

Direct Consolidation of γ -TiAl-Mn-Mo from Elemental Powder Mixtures and Control of Porosity through a Basic Study of Powder Reactions

T.K. LEE, J.H. KIM, and S.K. HWANG

A gamma titanium aluminide was made by elemental powder metallurgy. For consolidation of the alloy from powder blending, either hot extrusion or hot forging was used. A good combination of tensile yield strength and ductility was obtained by hot extrusion that produced a grain size of 50 μm . Consolidation by forging, however, resulted in a porous microstructure. On the basis of an investigation of the cause of the porosity by an Al/Ti diffusion couple experiment and by characterization of the temperature peaks due to an exothermic reaction among elemental powder particles, it was concluded that a transient phase such as TiAl_3 was the culprit. Being the source of Al diffusion, the transient phase leaves behind Kirkendall voids when it forms prior to the major exothermic reaction among elemental powder particles. From this study, two processing techniques to circumvent the porosity were proposed and verified: a fast heating to the consolidation temperature or sufficient soaking above the reaction temperature prior to consolidation. A sound, fully lamellar, β -phase controlled microstructure was obtained by these methods.

I. INTRODUCTION

GAMMA TiAl alloys have been under extensive study^[1-5] because of their low densities and potential applications at high-temperature environments. There are two basic techniques for producing the gamma alloys: ingot metallurgy^[6,7] and powder metallurgy.^[8,9] Among the powder metallurgy techniques, the elemental powder method (EPM) is an attractive technique due to economical reasons. Near-net-shape processing is feasible, provided a good control of porosity is ensured. The EPM processing consists of blending elemental powders, compaction, and consolidation at an elevated temperature. A favorable combination of strength and ductility can be achieved in the gamma alloys processed by EPM and consolidated by hot extrusion.^[5] Hot forging is an alternative route to consolidate the elemental powder mixture. In this process, however, pores may develop and cause difficulties in application of the materials. Pores in this alloy system may form due to Kirkendall diffusion.^[10,11,12] It has not been shown, however, when and where the pores develop during the processing of the EPM-processed gamma alloys. The present article addresses this question and attempts to find ways to circumvent the porosity.

II. EXPERIMENTAL PROCEDURE

The nominal composition of the experimental alloy studied was Ti-46.6Al-1.4Mn-2Mo (at. pct). The actual com-

position verified by chemical analysis was Ti-47.3Al-1.1Mn-2.1Mo (at. pct) with the content of the residual oxygen and nitrogen being 1800 and 40 ppm, respectively. Elemental powders used for the alloy fabrication have shapes as depicted in Figure 1. The vendors for each powder were Micron Metals (Milwaukee, WI) for Ti and CERAC (Salt Lake City, Utah) for Al, Mn, and Mo. The titanium powder was produced by the magnesium reduction method, whereas the Al, Mn, and Mo powders were produced by atomization, an electrolytic method, and melting, respectively. For Ti and Al, the mean particle sizes were 70 and 20 μm , respectively, and the purity was higher than 99.5 pct. For Mn and Mo, the mean particle sizes were 10 and 20 μm , respectively, and the purity was higher than 99.9 pct. Elemental powders were mixed for 4 hours using a V-blender without any additive. The blended powder mixture was cold compacted into a cylindrical rod of 50 mm in height and 22 mm in diameter. The compacted powder blendings were sealed in steel cans and degassed to 10^{-3} torr. The degassing sequence consisted of 1 hour at room temperature, 2 hours at 250 $^\circ\text{C}$, and 4 hours at 500 $^\circ\text{C}$, all in one series. The degassed cans containing compacts of blended powders were consolidated either by hot extrusion or hot forging. Hot extrusion was conducted at 1150 $^\circ\text{C}$ using an extruder having a 700-ton capacity. The reduction ratio of the extrusion was 10:1 in diameter. Hot forging was conducted by a 100-ton press at a rate of 50 cm/s^{-1} with a reduction of 20 pct in volume. Heat treatment was conducted in an argon atmosphere. A fully lamellar microstructure was obtained by a heat treatment consisting of holding 0.5 hours at 1400 $^\circ\text{C}$, air cooling to room temperature, reheating to 1365 $^\circ\text{C}$ and holding for 1.5 minutes, air cooling to room temperature, reheating to 900 $^\circ\text{C}$ and holding for 6 hours, and then air cooling to room temperature. Tensile testing was conducted in air at various temperatures with a strain rate of 10^{-3} s^{-1} . The gage length and gage diameter of round tensile specimens were 16 and 4 mm, respectively.

