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**SOLID STATE BONDING OF ALUMINUM
AND NICKEL**

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METALLURGY AND CERAMICS

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ABSTRACT

The solid state bonding of aluminum and nickel was studied as a function of temperature, pressure, and time at pressure. This was done by means of tensile tests and intermetallic penetration rates.

The initial results indicated that as the temperature for reaction was raised tensile strength increased, reached a maximum, declined and then leveled off. Plots of the log of penetration coefficient, P , versus the reciprocal of absolute temperature were straight lines.

As the pressure was increased for various isotherms the tensile strength showed: incipient bonding occurring at 400°C , increasing very sharply at 450° and 500°C ; at 550°C increasing slowly and to a lower maximum than for the 450° and 500°C temperatures and at 600°C , rising slowly from a relatively moderate maximum strength. The penetration coefficient was found to decrease as the pressure increased. This decrease was more rapid as the temperature of the reaction was raised. It was found that a zero penetration coefficient, i.e., no intermetallic formation occurred at 500°C if the pressure were high enough, namely, over 26 tons per square inch, tsi. Suppression of the alloy zone formation caused tensile strength to rise. Increased pressure did not apparently change the slope of the $\log P$ vs $1/T$ curves.

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ABSTRACT (cont'd)

Increased time at a pressure of 11 tsi for various isotherms caused tensile strength to rise to a maximum and then slowly decline from this value. The greatest tensile strength was found to occur at four minutes for all the isotherms used.

Metallographic investigation showed, as-polished, two intermetallic phases at the aluminum-nickel bond interface.

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I. INTRODUCTION

The techniques of bonding different metals to each other include brazing, welding, welding by application of a molten phase, and solid state welding without any molten phase present. This last technique, the pressing of metals either at room temperature or at some temperature below the melting point of the metals to be joined, seems to offer interesting possibilities for bonding purposes. However, this technique has not been completely investigated, and very little is known about the diffusion phenomena which occur during solid state welding. Some previous work on the effects of pressure on diffusion⁽¹⁾ was inconclusive.

This paper is concerned with an ~~investigation~~ regarding the effect that the processing variables, such as pressure, temperature and time at pressure, have in solid state bonding. The tests were made with aluminum and nickel and special emphasis was given to the strength of the bond between these two metals and to the intermetallic penetration rate during the bonding process.

It seems that the effect of pressure on the intermetallic penetration is of particular importance for practical purposes and this effect was therefore studied in detail.

(1) F.J. Radavich and R. Smoluchowski, "Influence of Pressure on Intermetallic Diffusion", Phys. Rev. 65, 62, & 248 (1944).

II. PROCEDURE

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A. Apparatus

The means of solid state bonding used for this study was that of the hot-pressing technique. This method requires the equipment pictured in Fig. 1 and involved the following procedure.

The two metals to be bonded were placed in an aquadag lubricated 18-4-1 tool steel die, 16" high by 1.440" I.D., between punches of 1.366" diameter made of the same material. A thermocouple well was located in the die body 3-1/2" down from the top of the die while another well was located centrally in the bottom punch 8-1/2" from the bottom of the die. This die assembly was located in three cylindrical ceramic heating furnaces placed in tandem. Each furnace was individually controlled by a power transformer (Variac). The die and furnaces were in turn placed in a water-cooled, stainless steel pot which could be evacuated. On this was bolted a cover containing a centrally located Wilson seal with an 18-4-1 ram 1" in diameter running through it. After sealing, the pot was evacuated by a roughing pump to 200 microns pressure, after which a diffusion pump was used to bring the pressure down to 5 to 15 microns. At this pressure the furnaces were turned on. As soon as the furnaces started to heat up, out-gassing of the entire unit raised the pressure to 30 - 400 microns. By the time the specimens were at temperature ready to be pressed,

approximately one-half hour, the vacuum pumps had re-established the 5 to 15 micron pressure. Once the desired temperature was reached, the required pressure was applied for a predetermined length of time to the 1" ram through to the top punch and to the specimen. When the time elapsed for keeping the specimen under pressure the pressure was released, the energizing coil current turned off and the assembly allowed to cool. After cooling, the die was removed from the pot and the specimen was ejected.

