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DIFFUSION BONDING OF IN 718 TO VM 350 GRADE MARAGING STEEL

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ABSTRACT

Diffusion bonding studies utilizing a "Gleeble" have been conducted on IN 718, VM 350 and the dissimilar alloy couple, IN 718 to maraging steel. The experimental processing parameters critical to obtaining consistently good diffusion bonds between IN 718 and VM 350 are determined. Interrelationships between temperature, pressure and surface preparation were explored for short bonding intervals (< 15 min) under vacuum conditions. Successful joining was achieved for a range of bonding cycle temperatures, pressures and surface preparations.

The quality of the bond was evaluated by several test methods. Metallographic investigation of the joint for grain growth across the original interface and for growth of the diffusion zone was considered an important criterion of bond quality. Tensile testing for a simple butt weld configuration of both heat treated and as bonded samples was also used as a basis for this judgment.

Both annealed and heat treated diffusion couples were studied. A compatible heat treatment for both alloys was found to be possible. This simultaneous heat treatment resulted in bond tensile properties that were comparable to those obtained in the weaker parent material. The drastic yield strength differences and work hardening characteristics in the two alloys when heat treated insures the creation of a notch effect near the bond interface, with failure in this region likely. Therefore, the strength of the weaker parent material is used as a criterion for a successful tensile test of the heat treated bond. Studies of VM-350/VM-350 couples in the as-bonded condition showed a greater yielding and failure outside the bond region. This characteristic of failing outside the bond region is not necessarily a useful criterion for dissimilar alloy couples.

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INTRODUCTION

The desire to obtain quality joints in materials has long been of prime interest. The diffusion bond is capable of providing that superior joint [1, 2, 3] for many alloy systems. The process of diffusion bonding is best described as the formation of a metallurgically sound joint by causing interdiffusion of the surface atoms of either similar or dissimilar materials [4]. This bonding is usually performed at conditions below the melting point of any of the phases present, but high enough to assure fast diffusion rates. However, it is possible and often desirable to diffusion bond in the presence of a liquid phase which is transient [5]. Of the many types of diffusion bonding, the one considered in this research program is "yield strength controlled". This process uses a pressure during the formation of the joint that is higher than the yield strength of the weaker parent material [4].

Diffusion bonding is an attractive process as it virtually eliminates the inhomogeneity associated with recast structure and its attendant problems of segregation and varied grain size which are found in fusion welds. Diffusion bond properties approaching those of the parent material can be obtained. This results in engineering designs which require no increase in section size at the joint [2].

Many applications which require the superior metallurgical characteristics of the diffusion bond are also involved with extensive use of special high strength alloys [2]. Interest in diffusion bonding is often the result of finding that this technique is the only possible

method of joining high strength materials without producing highly inferior mechanical properties in the joint region. The successful joining of superalloys and specialty alloys often necessitates avoiding the formation of recast structures (fusion weld zone) making diffusion bonding the most attractive joining process [3].

Kaarlela and Margolis [3] describe some of the requirements for achieving good diffusion bonds in many of the superalloy systems. In their study, it was pointed out that acceptable bonds in iron base and nickel base alloys strengthened by aluminum and titanium were difficult to achieve. These alloys were bondable only at the highest temperatures and pressures ordinarily employed for the process, or by drastically increasing bonding time. Bartlett [6] indicates the need for very high temperatures (2000 to 2200°F) for bonding either iron base or nickel base alloys, due to low solubility of interstitials in these elements.

The problems associated with a jet engine shaft have suggested the possibility of a superalloy-iron base diffusion couple for overall superior fatigue properties. In this case, there are different requirements along the shaft for temperature and strength. The use of dissimilar materials along the shaft could meet these requirements offering considerable savings in terms of weight and elimination of complex cooling schemes. The purpose of this research was to determine the feasibility of diffusion bonding an 18% nickel maraging steel to a precipitation hardenable nickel base alloy and to determine a simultaneous heat treatment after bonding to obtain maximum mechanical properties of the two materials. Parameters which influenced the bonding process were studied with respect to the ease of sound joint formation. The bonding conditions which gave the most satisfactory results in terms of tensile strength and good microstructure were determined.

EXPERIMENTAL PROCEDURE

SURFACE PREPARATION

The samples used were 1-1/8 to 1-1/4 inches long and 1/4 of an inch in diameter. A flat steel block, drilled to hold the samples, was used to obtain different controlled surface roughnesses. Set screws held the longitudinal axes of the samples perpendicular to the faces which were polished or lapped. G. V. Alm [4] has indicated advisable surface finishes of 16 rms or better for bonding superalloys. The surface conditions obtained by using the polishing papers and lapping wheels were calibrated using both steel and glass, as material hardness affects the surface finish. Table 1 shows that surfaces equal to or better than the 16 rms were used. The ends not prepared for bonding were chamfered to fit the specimen holders in order to provide good electrical contact in the "Gleeble".

Two cylindrically ground bars 5/8 of an inch in diameter were used to hold the specimens. One end of each bar was clamped in the "Gleeble". The free ends of the bars were drilled to hold the specimens with three centering set screws placed 120 degrees apart. The bars themselves were kept aligned by two collars spaced by insulated plates. These support devices are shown assembled in Figure 1.

ATMOSPHERE

The entire specimen support apparatus is shown in Figure 1 as assembled. The vacuum chamber as prepared for bonding is shown in Figure 2.