T.K. LEE, formerly Graduate Student, Department of Metallurgical Engineering, Inha University, is on leave for military service, Korean Army, Seoul, Korea. J.H. KIM, formerly Graduate Student, Department of Metallurgical Engineering, Inha University, is Senior Research Engineer, Technical Center, Daewoo Motor Co., Ltd., Incheon, Korea, 403-714. S.K. HWANG, Professor, is with the Department of Metallurgical Engineering, Inha University, Incheon, Korea 402-751.

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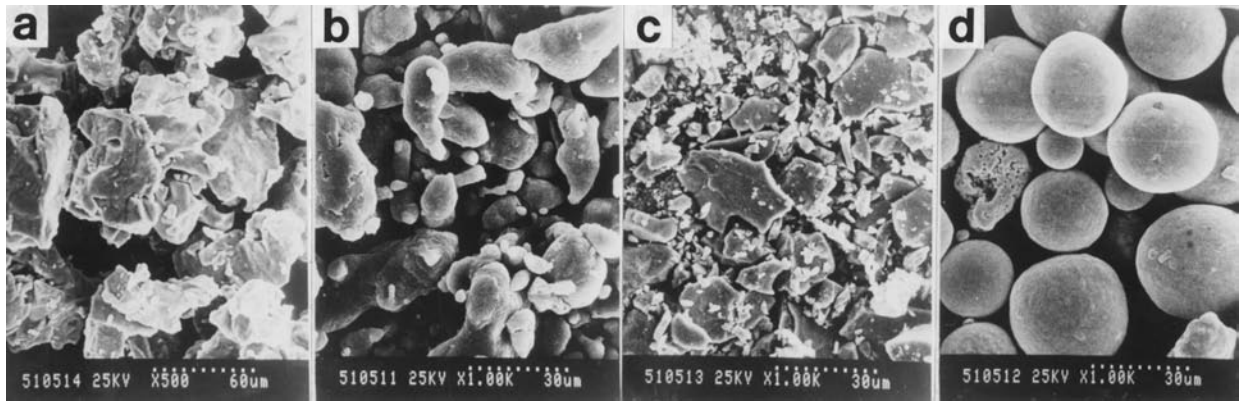


Fig. 1—Morphology of elemental powder particles: (a) Ti, (b) Al, (c) Mn, and (d) Mo.

Table I. Mechanical Properties of Ti-46.6Al-1.4Mn-2Mo as a Function of Temperature and Grain Size

Properties		Temperatures	Temperatures				
			Room Temperature	400 °C	600 °C	800 °C	1000 °C
0.2 pct yield strength (MPa)	grain size (μm)	55	548	531	512	478	292
	100	504	NA*	413	412	340	
	600	457	NA	NA	387	265	
Tensile strength (MPa)	grain size (μm)	55	606	596	600	619	338
	100	547	NA	507	527	393	
	600	486	NA	NA	482	338	
Elongation (pct)	grain size (μm)	55	3.1	3.5	3.7	4.2	13.4
	100	2.3	NA	3.1	3.9	8.1	
	600	0.9	NA	NA	3.4	7.0	

*NA: not available.

III. RESULTS

A. Tensile Properties of Hot-Extruded Ti-46.6Al-1.4Mn-2Mo

The tensile properties of an experimental Ti-46.6Al-1.4Mn-2Mo alloy with a fully lamellar structure and various grain sizes are listed in Table I. Multiple specimens were used to generate the data in the table: three to five samples for the room-temperature testing and two to three samples for the elevated-temperature testing. At room temperature, the 0.2 pct-offset yield strength and the tensile strength varied in the ranges of 450 to 550 MPa and 490 to 610 MPa, respectively. Both strengths gradually decreased with temperatures up to 800 °C, and then decreased abruptly at 1000 °C. The temperature dependency of the strength appeared most strongly in fine-grained specimens, the probable reason being grain boundary sliding at high temperatures.