B. Specimen Preparation

Two different types of specimens were made for this investigation. One was for subsequent tensile test while the other was for determination of intermetallic penetration. The specimens were prepared as follows:

1. Tensile Bars

Commercially rolled nickel pieces $1/4$ " thick and 1.366" in diameter were placed between commercial rod of 2-S aluminum $1/2$ " thick and 1.366" in diameter. This sandwich in turn was slipped into a 2-S aluminum sleeve 1.344" O.D. and 1.370" I.D. This sleeve lined the couple up and prevented the aquadag lubricant from getting in between the aluminum-nickel interfaces. Immediately prior

II. PROCEDURE (cont'd)

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to the specimen assembly the nickel was abraded on its flat surfaces with 320 grit silicon carbide paper, producing clean, smooth surfaces. The aluminum was chemically cleaned just before assembly by

1. Degrease in acetone.
2. Distilled water rinse.
3. Immersion for three minutes in 5% NaOH at 70°-80°C.
4. Distilled water rinse.
5. Immersion for two minutes in 50% HNO₃ solution at room temperature.
6. Distilled water rinse.
7. Dry in a blast of gas.

Once the above sandwich was hot-pressed and ejected, the specimen was machined, such that the aluminum sleeve was removed and the nickel and part of the aluminum on both sides of it were taken down to a diameter of 1-1/4". The remaining aluminum was then threaded, see Fig. 2, and the bar so produced was tested for tensile strength. In all

II. PROCEDURE (cont'd)

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instances the specimens broke during the test at the nickel-aluminum interface and never within the aluminum or nickel. The ultimate tensile strength values at times showed considerable scatter for a set of given reaction conditions. Because of this, as many as three to five specimens were made for a particular set of conditions. The trend of the average tensile strengths obtained was not as conclusive as was the trend of the maximum tensile strengths, the latter values being obtained under optimum reaction conditions. Therefore, the values of ultimate tensile strength given in this report are maximums.

2. Penetration Couples

The preparation of penetration couples was very similar to that of assembling the tensile bars; however, molybdenum strips $3/8$ " square and 0.001" thick were placed at the center of the aluminum-nickel interfaces as the couples were being assembled. The molybdenum strips were used as inert markers for they did not react with either the aluminum or nickel at the reaction conditions used. In addition, the molybdenum prevented aluminum-nickel interaction where they came between the aluminum and the nickel. In this manner they, therefore, acted as reference lines for eventual penetration measurements, i.e., the extent of

penetration was measured from both sides of the aluminum-nickel interface, as defined by the molybdenum strip, up through and to the deepest penetrations of the aluminum-nickel intermetallic compounds formed.

C. Penetration Measurements and Methods of
Calculating Penetration Constants

After the assembly was hot-pressed and ejected from the die, a quarter-pie shaped section was cut from it, mounted, and measurements of thirty of the deepest penetrations into the aluminum and into the nickel were determined microscopically. What was done was to project the image of the aluminum-nickel interface at 2000X magnification onto the screen of the metalloscope being used and measure the penetrations to the nearest 64th of an inch. The thirty measurements were averaged and this average was then converted to cm. Once this value was obtained, the penetration data were calculated from the following formulae:

$$P = X^2/t$$

X = penetration in cm

t = time in seconds at temperature and
under pressure

P = penetration coefficient in cm²/sec

II. PROCEDURE (cont'd)

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$$P = P_0 e^{-Q/RT}$$

$$\ln P = \ln P_0 - Q/RT$$

P_0 = temperature dependent constant

Q = activation energy for penetration in cal/gm-atom

R = universal gas constant

T = absolute temperature of reaction

$\ln P_0$ = intercept on $\ln$ axis of $\ln P$ vs $1/T$ curve

A typical view of a sectioned penetration couple may be seen in Fig. 3.

III. RESULTS

A. Ultimate Tensile Strength

1. Effect of Pressing Temperature

The effect of pressing temperature on the ultimate tensile strength of aluminum-nickel couples was first determined, see Table I for the data obtained. Fig. 4 is a plot of the maximum ultimate tensile strength obtained versus varying pressing temperatures. Three curves are shown, each representing specimens pressed at different applied pressures, namely, 2, 11 and 20 tsi. In all instances pressure was applied for a period of four minutes.