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TABLE 1

CALIBRATION OF POLISHING PAPERS AND LAPPING WHEELS

Wheel or Lap	Surface Finish, Microinches (Arithmetric Average)
With Steel:	
400 grit	4.0 — 6.0
600 grit	2.0 — 4.0
5.0 micron alumina	1.0 — 1.4
0.3 micron alumina	——————*
With Glass:	
220 grit	10.0 — 15.0
320 grit	8.0 — 12.0
400 grit	2.8 — 3.2
600 grit	2.0 — 2.4
5.0 micron alumina	——————*

*Profilometer scratches were noticed
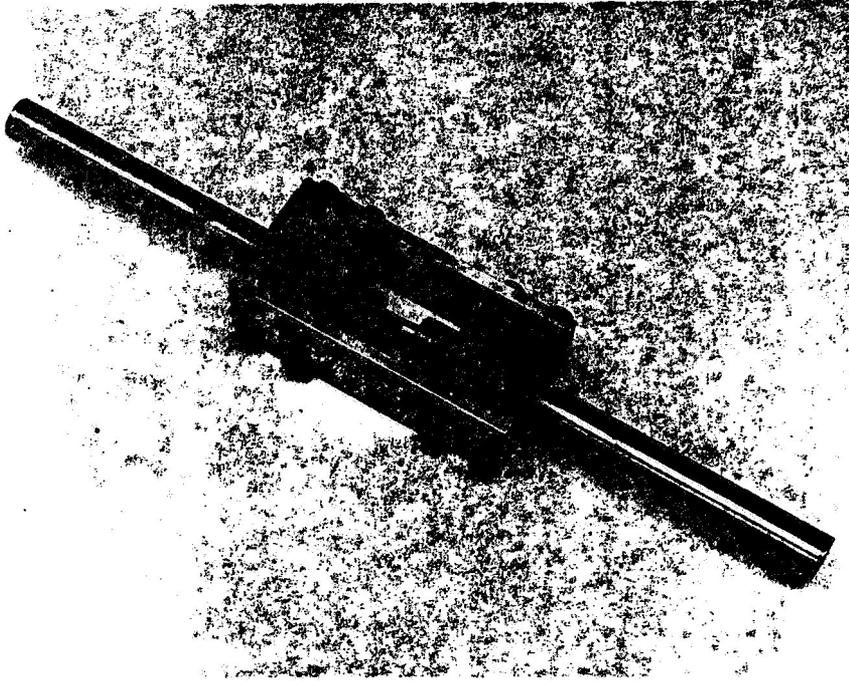at these values of surface finish.
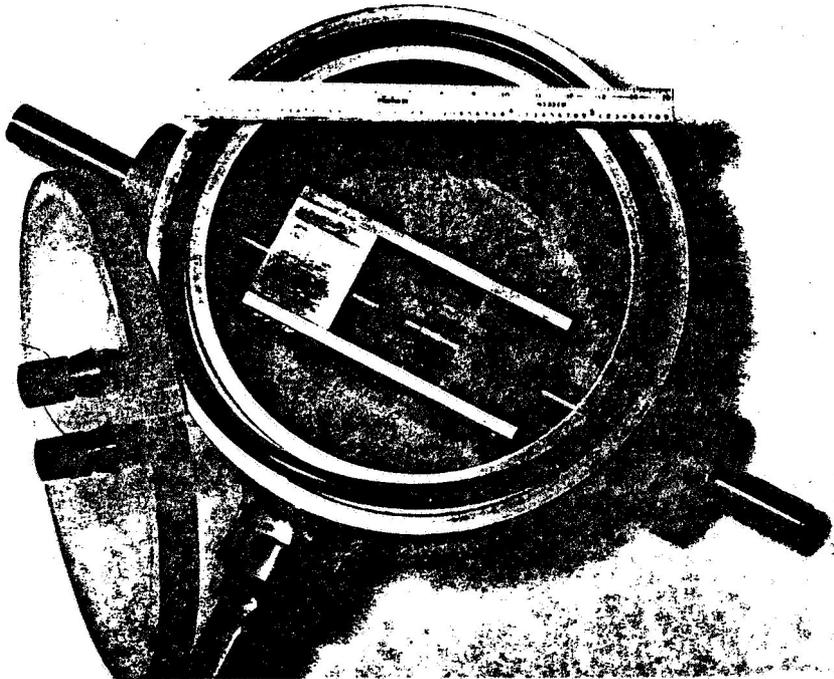
Fig. 1. — Specimen support device

Fig. 2. — Vacuum chamber

Mechanical and diffusion vacuum pumps were used in series to evacuate the chambers and a vacuum of 10^{-4} torr (0.1 microns of mercury) was consistently achieved. The samples were held apart in vacuum for at least ten minutes in order to remove molecular oxygen from the surfaces to be bonded. All samples were preloaded to prevent arcing at the bond surfaces.

"GLEEBLE" CONTROL

The "Gleeble", shown in Figure 3, was programmed to control several of the bonding parameters. The program generator on the "Gleeble", Figure 4, was set up to trigger the compressive load on the samples before heating. The value of this load is controllable and can be varied manually during the bonding process. Cline [2] indicates the pressure ranges necessary for bonding to be near 10,000 psi, and these values were used as a first approximation. The program generator also controls the rate of heating, the maximum temperature, and the time at temperature. Feedback for temperature control was provided by a chromel-alumel thermo-couple percussion welded to the sample 1/16th of an inch or closer to the bond surface. The temperature range used was 1/2 to 2/3 the melting point of the lower melting temperature alloy, a rule of thumb suggested by Alm [4]. A multichannel recorder was used to obtain the simultaneous readout of temperature, load, and deformation as a function of time. Complete monitoring of the bonding variables was maintained throughout the bonding cycle.