The room-temperature tensile elongation of the experimental alloy varied in the range of 1 to 3 pct, depending on the grain size. The ductility increased with decreasing grain size. Brittle cracking in TiAl alloys is known to originate from boundaries of lamellar colonies.^[13] Stress concentration for cracking increases with grain size, resulting in brittleness in the large-grained microstructure. The temperature dependence of the tensile elongation showed a trend reverse to that of tensile strength: it increased grad-

ually up to 800 °C and then abruptly to 13 pct at 1000 °C in fine-grained material.

The amount of Ti₃Al seems to be important in the tensile ductility of the TiAl-based alloys.^[14] Chan and Kim suggested^[13] that 10 to 15 pct of Ti₃Al is optimum for ductility. The Ti₃Al phase absorbs oxygen, nitrogen, and carbon interstitially, which are known to be harmful to the ductility.^[15] Too much Ti₃Al phase, however, is detrimental due to its inherent brittleness.^[16] The microstructures of the present tensile specimens contained less than 10 pct of Ti₃Al phase.

B. Reaction Couple Experiment

The basic reaction among the elemental powder mixture in the present study is reactive sintering.^[17] In order to investigate the cause of porosity, it is essential to understand the characteristics of phase transformation during reactive sintering. The reaction among particles during hot forging is hard to reproduce in the laboratory. It is possible, however, to simulate the reaction sequence and the products of the reaction during reactive sintering. For this purpose, a reaction couple experiment was conducted to determine the formation sequence and distribution of the transient phases between pure elements. In this experiment, pure Ti and molten Al were used to simulate the interaction between the elemental particles.

The reaction couple experiment was designed such that molten Al heated to 800 °C was poured into a case made of 2-mm-thick AISI 304-type stainless steel in which a plate of pure Ti was held vertically. The size of the Ti plate was 20 × 10 × 2.5 mm, and the surface was ground and cleaned by nitric acid. The molten Al immediately solidified at the surface of the Ti plate, but the extent of the reaction zone was insignificant at the interface when solidification was complete. When the bonded couple was reheated, however, a reaction zone appeared. The characteristics of the zone were studied as a function of heat-treatment conditions. Three different characteristics were exhibited: solid-state reaction between Al and Ti, partial melting of Al at the interface, and extensive melting of Al, corresponding to the heating temperatures of 620 °C, 700 °C, and 1000 °C, respectively.

When the reaction couple was heated to 620 °C for 65 hours in a vacuum of 10⁻³ torr, the reaction zone consisted of the transient intermetallic compounds of Ti and Al. It

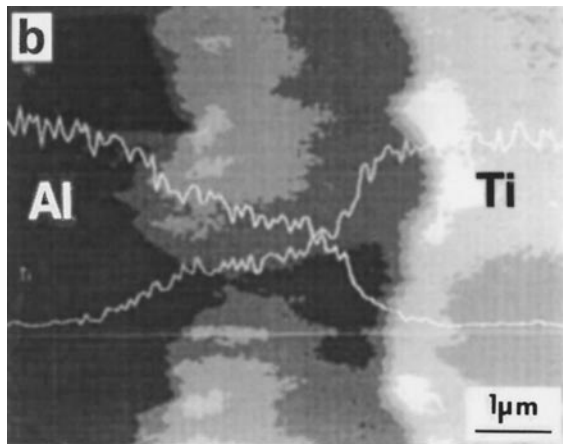
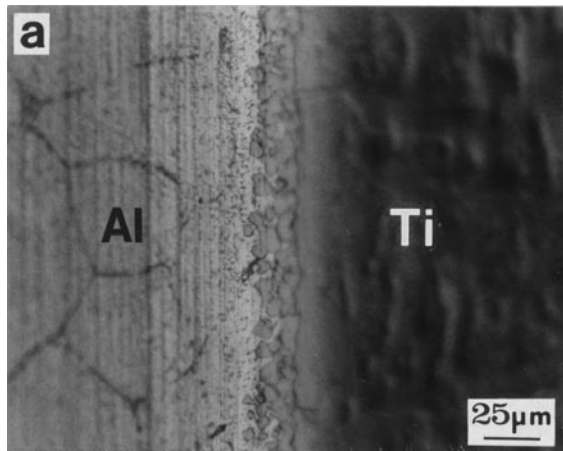


Fig. 2—Ti-Al reaction couple heated to 700 °C by the heating rate of 700 °C and air cooled: (a) light micrograph and (b) SEM image with EPMA results of Ti and Al overlapped.