The curves show an increase in strength with increasing temperature and specifically for the 11 and 20 tsi pressures; the curves reach a maximum, decline, and then indicate a leveling-off period at temperatures above approximately 550°C.

It is observed that as the pressure increases, the maximum tensile strength achieved also increases. Both the 11 and 20 tsi curves exhibit maximum strength at approximately 500°C. (Some work done at 5 tsi indicated that maximum tensile strength also occurred around 500°C). Note that the initial rising and falling slopes of each curve decreases in rate of rise and fall, respectively, as the pressure used decreases. In addition, it is observed that the beginning of incipient bonding occurs at lower temperatures as the pressure is increased.

2. Effect of Applied Pressure

Fig. 5 shows the effect applied pressure held for four minutes has upon the maximum ultimate tensile strength of aluminum-nickel couples. See Table I for the data. These experiments were carried out at a series of isotherms, specifically, 450°, 500°, 550°, and 600°C. Incipient bonding was found to occur at 400°C for pressures of 11 and 20 tsi. This is not shown on the illustration.

At 450°C tensile strength is seen to increase rapidly while at 500°C the bonding increases even more rapidly, and to much higher values for increased applied pressure. At 550°C the increase of tensile strength with applied pressure is much more gradual than at 500°C, and the curve shows a leveling-off portion. At 600°C the tensile strength starts out at a moderately high level and then as the pressure increases to 23.5 tsi it increases slightly.

3. Effect of Time at Pressure

Fig. 6 is a plot of the maximum ultimate tensile strength vs time at 11 tsi pressure for a number of isotherms, namely, 500°, 550°, and 600°C. All curves exhibited the same tendency, that is they rise, reach a maximum, and then decline. It can be seen that maximum strengths are developed when the specimen is held at pressure for approximately four minutes. It can also be seen that once again maximum strengths occur at the 500°C isotherm. Table II is a tabulation of the data obtained from this series of experiments.

B. Aluminum-Nickel Intermetallic Penetration

1. Effect of Pressing Temperature

Fig. 7 is a plot of the log of the penetration coefficients into aluminum and into nickel versus the reciprocal of the absolute pressing temperature. See Table III for the data. These specimens were reacted at various isobars, namely, 5, 11, and 20 tsi, all held at pressure for two minutes.

The upper three curves are for aluminum-nickel penetration into aluminum while the lower three are for aluminum-nickel penetration into nickel. (The top three curves are considered more accurate than the bottom three since the extents of penetrations were more readily measured). Note that the curves are straight lines. This shows the penetration data of aluminum-nickel intermetallics conform to the equation $P = P_0 e^{-Q/RT}$ and in turn shows the penetration effect is dependent upon the diffusion phenomenon.

In addition, it is seen that the curves are displaced from each other, indicating that the isobar used has a definite effect upon the penetration rate. Table IV is a tabulation of the calculated activation energies and P_0 values for the six curves presented.

The three top curves of Fig. 7 are approximately parallel and the activation energies calculated for the three curves are about the same. This fact indicates that the activation energy for the above penetration phenomenon is independent of pressure applied while penetration is occurring.

Use was made of the curves of Fig. 7 to obtain X values from the equation $X = \sqrt{Pt}$, t, being taken as four minutes to plot the curves of Fig. 8. These show the dependence of nickel-aluminum intermetallic penetration into aluminum for increasing pressing temperatures. One curve represents an applied pressure of 11 tsi while the other represents 20 tsi. Both are seen to rise steeply in an exponential manner as the temperature is increased. The curve for the 20 tsi pressure is displaced downward from that of the 11 tsi curve, that is, for the same reaction temperature and time at pressure there is less penetration at 20 tsi than at 11 tsi.