METALLOGRAPHY

Immediately after bonding, the samples were prepared for metallographic examination. Metallographic polishing and etching procedures used are standard. The entire metallographic processing procedure is presented in reference [7]. Some of the samples were mechanically tested in the
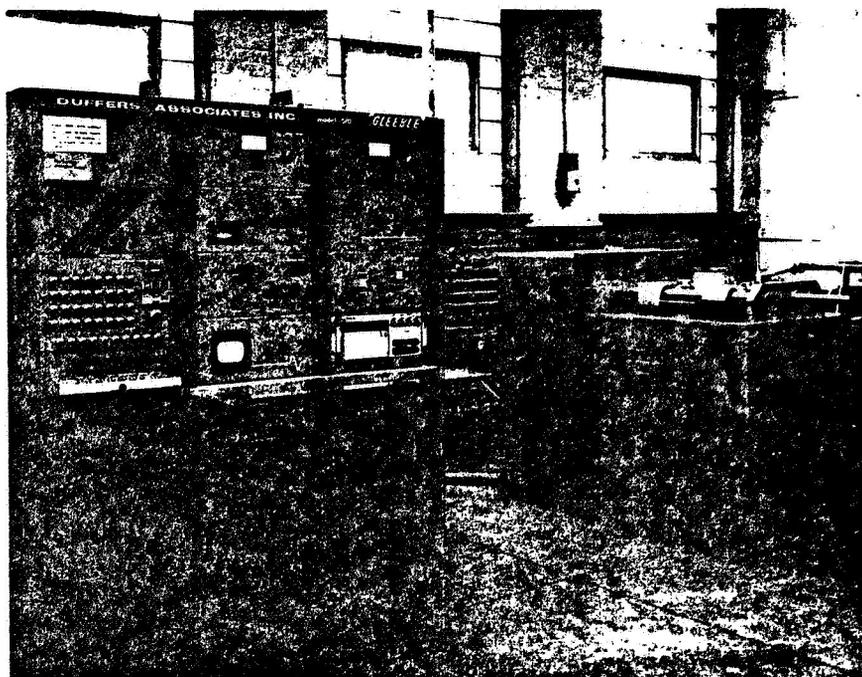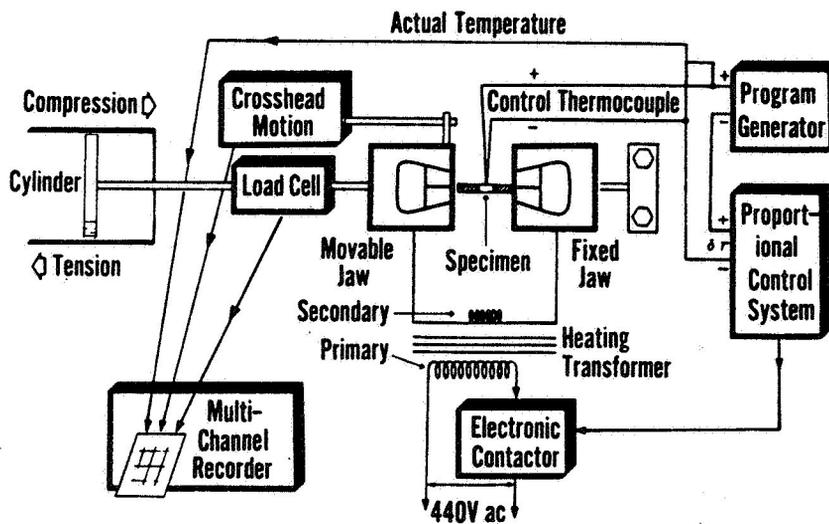
Fig. 3.—The "Gleeble"

Fig. 4.—Schematic of "Gleeble" control system

as-bonded condition, while other couples were heat treated after metallographic inspection and then mechanically tested.

HEAT TREATMENT

The heat treatment of the dissimilar alloy couple consisted of several steps, shown diagrammatically in Figure 5. The first sequence in the heat treatment was to solutionize the IN-718 at 1750°F for one hour. This was followed immediately by an air quench. The IN-718/VM-350 couple was reheated to 1325°F and held for 8 hours, then cooled at 100°F per hour to 1150°F and held for 8 hours, and finally air quenched. This treatment resulted in precipitation hardening of the IN-718 alloy and solutionizing of the VM-350 alloy. The VM-350 was then age-hardened for 3 hours at 930°F in an air atmosphere. Since the heat-treatment prescribed for IN-718 includes in its cycle the solutionizing treatment required for VM-350, the entire cycle for IN-718 is used as a solutionizing treatment for the VM-350.