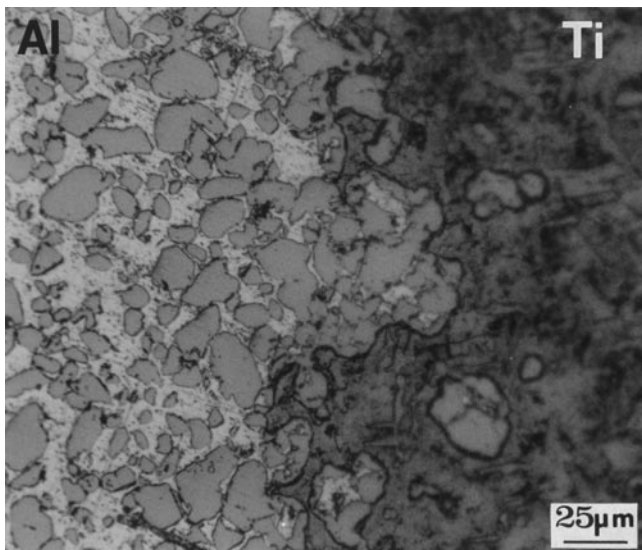


Fig. 3—Light micrograph of Ti-Al reaction couple heat treated to 1000 °C and held for 3 h. Note the advancement of the Ti front toward molten Al.

was surmised, therefore, that the transient phases also form during heating of the elemental powder mixture below the melting temperature of Al. The extent of the reaction will

increase with time, the thickness of the transient phases increasing with slow heating.

When the reaction couple was heated to 700 °C at a rate of 20 °C min⁻¹ and air cooled immediately, a diffusion of Ti toward Al was observed, as shown in Figure 2. The sequence of the reaction is led off by the formation of Ti-Al compound at the contact zone and concomitant diffusion of Ti and Al in the opposite direction. The diffusion of Ti proceeds by dissolution of pure Ti into molten Al, whereas that of molten Al occurs preferentially along the grain boundaries of solid Ti. Reaction products appear as lumpy particles near the original Ti/Al interface and a corrugated interface, as shown in Figure 2(a). An expanded view of the interface by scanning electron micrograph and the results of an electron probe microanalysis are shown in Figure 2(b).

The thickness of the interface phase was about 2 μm, and the concentration profiles of Ti and Al indicated a gradual change of the composition toward the Al side and an abrupt transition toward the Ti side. The area of flat concentration profile is presumed to be TiAl₂, whereas that of the gradual concentration transition is TiAl₃, which was also confirmed by the wavelength dispersive spectrometry analysis.

As the reaction couple was heated to 1000 °C and held for 3 hours, the reaction products penetrated deep into the molten Al side and, furthermore, they assumed a different morphology as the distance from the original Ti/Al interface increased. Near the interface, lumpy shaped particles prevailed, as shown in Figure 3, whereas a cuboidal shape and then a rodlike shape became progressively more abundant at the far end of the interface, *i.e.*, deeper into the molten Al side. It is conjectured that the diffusion front of Ti dissolved in molten Al and the concurrent growth of Ti-Al compound became increasingly anisotropic as the level of Ti supersaturation decreased.

Overall, the characteristics of the transient phases formed between Ti and Al were consistent with those identified by van Loo and Rieck.^[18]

C. The Reaction Temperatures

During heating of the elemental powder mixtures, a rigorous exothermic reaction among the pure elements is expected to occur. The time required for the reaction is very short, about 0.5 seconds, and the reaction is considered to be adiabatic. Depending on the initial state of the mixture and the heating conditions, the amount of the exothermic heat varies, which affects the phase transformation characteristics significantly. It is important, therefore, to define the characteristic reaction peaks in terms of temperature. Differential thermal analysis (DTA) was conducted to define the reaction temperatures, and the result for a blended elemental powder mixture of Ti-46.6Al-1.4Mn-2Mo is shown in Figure 4.