Fig. 9 is based on previously discussed tensile strength data and the calculated penetration depths of the curves in Fig. 8. Here is shown how tensile strength and penetration are related. Both curves are similar in general appearance in that they rise rapidly, decline and then tend to level off. The greatest strength was attained when using 20 tsi

although the decline in strength for this curve was much more rapid than for the 11 tsi isobar curve. In addition, the final strength of the level-off period, i.e., for greater penetrations, was lower for 20 than for 11 tsi.

2. Effect of Applied Pressure

Fig. 10 shows a plot of data from Table III indicating how the penetration coefficients of aluminum-nickel inter-

metallics into aluminum are affected by pressure. This was done at various isotherms, namely, 500°, 550°, 575°, and 600°C. All the curves are observed to be straight lines with increasing slope as the temperature increases. It can be seen that the penetration rates decrease as the applied pressure is increased and that all the curves tend to approach a small range of pressures at "0" penetration rate.

Actual experiments conducted at 500°C at pressures close to and above that required for "0" penetration rate confirmed the finding. That is, no intermetallic formation was observed when sufficiently high pressure was used, as indicated in Fig. 11. Located on this figure are representative metallographic pictures of the bond interface. The dark zone disappears between 11 and 20 tsi pressures and the light colored zone continues to diminish and tends

to become discontinuous even at higher pressures. Finally, at 34 tsi no trace of either alloy zone can be seen. Unfortunately, we were not able to make tensile tests at all the investigated points for, as the pressure increased, exceptionally severe extrusion of the aluminum occurred in our already deformed die and tensile bars were not readily hot-pressed. However, it should be pointed out that, with the disappearance of the dark zone, tensile strength rose over three times that obtained when both zones were present.

The reason for this decrease in penetration rate as the pressure is increased is thought to be as follows. The intermetallics that are formed between nickel-aluminum during solid state welding under the reaction conditions used have a combined volume greater than the nickel and aluminum total volumes entering into the reaction. Thus there would tend to be an overall expansion. When pressure is applied externally, this expansion is restricted. As the pressure increases, this restriction is further increased until a point is reached where the applied external pressure is equal to or greater than the internal expansion pressure. When this occurs the intermetallics are inhibited from forming. As observed from Figs. 4 and 9, there is a peak tensile strength associated with aluminum-nickel

couples reacted under different applied pressure, namely, 11 and 20 tsi held for four minutes at 500°C. In addition it was stated previously that a similar peak value was obtained for specimens reacted at 5 tsi for four minutes at 500°C. By use of P values obtained from Fig. 7, the penetrations were obtained for each of the above pressures at 500°C where the peak tensile strengths occurred. The data obtained, see Table V, are plotted in Fig. 12 as the penetration of aluminum-nickel intermetallics into aluminum for maximum tensile strength versus the applied pressure.

The three points tend to indicate the curve is a straight line. Extrapolation to zero pressure, i.e., the equivalent of bonding by diffusion in a furnace at atmospheric pressure, indicates that maximum aluminum-nickel strength will occur when the aluminum-nickel penetrates the aluminum to a depth of about 10.5×10^{-4} cm.

C. Metallographic Results

Fig. 13 is a group of three photomicrographs showing the effect of temperature and pressure upon the extent of formation of inter-metallic nickel-aluminum zone band widths. Pictures "A" and "B" represent the effect of temperature. Both specimens were pressed at 5 tsi for two minutes, one at 550°C, the other at 600°C. The

III. RESULTS (cont'd)

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zones for the 600°C specimen are much thicker than for the 550°C specimen. The two specimens "B" and "C" show the effect of pressure on zone thickness. Both specimens were pressed at 600°C for two minutes, "B" at 5 tsi and "C" at 20 tsi. The zones of the specimen pressed at 5 tsi are much thicker than those of the specimen pressed at 20 tsi.

In most cases the presence of two distinct intermetallic zones was seen, this for unetched specimen preparation. X-ray diffraction study suggests the dark zone, blue-grey, next to the nickel to be the γ -phase, Ni_2Al_3 . The other zone, next to the aluminum and light in color, has been tentatively identified by etching techniques as the β -phase, Al_3Ni .

