was started in December 1954 to investigate the phenomenon in detail. The first phase of the investigation consisted of a literature survey of those phenomena considered to have a bearing on the problem: (1) the kinetics of intermediate phase formation, and (2) the effect of stress on diffusion rates. The findings were summarized briefly in the last progress report⁽⁶⁾. As a result of the literature survey, the aluminum-nickel alloy system was chosen as the first to be investigated. The following plan of attack was adopted:

- (1) The layers forming during diffusion bonding under atmospheric pressure and at higher pressures were to be identified.
- (2) The time law (or laws) governing the rate of growth of the intermetallic layers was to be determined for diffusion runs under atmospheric and higher pressures.
- (3) If, as appeared likely, pressure exerted its effect on layer growth by altering the diffusional mechanisms, measurements were to be made of the diffusion coefficients in the individual phases of the Al-Ni alloy system, and the influence of pressure on diffusion rates was to be determined.

The results of the work prior to June 30, 1956 have been previously discussed in detail⁽⁶⁾. The conclusions reached may be summarized as follows:

- (1) Diffusional bonding in the Al-Ni alloy system in the temperature range 400° to 625°C occurs by the formation and growth of the Ni_2Al_3 (gamma) and the NiAl_3 (beta) layers; other phases are either not present or occur in sub-microscopically thin layers.
- (2) The growth of the gamma layer in the temperature range 400° to 625°C is controlled by volume diffusion in the gamma phase, once a minimum layer thickness of the order 0.025 to 0.030 mm is reached. Prior to this, the layer grows more rapidly than volume diffusion considerations would predict.

- (3) Tentatively, the chemical diffusivity in the gamma phase has been calculated to be of the order of 0.7×10^{-10} cm²/sec at 600°C; the heat of activation is in the vicinity of 32,000 cal/mol over the temperature range 400° to 625°C.
- (4) The beta phase grows at a much less rapid rate than does the gamma phase; also, the growth of the former is relatively temperature insensitive in the range 400° to 625°C.
- (5) Pressure decreases the rate of growth of the gamma layer; tentatively, it appears that one effect of pressure is to lower the chemical diffusivity in the gamma phase; it is estimated that a pressure of five tsi lowers the diffusion coefficient by 33% at 600°C.

During the present reporting period, additional data relating to the growth kinetics of the gamma phase under elevated pressure have been obtained; a more careful search has been made for the presence of the NiAl (delta) and Ni₃Al (epsilon) "missing" phases; a method has been devised to determine the growth kinetics of the beta phase; attempts have been made to produce incremental diffusion couples of gamma-phase material; an apparatus has been built to obtain concentration-penetration curves in thin inter-metallic phase layers by means of an x-ray absorption technique; and finally, the study of the kinetics of intermediate phase layer growth has been extended to other two-component systems such as the silver-copper, antimony-tin, and antimony-aluminum systems, in which the phase equilibria are considerably simpler than those in the aluminum-nickel system. The progress made to date is reviewed in some detail in the succeeding sections of this report and in three other papers^(7,8,9) which have been or which are being submitted for publication.

II. EXPERIMENTAL PROCEDURE

In the previous progress report⁽⁶⁾, the preparation of the aluminum-nickel diffusion couples, the diffusional bonding runs, and the method of layer thickness measurement were described in detail. During the past year, considerable effort has been expended in an attempt to measure directly the chemical diffusion coefficient in the gamma phase. The first approach involved the diffusion of an aluminum-nickel diffusion couple for 336 hours at 600°C at atmospheric pressure so that a gamma-phase layer at least 0.3 mm thick would form. Subsequently, one-mil layers were removed from the region of the couple in which the diffusion zone was located. It was found that the strains which had developed during the diffusional anneal had produced a pronounced curvature in the gamma layer. The resultant error was so serious that it was impossible to obtain a valid concentration-penetration curve. In view of this failure, two concurrent

efforts were begun, as follows: (1) the preparation of incremental couples in the gamma phase; and (2) the application of an x-ray absorption technique to the determination of concentration-penetration curves in an aluminum-nickel diffusion couple (in which sectioning and chemical analysis would be eliminated and the error resulting from the gamma-phase curvature would be minimized). The developments stemming from this work and from other phases of the over-all program are reviewed below:

A. Preparation of Gamma-Phase Incremental Couples

Several techniques were explored in an effort to prepare the gamma phase in bulk form such that the material would be suitable for the preparation of diffusion couples. An attempt was made to prepare the alloy in which a charge of aluminum and nickel of the proper composition was heated into the liquid region (1650°C) for two hours under an argon atmosphere and then solidified. A porous, brittle casting of non-uniform composition was obtained with the denser nickel-rich phases being segregated at the bottom of the ingot. The results showed such little promise that the melting technique was abandoned. Following this, several powder metallurgical techniques were tried, as follows:

- (1) Aluminum and nickel powders were sintered in an open boat under a hydrogen atmosphere to a friable mass for 16 hours at 1100°C . Subsequently, an attempt was made to produce a dense body by pulverization of the product, the cold compacting of the powder, and the sintering of the given compact at 1100°C , but the results were unsuccessful.
- (2) Nickel and aluminum powders (-325 mesh) of the proper stoichiometry were hot pressed in the temperature range 900° to 1050°C for one-half hour in graphite dies under a pressure of two tsi. The resulting structure appeared to be homogeneous; however, considerable porosity was present, and it did not appear that the specimens could be used for diffusion studies.

Finally, the following technique was evolved which produced satisfactory gamma-phase samples in bulk form:

- (a) Aluminum discs and nickel discs, 0.0045 and 0.0020" thick, respectively, are stacked alternately.
- (b) The assembly is interdiffused under a pressure of five tsi for 48 hours at 625°C (the upper temperature limit is the aluminum-nickel eutectic temperature of 640°C).
- (c) The resulting disc is homogenized for 24 hours at 825°C

to remove by diffusion any traces of the beta phase (the beta phase decomposes peritectically into the gamma phase and liquid at 854°C).

- (d) The homogenization treatment is completed at 950°C for 24 hours.

Care must be exercised in the selection of the proper thicknesses of the aluminum and nickel; otherwise, two phase regions appear in the microstructure, as shown by an exaggerated case in Figs. 1(a) and 1(b). A properly homogenized sample is shown in Fig. 2; the porous region is located in the region in which aluminum had originally been present in the stack and is a characteristic defect in the material prepared in the manner outlined.

Once gamma-phase discs were available, attempts were made to prepare incremental sandwich couples of gamma phase containing 59 w/o nickel vs. gamma phase containing 55 w/o nickel vs. gamma phase containing 59 w/o nickel, by the application of a pressure of one tsi at 600°C to the unbonded samples, for 48 hours. No bond was formed, since there was insufficient plastic deformation of the brittle intermetallic compound at the couple interfaces. Other couples were prepared by the evaporation of thin layers of aluminum onto all mating interfaces. It was hoped that the diffusion of aluminum into the adjacent surfaces would facilitate bonding. A test run was made at 600°C under a pressure of one tsi. Upon completion of the run, it was found that complete bonding had not occurred. The thickness of the aluminum at the interface was increased to 0.0001" and the run was repeated. This time, one of the interfaces in the sandwich couple appeared to be bonded, but the other couple was not completely bonded and failed. Recently, a gamma-phase incremental sandwich couple was bonded under a pressure of two tsi at 950°C for two hours in a graphite die. The specimen surfaces appeared cracked, but the bond was apparently strong. It appears, therefore, that it may be possible to prepare suitable couples by bonding them under pressure in graphite dies at elevated temperatures.

B. X-ray Absorption Apparatus for Diffusion Studies

If a two-component single-phase alloy made up of the elements A and B is irradiated by a monochromatic x-ray beam, the intensity of the transmitted beam is given by the following expression:

$$I_T = I_0 e^{-[w_A (\mu/\rho)_A + w_B (\mu/\rho)_B] \rho_{(A+B)} t}$$

where W_A and W_B are the weight fractions of A and B, respectively, (μ/ρ) is the mass absorption coefficient, $\rho_{(A+B)}$ is the density of the alloy, and t is the thickness of the sample. The relationship above can be utilized in a device which permits one to obtain concentration-penetration data in diffusion couples quite readily. A device of this sort, essentially similar to the one developed by Ogilvie⁽¹⁰⁾ and modified by Hilliard⁽¹¹⁾, has been constructed, a photograph of which is shown in Fig. 3. The apparatus consists of the following items: (1) a narrow slit, about 0.0005" wide, which is easily adjustable, and a second adjustable slit to define the beam height; (2) a micrometer head, which reads to 0.0001 " and which translates the sample holder across the narrow slit; (3) a sample holder; and (4) a silicon single crystal cut parallel to the (111) plane which furnishes the monochromatic radiation needed. (The second order (111) reflection is forbidden, so that the $\lambda/2$ contribution is eliminated.)