TENSILE SPECIMEN PREPARATION

Preparation for the mechanical testing was done using a tool post grinder, because of the specimen size and difficulty in conventional machining of the alloys. The grinding wheel was contoured to create a tensile shape directly. The specimens were tested in a universal testing machine, using a strain rate of 0.2 inches per minute. Control samples were tested for both the as-received and the as-bonded conditions.
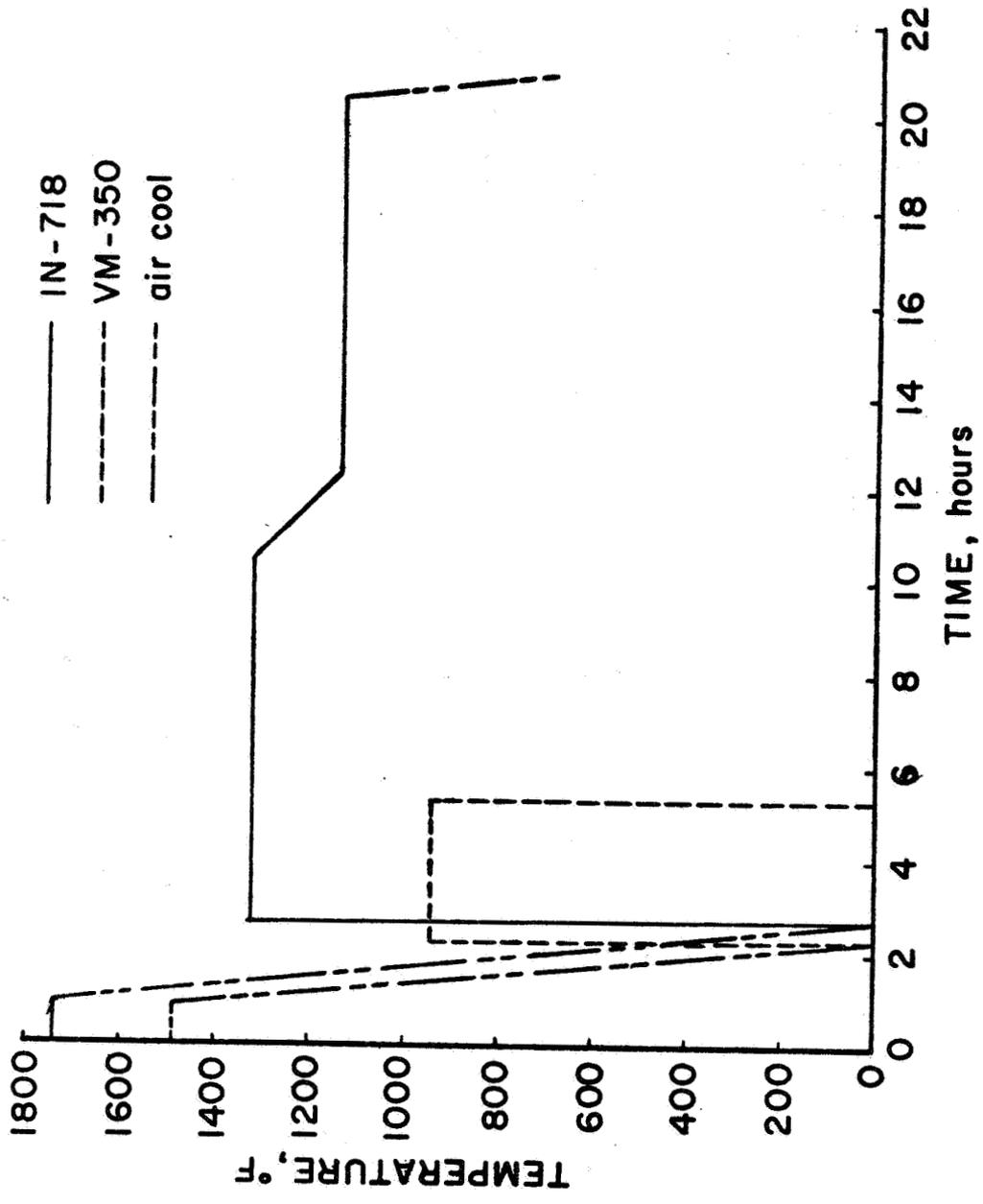
Fig. 5. IN-718 and VM-350 Heat-treatment Cycles.

VM 350/VM350 Couple

Early trials indicated that diffusion bonding of VM 350 to VM 350 was feasible utilizing controlled resistance heating in the "Gleeble". This couple was used to evaluate the bond quality produced by the "Gleeble". Failure was expected to occur outside the bond region for properly bonded VM 350/VM 350. Table 2 shows the bonding parameters used and the resultant tensile properties for VM 350 to VM 350 trials. Early attempts showed incomplete bonding with voids present in the joint, as typified in Figure 6. However, with increased load, rougher surface finish, and the same temperature, improved bonding was obtained as shown in Figure 7.

IN 718/VM 350 Couple

The bonding parameters and resultant tensile properties for the IN 718/VM 350 couple are presented in Table 3. These results indicate that the surface finish, produced by a 5μ lap produces the best diffusion bonding tensile properties for the IN 718/VM 350 couple for constant pressure and temperature conditions. Photomicrographs showing the size of the diffusion zone for three temperatures are presented in Figure 8. The temperature dependence of the diffusion bonding process is evident from the thickness change of the bonding zone with temperature. A quantitative relationship between diffusion zone size and temperature is presented in Figure 9. From the slope of this plot an activation energy for the process was determined to be 33,000 CAL/MOLE. Also evident in Figure 8 is a variation in the rate of grain growth for the two materials. This is shown in Figure 10. IN 718, while initially finer in grain size than VM 350, undergoes a more rapid rate of grain coarsening than the VM 350.

TABLE 2

BONDING PARAMETERS AND TENSILE PROPERTIES FOR VM 350VM 350 COUPLES

BONDING TEMPERATURE	SURFACE PREPARATION	TIME MIN	MAXIMUM[2] BONDING PRESSURE PSI	EQUILIBRIUM[2] BONDING PRESSURE PSI	PERCENT REDUCTION IN AREA	S_{ult} PSI
2161	Electron Polish	5	26300	14,600	- - -	122,000
2161	5 Min Lap	5	23800	10,900	39.3	157,500
2161	600 grit	5	25400	10,900	55.9	163,000
2161	400 grit	5	21900	10,200	59.0	163,500
1800[3]	___	5	___	___	72.6	171,800

1. Maximum value is due to thermal expansion

2. Equilibrium value, controlled by cylinder on "Gleeble"

3. As received stock subjected to bonding cycle for comparison

Fig. 6 Undesirable microstructure in joint region (voids)
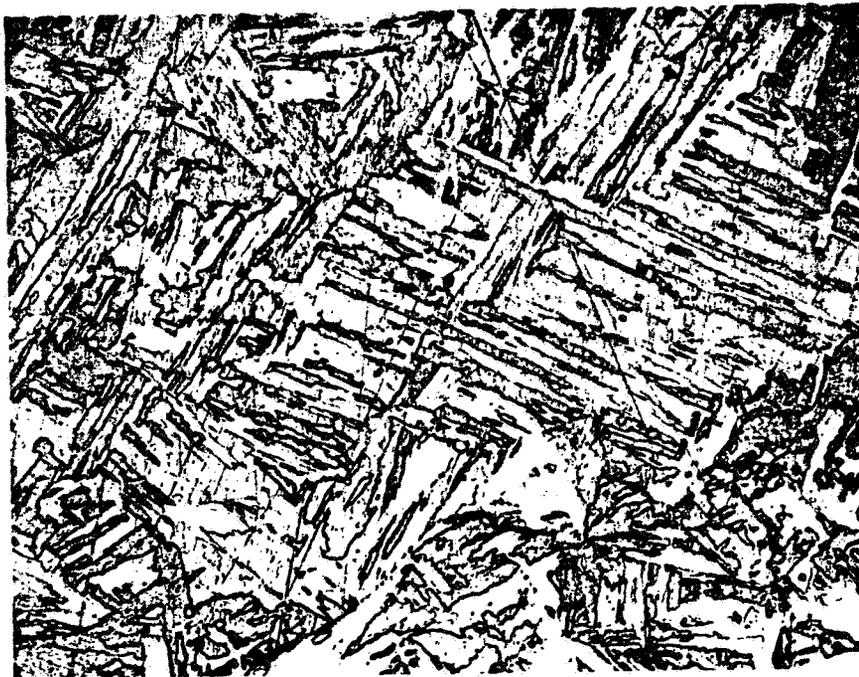for VM 350/VM 350 couple   375X