D. Possible Practical Applications of the Data Obtained

1. Prediction of Time at Temperature for maximum Strength of Aluminum-Nickel Couples

Penetration values obtained from Fig. 12, when used with the data obtained from the P vs 1/T curves of Fig. 7, enable one to determine the length of time necessary for an aluminum-nickel couple to be reacted at various temperatures to obtain maximum strengths. As an example, refer to Fig. 14, where the time to obtain a nickel-aluminum penetration of 10.5×10^{-4} cm into aluminum at atmospheric pressure is plotted versus various reaction temperatures. Note how rapidly this time decreases as the temperature is increased.

2. Predetermination of Time to Penetrate a Given Nickel Thickness at Various Temperatures

Fig. 15 is a practical application of the penetration information that was obtained. Here is a plot of the time for nickel-aluminum to penetrate through a given nickel thickness versus the pressing temperature at 11 tsi applied pressure. The lower curve is for nickel 1/4 mil thick, while the upper curve is for nickel 1/2 mil thick. If a four-minute pressing time is taken, it can be observed that the curve for 1/4 mil thick nickel is intersected at approximately 578°C. This means that a nickel film of this thickness would be just completely diffused through at approximately 578°C at a pressure of 11 tsi held for four minutes. A specimen was prepared and pressed under these conditions. Metallographic inspection showed that the nickel was just completely diffused through, indicating that the data obtained tended to be confirmed. It can be seen that 1/2 mil nickel provides a much greater factor of safety as far as being penetrated by the aluminum-nickel intermetallics that form. In a like manner the time to penetrate a given aluminum thickness at various temperatures can be predicted.

IV. SUMMARY

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A study was made regarding the effect that a number of variables had on aluminum-nickel solid state bonding. Among the variables investigated were temperature, pressure, and time at pressure. The following is a brief recapitulation of what was learned.

A. Ultimate Tensile Strength

1. Temperature Effect

As the temperature for reaction was raised, the following general stages of tensile strength were observed, these for a given applied pressure held for four minutes. The strength

- a. Increased
- b. Reached a maximum
- c. Declined
- d. Levelled off

2. Pressure Effect

The effects of increased applied pressure on aluminum-nickel strength when held at various isotherms for four minutes were:

- a. Incipient bonding at 400°C
- b. Strength increased very sharply at 450° and 500°C
- c. At 550°C, strength increased slowly and to a lower maximum than for "b" above.
- d. At 600°C, strength increased slowly from a relatively moderate maximum.

3. Time at Pressure

For various isotherms and using an applied pressure of 11 tsi for increasing lengths of time, the following effects on strength were observed;

- a. A rise to a maximum
- b. A slow decline from the maximum value.

It was found that the greatest strength occurred at four minutes for all isotherms used.

B. Intermetallic Penetration

1. Effect of Temperature

Plots of the log of penetration coefficient versus the reciprocal of the absolute temperature were straight lines.

2. Effect of Pressure

No significant change in slopes of the log P vs $1/T$ curves for aluminum-nickel penetration in aluminum was noted with increased pressure. However, the penetration coefficient was found to decrease as the pressure decreased. This decrease was more rapid as the temperature of the reaction was raised. The use of sufficiently high pressures at 500°C completely inhibited intermetallic phase formation.

IV. SUMMARY (cont'd)

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C. Metallographic Results

After reaction at low pressures, two intermetallic phases were observed between the aluminum and nickel. The one nearest the nickel was tentatively identified as the γ -phase, Ni_2Al_3 while the other was identified as the β -phase, Al_3Ni .

V. CONCLUSION

It is possible, by use of the data obtained, to predict tensile strength and resultant penetrations for aluminum-nickel couples made by the solid state bonding technique used. Examples of how this can be done have been given in this report.

Perhaps the most important findings were those of the effect pressure had upon tensile strength and penetration rates. The fact that embrittling intermetallics can be prevented from forming by use of sufficiently high pressures is of paramount importance. It is now conceivable to reconsider the evaluation of the bonding of various metals which previously were rejected because of embrittling intermetallics.

This work has proven both of academic as well as of practical importance. Because of its potential meaning, a broad fundamental program is being put into effect. This is being done in order to get a more basic theoretical understanding of the phenomenon discovered; i.e., the selective formation of alloys by proper pressure application during solid state bonding.