One of the difficulties involved is alignment of the diffusion couple with the slit. A photographic plate is fastened to one of the sample holders, and an image of the slit is recorded. Thereafter, the alignment jig is used under a microscope, so that the couple can be aligned with the recorded slit image. Once the sample is aligned on the apparatus, the sample is translated by means of a motor which is synchronized with the chart recorder. A scintillation counter is used for intensity measurements, since it eliminates the need of calibration foils. A study of a diffusion couple is made in a relatively short time if the synchronous motor traverse is used. If maximum accuracy is desired, the sample is traversed manually, very small displacements are employed, and counting techniques are used.

At the present time, an attempt is being made to prepare a suitable specimen for analysis. The maximum thickness of a specimen is currently being determined as a function of the energy of the x-ray beam, so that the proper x-ray targets can be selected.

C. Preparation of Other Diffusion Couples

A number of antimony-tin, aluminum-antimony, and copper-silver diffusion couples have been prepared for exploratory diffusion runs. Commercially pure tin was rolled into strip 0.015" thick, and one-half inch diameter discs were punched out and electrolytically polished. Commercially pure antimony (virgin metal) was melted in a one-half inch diameter evacuated Vycor capsule at 750°C, slow-cooled, and then annealed at 400°C for 24 hours. Sections one-eighth inch thick were cut off, ground parallel,

and polished by hand on #4-0 emery paper. Diffusion couples were prepared as follows: Stacks of alternately spaced tin and antimony discs were built up and inserted into small screw presses. The screws were tightened to the same tension with a torque wrench, and the assemblies were annealed in air at various temperatures. Aluminum-antimony couples were prepared from aluminum discs made in the manner described in the previous progress report⁽⁶⁾ and from antimony discs made in the manner described above. The couples were annealed in the hot pressing apparatus. The copper-silver diffusion couples were made from 0.004" discs which were punched out of commercially pure sheet and then electrolytically polished. The couples were bonded in the hot pressing apparatus at 600°C for one-quarter hour. Subsequently, they were sectioned longitudinally. One section of each was kept for control purposes, and the other section was diffusion annealed at the desired temperature and time in evacuated Vycor capsules.

III. EXPERIMENTAL RESULTS AND DISCUSSION

A. Aluminum-Nickel System

It was stated in the last annual progress report⁽⁶⁾ that diffusion bonding in the aluminum-nickel alloy system in the temperature range 400° to 625°C occurs by the formation and growth of the beta and gamma layers. Other phases are either not present or occur in submicroscopically thin layers. Contrary to this conclusion, it has been ascertained by the diffusion of gamma-nickel couples at temperatures of 800°, 900° and 1050°C that the "missing" delta and epsilon phases should be present in an aluminum-nickel couple diffused below the eutectic temperature as very thin but observable layers. This hypothesis has been confirmed by diffusion of an aluminum-nickel specimen for 340 hours at 600°C. Careful examination of the interface between the nickel and the gamma phase at a magnification of 1500X revealed the delta and epsilon layers in thicknesses of two microns or less. The experiments and their implications are discussed in detail by Castleman and Seigle^(7,9).