There was a major peak in the DTA curve associated with the reaction between the elemental powder mixture. This peak mainly corresponds to the exothermic reaction between pure Ti and Al. According to Yi *et al.*,^[12] two characteristic temperatures of the reaction may be defined with this peak: the ignition temperature (T_{ig}) and the finish (T_f) temperature. These two temperatures represent the start and completion of the reaction.

Although the major peak, as determined by DTA, corresponds to the reaction between Ti and Al, the nature of

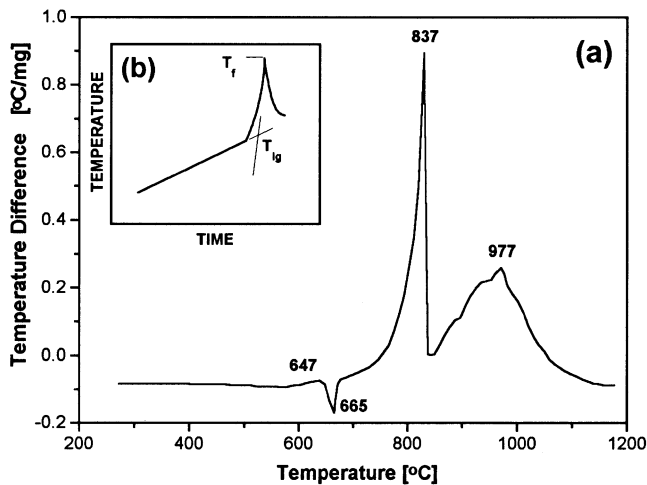


Fig. 4—The reaction temperatures of Ti-46.6Al-1.4Mn-2Mo determined by DTA: (a) typical DTA curve and (b) definition of the ignition temperature (T_{ig}) and the reaction finish temperature (T_f). The specimen for this analysis was made from elemental powder mixture compacted at room temperature.

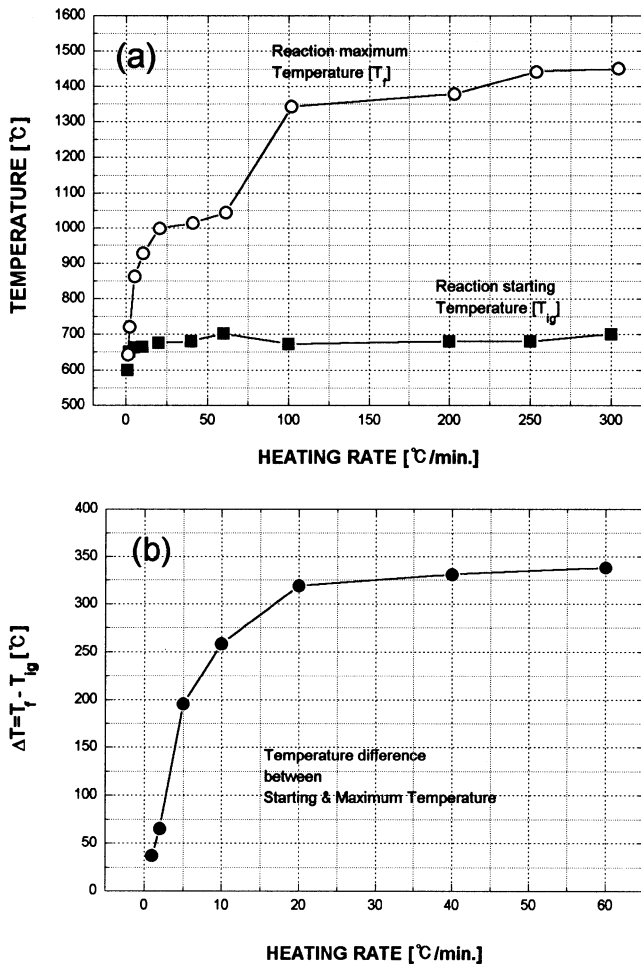


Fig. 5—Influence of the heating rate on (a) the reaction temperatures and (b) the temperature gap between the two reaction temperatures. The sample can was preheated to 500 °C for degassing prior to reaction.

the reaction is more complicated than what is represented by a single peak. Aluminum may melt and form a transient second phase with Ti prior to the main reaction. The extent