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The fine work of A. Bartozak and W. Marz in preparing all the hot-pressed specimens and of J. Nylin for his penetration measurements is hereby acknowledged.

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HOT PRESSING APPARATUS

FIG. 1

Fig. 2 - Typical tensile test specimen, actual size

Fig. 3 - Typical penetration couple, actual size

AL-Ni TENSILE STRENGTH VS PRESSING TEMPERATURE

FIG. 4

Fig. 5— Al-Ni TENSILE STRENGTH vs. APPLIED PRESSURE
HELD FOUR MINUTES.

AL-Ni TENSILE STRENGTH VS TIME AT PRESSURE (11 ksi)
FOR VARIOUS TEMPERATURES

FIG 6

Fig.7 - EFFECT OF TEMPERATURE ON Ni-AI PENETRATION.

Fig.8 - PENETRATION INTO ALUMINUM vs. PRESSING TEMPERATURE.

Fig.9- TENSILE STRENGTH vs. PENETRATION INTO ALUMINUM.

AL-Ni INTO AL PENETRATION COEFFICIENT VS APPLIED PRESSURE

FIG. 10

Fig. II - PENETRATION COEFFICIENT INTO ALUMINUM AT 500°C vs. APPLIED PRESSURE.

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Fig. 12 - PENETRATION INTO ALUMINUM FOR MAXIMUM TENSILE STRENGTH vs. APPLIED PRESSURE.

A - Unetched diffusion couple, 550°C,
5 tsi, 2 minutes, 1500X

B - Unetched diffusion couple, 600°C,
5 tsi, 2 minutes, 1500X

C - Unetched diffusion couple, 600°C,
20 tsi, 2 minutes, 1500X

Fig. 13

Fig. 14-- TIME TO OBTAIN 10.5 μ PENETRATION INTO ALUMINUM AT ATMOSPHERIC PRESSURE vs TEMPERATURE.

TIME FOR Ni-AL PENETRATION THROUGH Ni VS PRESSING TEMPERATURE

TABLE I

Ultimate Tensile Strength, psi, as a Function
of Temperature and Pressure

Four mins. at Pressure tsi	Pressing Temperature, °C				
	400	450	500	550	600
23.0	-	-	-	-	5100
20	10	10,800	19,000	4425	2610
11	30	150	5940	4275	4120
2	-	-	zero	1250	3550

TABLE II

Ultimate Tensile Strength, psi,
as a Function of Time at 11 tsi

Pressing Time mins.	Pressing Temperature, °C		
	500	550	600
1	-	-	2180
2	4770	3720	3500
4	5940	4275	4120
10	3760	3360	2970

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TABLE III

Penetration Data for Various Pressures
Held Two Minutes

5 tsi				
Into Al			Into Ni	
Temp. °C	$X \times 10^3$ cm	$P \times 10^9$ cm ² /sec	$X \times 10^3$ cm	$P \times 10^9$ cm ² /sec
600	2.09	36.2	0.785	5.15
575	1.63	22.6	0.655	3.59
550			0.450	1.70
<u>11 tsi</u>				
625	-	-	0.725	4.40
600	1.79	26.6	0.570	2.73
575	1.32	14.6	0.340	0.975
550	1.00	8.35	0.280	0.650
530	0.765	4.97	0.177	0.262
500	0.535	2.38	0.170	0.240
<u>20 tsi</u>				
600	1.25	13.2	0.350	1.03
550	0.655	3.58	0.220	0.404
500	0.335	0.943	0.140	0.164

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TABLE IV

Penetration Constants for Aluminum-Nickle Couples
Hot-Pressed at Various Pressures

	5 tsi		11 tsi		20 tsi	
	Penetration Into		Penetration Into		Penetration Into	
	Ni	Al	Ni	Al	Ni	Al
Activation Energy, Q, calories gm-atom	36,500	32,300	41,000	33,000	26,700	37,400
P_0 , cm ² /sec	13.0	4.6	58.9	5.3	2.7	31.6

TABLE V

Penetration for Peak Tensile Strength, x 10 ⁻⁴ cm	Applied Pressure, tsi
9.0	5
7.4	11
4.8	20