The layer growth data previously reported⁽⁶⁾ and obtained on aluminum-nickel diffusion couples diffused at atmospheric pressure and at a pressure of five tsi are listed in Table I. The additional data obtained during the past year on aluminum-nickel, aluminum-gamma phase, and gamma phase-nickel couples are listed in Table II. The kinetics of growth of the beta, gamma, delta and epsilon phases have been previously discussed in detail⁽⁹⁾. The pertinent conclusions reached are as follows:

1. Volume diffusion is the rate-controlling factor affecting the kinetics of growth of the gamma phase after a minimum thickness of 0.025 to 0.030 mm has been reached. It is estimated that the 600°C chemical diffusion coefficient is about 9.1×10^{-10} cm²/sec and that the heat of activation for diffusion is of the order of 31,000 cal/mol.
2. The rate-controlling factor affecting the kinetics of growth of the gamma phase in the early stages is not yet clear. Two possible explanations are advanced as follows: (a) An incubation period may exist prior to the nucleation of the gamma phase which affects the subsequent course of interdiffusion until the diffusion time becomes long in relation to the incubation time period; or (b) the initial boundary concentrations are not equilibrium concentrations and delay the course of interdiffusion until they approach the equilibrium values.
3. Applied pressure exerts its effect upon the growth kinetics of the gamma phase by decreasing the value of the chemical diffusion coefficient. It is estimated that the coefficient is reduced by 27% as the pressure increases from atmospheric to five tsi. The decrease in the diffusion coefficient is equivalent to an increase in the heat of activation of 470 cal/mol. Experimentally, pressure increased the heat of activation negligibly; but this is not surprising, since the estimated precision of the calculation is ± 1000 cal/mol.
4. Volume diffusion is the rate-controlling factor affecting the kinetics of growth of the beta phase after a minimum thickness of about 0.040 mm has been reached. It is estimated that the 600°C chemical diffusion coefficient is about 1.8×10^{-11} cm²/sec and that the heat of activation for diffusion is of the order of 27,000 cal/mol.
5. Tentatively, it seems that the effect of pressure appears to be much less marked upon the growth of the beta phase than upon that of the gamma phase.
6. Tentatively, it appears that volume diffusion also governs the growth kinetics of the delta and epsilon phases.

It will be noted that some of the statements made in the first progress report⁽⁶⁾ are at variance with those made above. First, the value of the chemical diffusion coefficient in the gamma phase

was estimated to be about 0.7×10^{-10} cm²/sec at 600°C, whereas the value stated above is 9.1×10^{-10} cm²/sec. The reason for the discrepancy is twofold: (1) An arithmetical error was made in the earlier calculation; and (2) it was assumed in the earlier calculation that the gamma phase existed in metastable equilibrium with the zeta nickel-rich solid solution. In view of the discovery that the delta and epsilon phases are actually present in aluminum-nickel couples diffused at 600°C, it is felt that it is reasonable for one to assume the existence of a gamma-delta interface and the corresponding interface concentrations in making the calculation. Second, the interpretation of the gamma phase growth during the transient stage has been changed. It has been pointed out by Castleman and Seigle⁽⁹⁾ that layer growth is proceeding at a faster rate than would be predicted by volume diffusion considerations, but that the total layer thickness is less than would have been obtained had volume diffusion been the rate-controlling factor at the outset. Many of the phenomena that could conceivably affect layer growth in the early stages (short circuiting diffusion paths, strain effects at interfaces, etc.) would lead to the reverse effects. Consequently, since the assumption of an incubation period for the gamma phase leads to results which are consistent with the observed curvature, it is felt that the question of delayed nucleation must be seriously considered. Finally, in the previous progress report, it had been tentatively concluded that the growth of the beta phase is relatively insensitive to temperature. This was based on measurements of the beta phase made in aluminum-nickel couples in which the growth of the beta phase is quite irregular. During the past year, it has been possible to determine the kinetics of growth of the beta phase much more precisely with the use of aluminum-gamma phase couples in which the beta phase is the only phase nucleating and growing at the aluminum-gamma phase interface.

B. Other Systems

A study has been begun of the kinetics of interdiffusion in systems such as antimony-tin, aluminum-antimony, and copper-silver. The phase equilibria in these systems are far simpler than those in the aluminum-nickel system. For example, there are no intermediate phases at all in the copper-silver system and only one interface boundary; in the aluminum-antimony system, one intermediate phase exists which has a highly restricted range of solubility; the tin-antimony system also contains only one intermediate phase, which has a moderate solubility range. The aluminum-nickel system by contrast has four intermetallic phases.