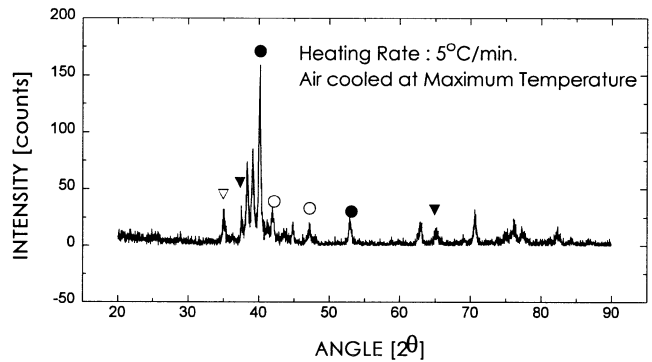


Fig. 6—X-ray diffraction spectra of Ti-46.6Al-1.4Mn-2Mo alloy made from reaction sintering of elemental powders with a heating rate of 5 °C/min. Four different phases, TiAl(\blacktriangledown), Ti_3Al (∇), $TiAl_3$ (\circ), and pure Ti(\bullet), were identified.

of these preliminary reactions will sensitively depend on the values of T_{ig} and T_f that are, in turn, affected by the heating rate.

In a separate experiment, the temperature profile with respect to a constant heating rate was obtained by directly measuring the temperature of powder compact samples. This was done by inserting thermocouples through the wall of cans containing the powder compact samples so that the temperature of the sample was directly recorded. A reaction temperature peak with essentially the same features as the case of the DTA experiment was obtained, for which the ignition temperature and the reaction finish temperature were identified. Both the ignition temperature and the reaction finish temperature increased with heating rate. As shown in Figure 5, the reaction temperatures abruptly increased with the heating rate in the range of a few degrees per minute to about 10 °C/min. Since the starting temperature of heating was 500 °C (the degassing temperature), the peak corresponding to the main reaction was rather small for the heating rate of 1 °C/min. At this heating rate, the reaction started at about 600 °C and completed at 630 °C. As the heating rate increased, the shape of the reaction peak became more pronounced and the reaction finish temperature also increased sharply. Beyond 20 °C/min, however, the rate of increase of T_f was less pronounced. Over the whole range of heating rates studied, the dependence of T_f on heating rate exceeded that of T_{ig} . The gap between the two characteristic temperatures, therefore, increased with heating rate. It increased rapidly with the heating rate up to 20 °C/min and then leveled off. The observed dependence of the reaction temperatures on the heating rate was confirmed by multiple measurements at a fixed (80 pct) powder compaction density. The compaction density may be a factor in the sensitivity of the reaction temperatures on the heating rate, which was not evaluated in this work.

D. $TiAl_3$ and Porosity

Varying the heating rate also affected the relative proportion of constituent phases. This was verified by X-ray diffraction analysis of samples which experienced different heating rates to the reaction maximum temperature. For all the heating rates employed, from 1 °C/min to 60 °C/min, the peaks corresponding to TiAl, Ti_3Al , $TiAl_3$, and pure Ti

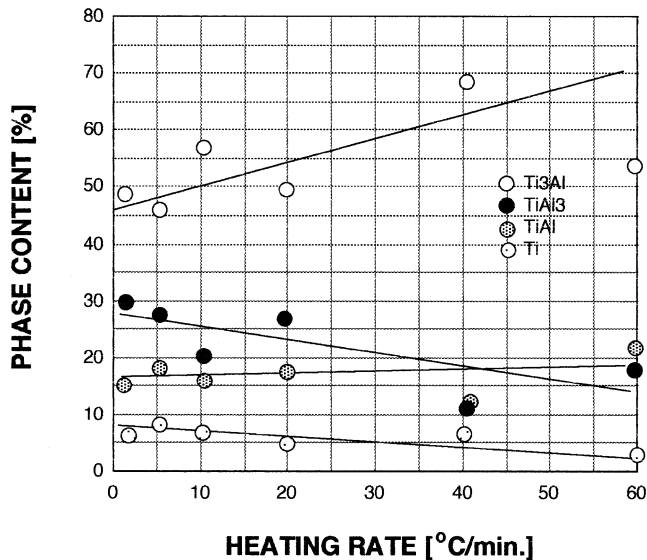


Fig. 7—Effect of heating rate on the constituent phases of Ti-46.6Al-1.4Mn-2Mo experimental alloy. The specimen, in the form of elemental powder mixture compact, was heated to the reaction finish temperature and cooled immediately.

were identified, an example of which is shown in Figure 6. The intensity of each peak varied with heating rate.