Fig. 7.   Desirable microstructure in Joint region
for VM 350/VM 350 couple   375X

TABLE 3

BONDING PARAMETERS AND TENSILE PROPERTIES FOR THE IN 718/VM 350 COUPLES

Bonding Temperature, °F	Surface Preparation	Time, Min.	Maximum[1] Bonding Pressure, psi	Equilibrium[2] Bonding Pressure, psi	Per Cent Reduction in Area IN 718	VM 350	Ultimate Strength, psi	Rockwell "C" Hardness IN 718	VM 350
As Bonded:									
1800	5.0μ lap	5	23,400	13,300	11.1	6.6	105,200[3]	23.4	26.4
1800	0.05μ lap	10	26,000	20,800	46.7	22.1	136,800[3]	22.7	32.3
1900	600 grit	5	18,200	10,900	7.9	2.2	76,100	(Too low	25.5
2000	400 grit	5	23,800	12,800	11.3	3.3	91,720	for R_c	21.9
Heat Treated:									
1700	0.5μ lap	5	10,000	6,670	5.7	4.0	196,000	39.1	52.0
1800	5.0μ lap	5	10,800	4,160	5.6	2.1	205,000	39.5	53.2
1900	600 grit	5	14,180	10,000	1.7	1.4	76,000[3]	41.5	50.0
1900	5.0μ lap	5	13,300	7,200	6.7	2.2	201,800[3]	40.6	51.6
1900	0.5μ lap	5	15,000	9,160	5.0	3.5	173,000	38.3	48.7
2000	600 grit	5	12,500	8,340	2.0	1.3	178,700[3]	38.8	48.7
2000	5.0μ lap	5	12,500	7,500	---	---	------	---	---

CONTROL SAMPLES

	Time, Min.	Maximum[1] Bonding Pressure, psi	Equilibrium[2] Bonding Pressure, psi	Per Cent Reduction in Area IN 718	VM 350	Ultimate Strength, psi	Rockwell "C" Hardness IN 718	VM 350
As-Bonded IN 718)1800°F	5	-----	------	58.6	---	151,800	21.0	----
" VM 350)	5	-----	------	----	72.6	171,800	----	29.0
Heat Treated IN 718[4]	--	-----	-----	13.6	----	184,000	42.0	----
" VM 350[4]	--	-----	-----	----	50.3	358,000	----	59.0

1. Maximum value, due to thermal expansion
2. Equilibrium value, controlled by cylinder on "Gleeble"
3. These specimens polished to remove grinding scratches
4. Manufacturers' data

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IN 718

VM 350

T = 1700°F as-bonded, 425X

IN 718

VM 350

T = 1900°F as-bonded, 425X

IN 718

VM 350

T = 2000°F as-bonded, 425X

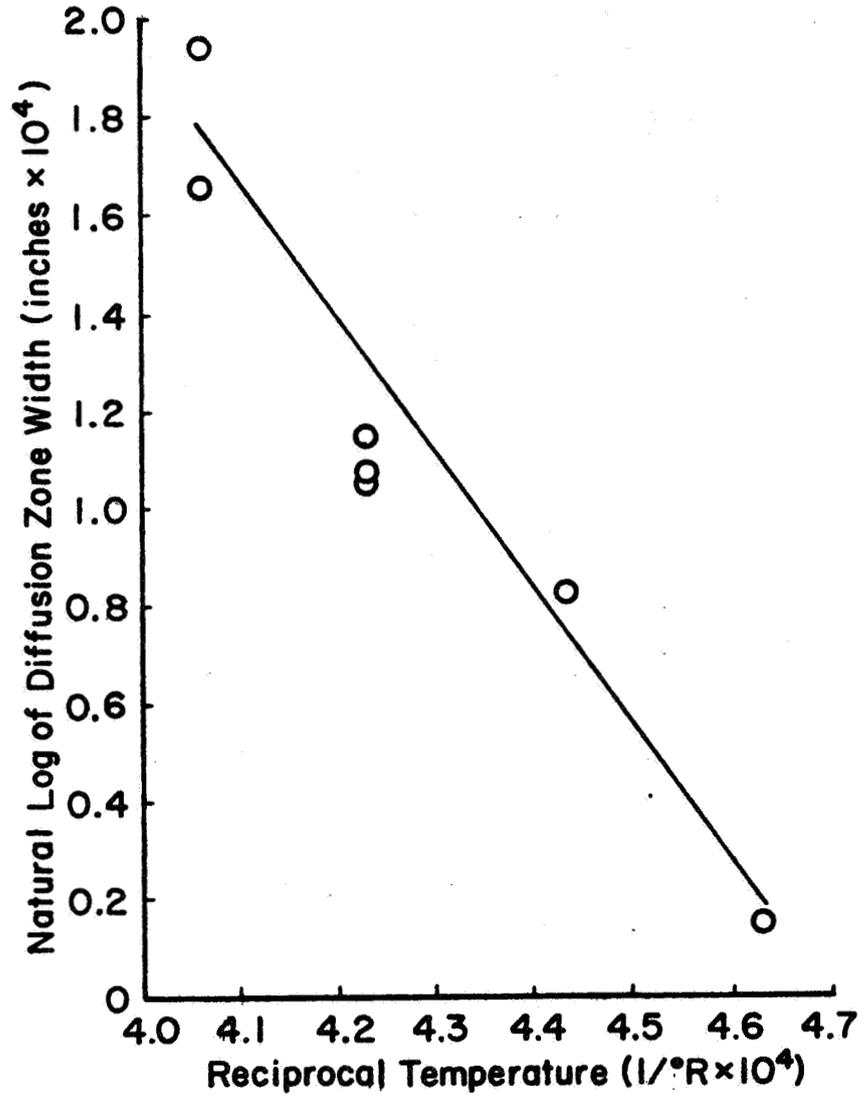Figure 8   IN 718/VM 350   Diffusion Zone Thickness for 1700°F, 1900°F, and 2000°F