Obviously, it is far easier to gain quantitative insight into interdiffusional phenomena in two-component systems containing one or two discontinuities in concentration and in the value of the diffusion coefficient than in a system containing five interfaces. As a matter of fact, a mathematical solution exists for the two-interface, two-component interface problem (originally solved by Vasileff and Smoluchowski⁽¹²⁾). In spite of the highly restrictive assumptions made in the derivation of the solution, a semianalytical examination of the implications of the derived equations can broaden one's understanding of interface movement and layer growth⁽⁸⁾. The Vasileff equations should prove quite useful in rationalizing the kinetics of interface movement and layer growth in the three aforementioned systems.

The data which have been collected to date have been obtained for the antimony-tin systems and are listed in Table III. The layer thicknesses and diffusion times are plotted on logarithmic scales in Fig. 4. The slope of the curve obtained is about 0.45, an indication that one or both of the phase boundaries is being displaced at a rate somewhat less than that predicted by the simple parabolic law of intermediate phase layer growth. If one assumes a more ideal behavior, a first approximation of the chemical diffusion coefficient in the β' phase is possible by the use of the method of calculation described by Castleman and Seigle⁽⁹⁾ and a value of $D_{\beta'} \approx 3 \times 10^{-9}$ cm²/sec is obtained.

The preliminary work done on the copper-silver system indicates that the alpha-beta interface is moving into the copper-rich alpha phase as diffusion progresses. However, the rate of motion is so slow that some difficulty is anticipated in obtaining accurate quantitative measurement of the displacement. Also, in the aluminum-antimony system, the first couple diffused for 96 hours at 600°C has yielded a very peculiar intermetallic layer formation, which is shown in Fig. 5. The significance of the observation is of course not clear at the present time.

IV. FUTURE WORK

The study of the kinetics of layer growth in the aluminum-nickel system is to be continued with the following specific objectives in mind:

- (1) The accuracy of the method of obtaining diffusion coefficients in the gamma phase will be verified by determination of a diffusion coefficient either with the aid of an incremental gamma phase sandwich couple

or with the use of the x-ray absorption technique on aluminum-nickel diffusion couples.

- (2) The values of the boundary concentrations at the beta-gamma and the gamma-delta phase interfaces and their time variation (if any) will be ascertained by means of the x-ray absorption technique. Once these values are known, it should be possible to check the suggested explanation of the kinetics of growth of the gamma phase in the transient stage.
- (3) With the same purpose in mind, an attempt will be made to determine, with the aid of the electron microscope, the extent (if any) of the existence of an incubation period preceding the nucleation of the gamma phase at 400°C.
- (4) An attempt will be made to refine the kinetic studies by an examination of the intrinsic diffusivities of the aluminum and nickel atoms rather than of the chemical diffusion coefficients. Experiments will be conducted to determine suitable markers for the aluminum-nickel system and these will be used in a determination of the diffusivities.

The kinetics of layer growth and interface movement in systems having much less complicated phase equilibria than those existing in the aluminum-nickel system will be continued. The data will be used to determine the precision with which experimental data fit calculated values obtained by the application of known solutions of the diffusion equation (such as the Vasileff equations⁽¹²⁾) to multi-phase diffusion in two-component systems.

A study will be made of the kinetics of layer growth in the uranium-aluminum system in the temperature range 480° to 620°C and the effects of pressure thereon. The purpose of the study will be to provide an independent verification of the surprising and highly interesting results reported by LeClaire and Bear⁽⁵⁾.