The volume fraction (V_k) of each phase (k) was calculated from the peaks according to the following formula:^[19]

$$V_k = \frac{\frac{\rho_k}{n_k} \sum_i \left(\frac{I_{ki}}{L_{ki}} \right)}{\sum_k \sum_i \frac{\rho_k}{n_k} \left(\frac{I_{ki}}{L_{ki}} \right)} \times 100 \quad [1]$$

where ρ is the density, n is the number of lattice planes used for calculation, and the values of I are the intensities of the diffraction peaks. The constant L is dependent on the lattice plane i ; therefore, L_i is defined as

$$L_i = \left(\frac{1}{v^2} m F^2 P \right) \quad [2]$$

where v is the volume of a unit cell, m is the multiplicity factor, F is the structure factor, and P is the Lorentz polarization factor.

It was found that the relative amount of TiAl and Ti₃Al increased, while that of TiAl₃ and pure Ti decreased, with heating rate. The trend of the content of each phase with varying heating rates is shown in Figure 7. In this figure, the major constituent appears to be Ti₃Al because the analysis was done for the samples heated to the reaction maximum temperature and cooled immediately. There was insufficient time for a complete reaction.

The preceding result indicates that the relative amounts of each phase vary with the heat input, which is determined by the temperature of heating and time elapsed in heating to that temperature. To verify this, another experiment was conducted to correlate the two factors with the resulting phases. The first phase of the experiment consisted of heating to temperatures above T_r at a constant heating rate of 5 °C/min and immediately cooling to room temperature. An illustration of the heating scheme is given in Figure 8(a). Since the heating rate was constant, the amount of time

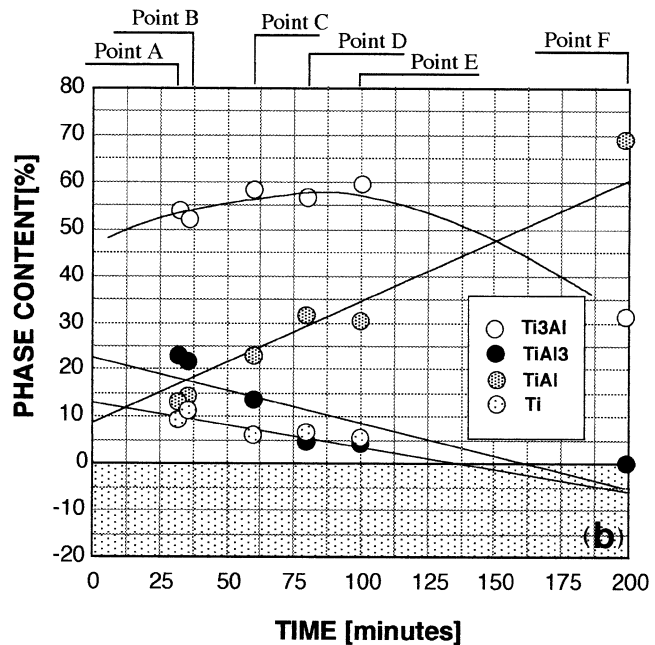
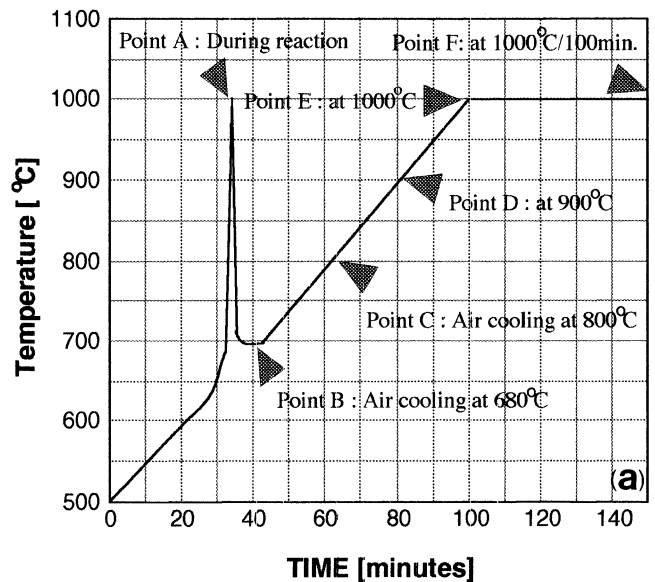