Fig. 9. Temperature Dependence of Diffusion Zone Width for IN-718 / VM-350 Couple.
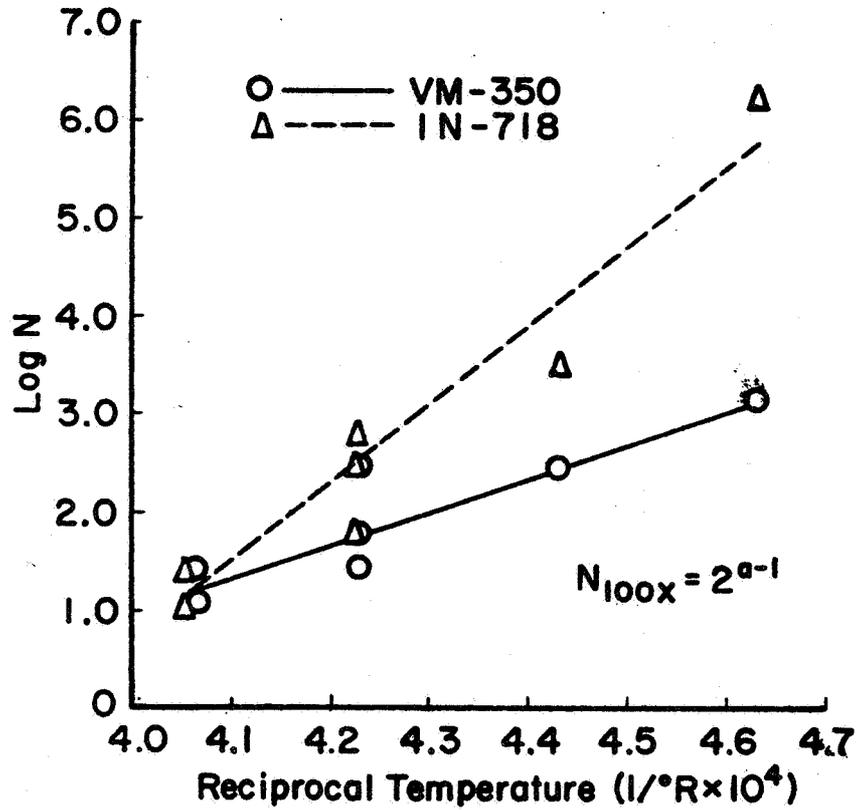
Fig. 10. Temperature Dependence for Grain Growth in VM-350 and IN-718.

PROPERTIES IN 718/VM 350 COUPLE

The results of tensile testing several as-bonded samples are presented in Table 3. The control tensile data for IN 718 and VM 350 which have experienced a similar bonding temperature-time cycle are also listed. As-bonded IN 718/VM 350 specimens exhibit ultimate strengths approaching that of the weaker parent material, IN 718. Rockwell C hardnesses for the simultaneously heat treated materials are in good agreement with expected values as shown in Table 3.