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TABLE I

SUMMARY OF LAYER THICKNESS DATA IN THE ALUMINUM-NICKEL SYSTEM

Number	Diffusion		Ni ₂ Al ₃ Layer		Total Layers		NiAl ₃ Layer		
Samples	Al-Ni Inter-faces	Temp. (°C)	Time (min.)	Average Thickness (mm)	Standard Deviation (mm)	Average Thickness (mm)	Standard Deviation (mm)	Average Thickness (mm)	Standard Deviation (mm)
ATMOSPHERIC PRESSURE									
3	3	400	420	0.0001	-	0.0032	±0.0001	0.0030	±0.0002
3	6		1050	0.0010	±0.0002	0.0046	±0.0009	0.0055	±0.0004
1	2		1200	0.0007	±0.0002	0.0098	±0.0002	0.0071	-
1	2		2400	0.0046	±0.0001	0.0133	±0.0011	0.0091	-
2	3		20160	0.0126	±0.0017	0.0336	±0.0026	0.0221	±0.0002
3	4	450	200	0.0007	±0.0002	0.0051	±0.0003	0.0039	±0.0001
6	10		420	0.0028	±0.0007	0.0096	±0.0001	0.0066	±0.0004
1	1		960	0.0076	-	0.0127	-	0.0070	-
3	8		2880	0.0165	±0.0018	0.0268	±0.0005	0.0109	-
3	8		5760	0.0326	±0.0040	0.0453	±0.0007	0.0162	0.0023
2	2	500	15	0.0005	±0.0001	0.0038	±0.0004	0.0034	-
2	3		30	0.0014	±0.0001	0.0055	±0.0005	0.0045	±0.0003
2	7		60	0.0042	±0.0001	0.0090	±0.0001	0.0051	±0.0002
2	7		180	0.0100	±0.0001	0.0169	±0.0005	0.0067	±0.0001
2	5		420	0.0185	±0.0002	0.0258	±0.0005	0.0080	-
1	3		960	0.0334	±0.0001	0.0408	±0.0007	0.0080	±0.0004
1	2		2880	0.0555	±0.0032	0.0631	-	-	-
1	1		5760	0.0825	-	-	-	-	-
2	4	550	15	0.0034	±0.0001	0.0068	±0.0001	0.0039	±0.0001
2	3		30	0.0082	±0.0002	0.0125	±0.0008	0.0054	±0.0008
3	4		60	0.0128	±0.0002	0.0176	±0.0010	0.0046	-
2	2		180	0.0249	±0.0004	0.0303	±0.0001	0.0061	-
1	2		420	0.0398	-	0.0436	-	-	-
3	3		960	0.0635	±0.0016	0.0704	±0.0006	0.0085	±0.0011
3	4		5760	0.1494	±0.0019	0.1614	±0.0057	0.0186	±0.0003
2	4	600	15	0.0094	±0.0001	0.0130	±0.0002	0.0038	0.0000
3	9		30	0.0167	±0.0005	0.0214	±0.0005	0.0055	0.0006
2	5		60	0.0304	±0.0002	0.0348	±0.0012	0.0051	0.0003
2	8		180	0.0535	±0.0010	0.0583	±0.0006	0.0061	0.0001
2	8		420	0.0798	±0.0011	0.0874	±0.0015	0.0067	-
2	3		960	0.1219	±0.0011	0.1289	±0.0052	0.0103	-
1	2		5760	0.2907	±0.0007	0.3071	±0.0010	0.0171	-
2	4	625	15	0.0144	±0.0001	0.0178	±0.0005	0.0035	±0.0001
2	8		30	0.0259	±0.0000	0.1099	±0.0003	0.0044	±0.0002
2	7		60	0.0343	±0.0005	0.0377	±0.0008	0.0051	0.0002
2	6		180	0.0582	±0.0010	0.0637	±0.0022	0.0080	0.0014
2	4		420	0.0959	±0.0022	0.1044	±0.0057	0.0117	0.0002
3	7		960	0.1319	±0.0012	0.1383	±0.0119	0.0097	-
5 TSI PRESSURE									
1	2	450	960	0.0048	±0.0001	0.0107	±0.0000	0.0061	±0.0002
1	3	500	180	0.0103	±0.0002	0.0151	±0.0010	0.0045	±0.0001
1	2		420	0.0161	±0.0002	0.0219	±0.0001	0.0058	±0.0000
1	3		4320	0.0591	±0.0014	0.0717	±0.0019	0.0136	±0.0005
1	4		5760	0.0714	±0.0005	0.0843	±0.0004	0.0134	±0.0006
1	2	550	960	0.0480	±0.0002	0.0562	±0.0002	0.0082	±0.0001
1	2	585	960	0.0805	±0.0005	0.0900	±0.0002	0.0095	±0.0000
1	2	600	5760	0.2500	±0.0063	0.2561	±0.0001	0.0150	±0.0003