Chemical analyses of the major solid solution elements and hardening elements necessary for precipitation hardening in the diffusion zone region are presented in Figures 11 and 12. These profiles were obtained using an electron beam microprobe with a spot size of 4μ . The traverse across the interface was indexed in 2μ steps until constant base alloy composition was achieved. Profiles for iron, nickel, cobalt, molybdenum, chromium, aluminum and columbium were determined. The width of the diffusion zone from a chemical analysis standpoint was considered the distance over which a gradient in any element occurred.
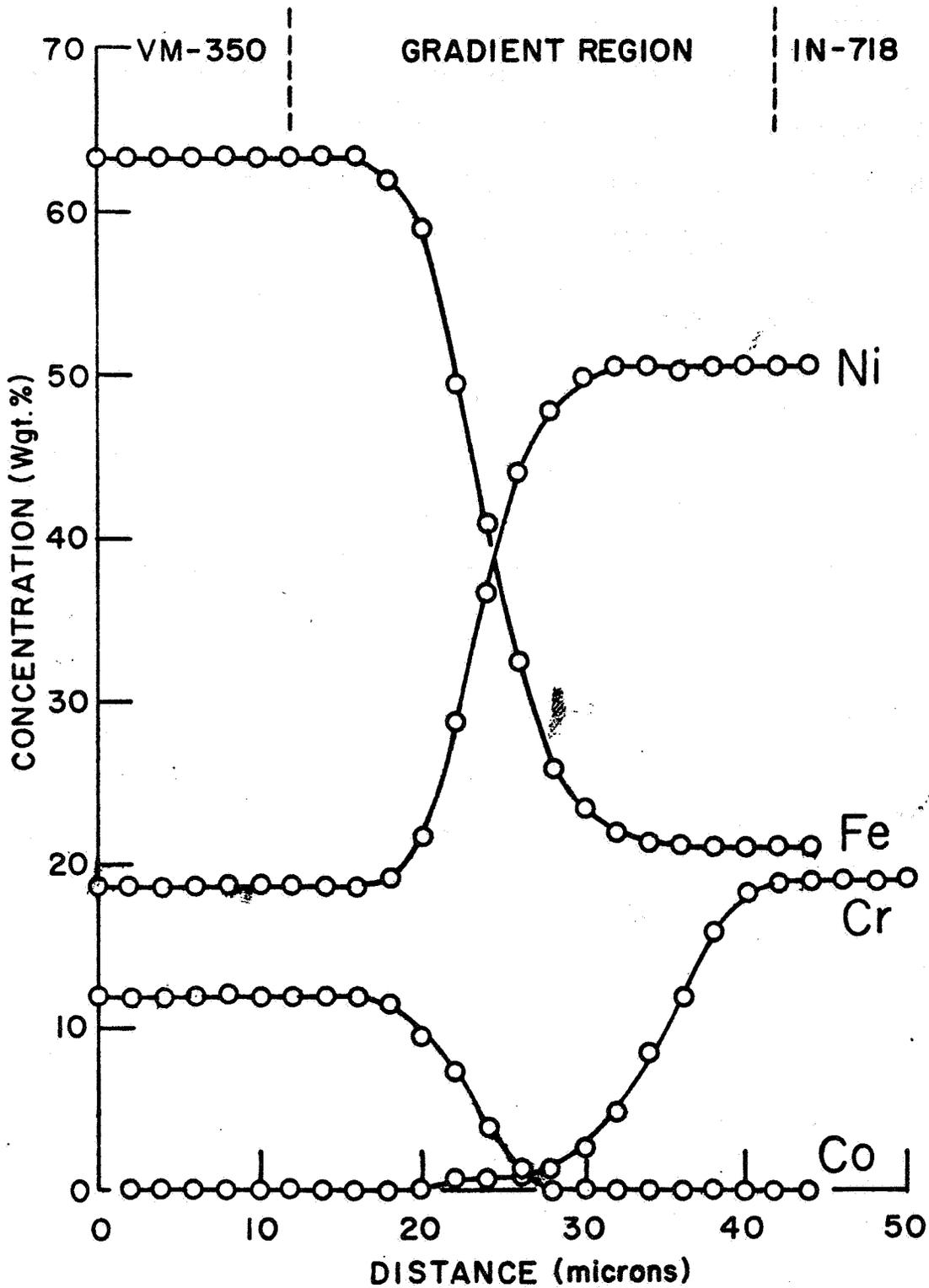
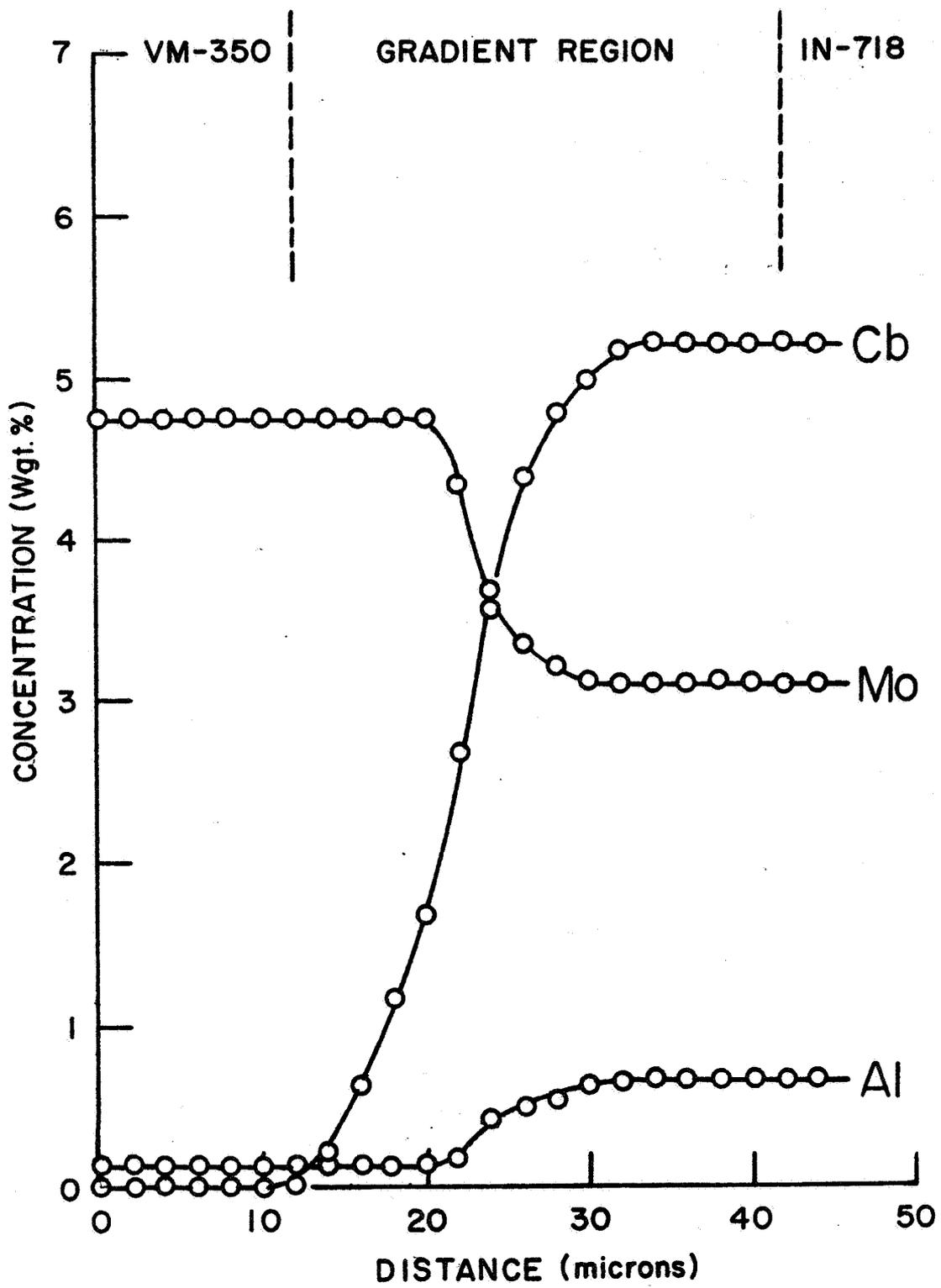Fig. II. IN-718 / VM-350 Diffusion Bond Major Element Profiles

Fig. 12. IN-718 / VM-350 Diffusion Bond Hardening Element Profiles

ANALYSIS OF RESULTS

VM-350/VM - 350 Couple

For diffusion bonding like materials, Owczarski, King, and O'Connor [8] have pointed out that ductility as measured by reduction in area is sensitive to the void content of the joint. They further state that void content decreases with time for a given temperature and pressure, until no trace of the original interface remains. If joint properties matching the parent material are to be obtained, grain boundary movement, which is rapid at higher temperatures, is considered essential to the bonding process [8].

For a 5 minute time interval, the VM-350 to VM-350 couple required a relatively high bonding temperature, in the neighborhood of 2150°F. The bonding pressure was approximately 10,000 psi after relaxation of stresses induced by thermal expansion. This stress was high enough to cause plastic flow of the material throughout the bonding cycle. Rough surface finishes provided for better plastic flow in the joint region, and therefore enhanced the rate of elimination of voids. Excellent bonding was achieved with failure in tension occurring outside the joint region. This clearly indicates that bond properties equal to or better than those of the base material can be achieved for this couple.

IN 718/VM350 Couple

Bonding of IN 718 to VM 350 showed that the width of the diffusion zone and grain size can be controlled by temperature. Figure 9 shows the temperature-width relationship to be exponential while Figure 10 indicates that the grain size of each alloy is exponentially related to the temperature, although the two alloys have significantly different grain growth rates. An

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upper bonding temperature limit of 2100°F has been established for the IN 718/VM 350 couple due to the onset of intergranular melting in the IN 718 alloy. The void content of the joint also decreases with time [9] which is likely due to interdiffusion effects in the mixed alloy couple.

PROPERTIES IN 718/VM 350 COUPLE

In this dissimilar couple there is a yield and ultimate strength differential for these two alloys. In the as-bonded condition the yield strengths differ by about 20,000 psi. However, in the heat treated condition, the difference is approximately 200,000 psi. Differences in the slopes of the stress-strain curves after plastic deformation begins shows that the IN-718 will flow plastically an appreciable amount more than the VM-350 for a given load. This behavior leads to the formation of a notch effect in the joint region. The only plastic flow which can occur in this couple is limited to deformation in the weaker material before stress concentration due to the notch effect causes the sample to fail in the joint.

The long heat treatment process of approximately 24 hours results in significant strengthening of both alloys. This heat treatment in air also provides a measure of good bond integrity as oxides formed during this process did not invade the joint and split the couple apart. The ultimate strengths attained for both the as-bonded and heat treated couples indicate joint strengths comparable to the ultimate strength of IN-718 in the as-bonded and heat treated conditions respectively. The ultimate strength of the diffusion zone is also increased with heat treatment, as it is capable of supporting the high tensile loads after heat treatment.

ELEMENT DISTRIBUTION IN DIFFUSION ZONE

The width of the diffusion zone observed metallographically was approximately 13 microns. When diffusion profiles are used to establish zone width, a value of about 30 microns is determined. This approach for

determining width includes the depletion or relative immobility of certain alloying elements. However, considering the more mobile elements, agreement with the 13 micron width is observed. By fixing an arbitrary center for the joint, dependent upon the diffusion profiles, the relative degree of diffusion of the various elements can be compared. On this basis, it is noted that nickel diffuses more rapidly into the VM 350 than iron into the IN 718. Although chromium and cobalt are present throughout most of the joint, they do not diffuse considerably into the VM 350 or IN 718 respectively. The strengthening elements, columbium and aluminum, diffuse appreciably through the joint in conjunction with nickel, making strengthening of the interdiffusion zone possible by a precipitation process similar to that for IN 718. This is in good agreement with observed strengthening effects noted in heat treated couples that were tensile tested.

CONCLUSIONS

1. The 'Gleeble' can be used for modeling the diffusion bonding
    process. This was demonstrated by successfully bonding the
    VM 350 to VM 350 couple.

2. Tensile testing and metallographic examination demonstrate that
    it is feasible to diffusion bond IN 718 to VM 350.

3. Deformation in the joint region assists bonding and is necessary
    for achieving consistently good bonds.

4. For a given time interval, the width of the diffusion zone and
    grain growth of both materials can be controlled during bonding
    primarily by variation in the temperature. An exponential rela-
    tionship with an activation energy of 33,000 CAL/MOLE exists
    between temperature and the diffusion zone width. Separate
    exponential relationships exist between temperature and the grain
    size of the two materials.

5. The IN 718/ VM 350 couple fails in the joint region for room
    temperature tensile tests due to drastic yield strength and work-
    hardening differences between the two alloys.

6. The simultaneous heat treatment of IN 718 and VM 350 is possible
    and results in joints with ultimate strengths equaling the heat
    treated ultimate strength of the weaker alloy, IN 718.

FURTHER WORK

1. Cyclic loading in tension and compression concurrently with cyclic heating and cooling should be investigated for their effects on grain growth.

2. The relationships between surface texture, intermediate foils, and inert or reducing atmospheres should be determined.

3. Shear, fatigue, and impact testing of the joint would supply important additional engineering design information.

4. Analysis for the presence of a strengthening mechanism in the diffusion zone is suggested.

5. The diffusion gradients present in the diffusion zone and base materials near the joint should be analyzed.

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