TABLE II

SUMMARY OF LAYER THICKNESS DATA FOR ALUMINUM VS. NICKEL

Number	Diffusion		Ni ₂ Al ₃ Layer		Total Layers		NiAl ₃ Layer		
Samples	Al-Ni Inter-faces	Temp. (°C)	Time (min.)	Average Thickness (mm)	Standard Deviation (mm)	Average Thickness (mm)	Standard Deviation (mm)	Average Thickness (mm)	Standard Deviation (mm)
<u>2.5 TSI PRESSURE</u>									
1	4	600	1560	0.1409	± 0.0008	0.1632	± 0.0008	0.0223	± 0.0002
<u>5 TSI PRESSURE</u>									
1	5	500	180	0.0070	± 0.0001	0.0140	± 0.0001	0.0070	± 0.0001
1	4	550	180	0.0203	± 0.0003	0.0276	± 0.0004	0.0073	± 0.0003
1	6		5760	0.1285	± 0.0022	0.1448	-	0.0162	± 0.0005
1	6	600	180	0.0437	± 0.0019	0.0516	± 0.0003	0.0080	± 0.0003
1	4		420	0.0725	± 0.0003	0.0808	± 0.0005	0.0083	± 0.0002
1	5		1080	0.0998	± 0.0006	0.1101	± 0.0008	0.0103	± 0.0002
<u>10 TSI PRESSURE</u>									
1	6	500	5760	0.0655	- 0.0001	0.0803	± 0.0006	0.0148	± 0.0004

NICKEL VS. GAMMA PHASE (Ni₂Al₃) CONTAINING 59 w/o NICKEL

Number	Total Sample Measurements	Diffusion		Layer		Total Layers		Layer	
		Temp. (°C)	Time (min.)	Average Thickness (mm)	Standard Deviation (mm)	Average Thickness (mm)	Standard Deviation (mm)	Average Thickness (mm)	Standard Deviation (mm)
1	21	800	1440	-	-	0.0069	± 0.0022	-	-
1	98	900	360	0.0083	± 0.0014	0.0178	± 0.0012	0.0095	± 0.0010
1	45		1440	0.0177	± 0.0010	0.0365	± 0.0016	0.0183	± 0.0013
1	57	1050	1440	0.0068	± 0.0008	0.1168	± 0.0017	0.1118	± 0.0020

ALUMINUM VS. GAMMA PHASE (Ni₂Al₃) CONTAINING 55 w/o NICKEL

<u>Number</u>		<u>Diffusion</u>		<u>NiAl₃ Layer</u>	
Sample	Total Measure- ments	Temp. (°C)	Time (min.)	Average Thickness (mm)	Standard Deviation (mm)
<u>1 TSI PRESSURE</u>					
1	77	500	5760	0.0259	± 0.0085
1	105	550	5760	0.0422	± 0.0084
1	88		14640	0.0736	± 0.0056
1	103	600	180	0.0146	± 0.0051
1	108		420	0.0167	± 0.0066
1	62		2880	0.0440	± 0.0034
1	45		5760	0.0656	± 0.0042
1	78		14640	0.1005	± 0.0085
1	100	625	1470	0.0483	± 0.0039
<u>5 TSI PRESSURE</u>					
1	119	600	5760	0.0666	± 0.0053

TABLE III

SUMMARY OF LAYER THICKNESS DATA IN THE ANTIMONY-TIN SYSTEM

<u>Number</u>		<u>Diffusion</u>		<u>Layer</u>	
Sample	Total Measure- ments	Temp. (°C)	Time (min.)	Average Thickness (mm)	Standard Deviation (mm)
1	76	220	270	0.0316	± 0.0051
1	15		450	0.0416	± 0.0037
1	30		1560	0.0708	± 0.0072
1	40		2880	0.1060	± 0.0027
1	24		4320	0.1089	± 0.0049
1	31		8640	0.1506	± 0.0054
1	82		18720	0.1837	± 0.0102

250X

Unetched

Fig. 1(a) Aluminum-nickel discs alternately stacked and diffused for 48 hours at 600° .

Fig. 1 (b) Same as above but photographed under polarized light.

250X

Unetched

Fig. 2 Aluminum-nickel discs alternately stacked and diffused for 116 hours at 600°C under 2.5 tsi pressure, followed by homogenization anneals at 800°C for 2 hours and at 950°C for 1 hour.

Fig. 3 X-ray absorption apparatus.

Fig. 4 Growth of β' phase in tin-antimony diffusion couples annealed at 220°C

500X

Unetched

Fig. 5 Aluminum-antimony diffusion couple annealed at 600°C for 96 hours under 1 tsi pressure.