Fig. 8—Dependence of the relative amount of phases on the heating temperature in powder mixtures of Ti-46.6Al-1.4Mn-2Mo heated at a rate of 5 °C/min: (a) heating temperatures with respect to the reaction curve and (b) relative amount of phases in the samples heated to the temperatures indicated in (a). Note the maximum in the amount of Ti₃Al and the steady increase of the amount of TiAl with time elapsed in heating to each temperature.

required to reach each heating temperature was different. In this experiment, the relative amount of the resulting phases varied with the time required to reach the heating temperature, as shown in Figure 8(b). While the amount of TiAl increased steadily with the time elapsed during heating, that of Ti₃Al showed a maximum at about 90 minutes that corresponded to about 950 °C. Beyond 150 minutes, TiAl became the dominant phase.

The amount of TiAl increased not only with time of heating but also with holding time at a fixed temperature. The mixture of Ti-46.6Al-1.4Mn-2Mo was heated to 1000 °C at various heating rates ranging from 5 °C/min to 60

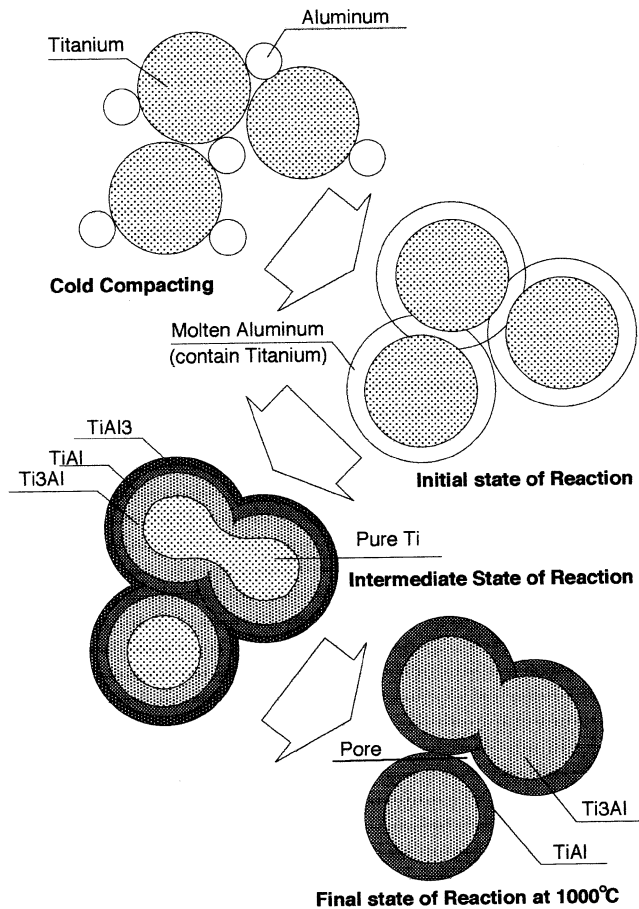


Fig. 9—Schematic model of pore formation due to the $TiAl_3$ phase during the reaction sequence among elemental powder particles.

$^{\circ}C/min$, and the sample was held for up to 2 hours. The amount of $TiAl$ phase increased with holding time, showing a parabolic trend: it increased sharply initially, then leveled off after prolonged heating. This trend was observed regardless of the heating rate. Heating to different temperatures at a fixed heating rate and holding at the temperature increased the volume percent of $TiAl$, also parabolically. Higher holding temperatures, however, resulted in a more rapid increase of $TiAl$ in the initial period of holding.

Constitution of the phases prior to forging appears to be an important factor for porosity because of its influence on Kirkendall diffusion. The relative proportion of $TiAl$ to $TiAl_3$ is particularly important since the latter phase is a source of the problematic diffusion. It was attempted to verify this hypothesis by two experiments: first, by reactive sintering using a very high heating rate to minimize the amount of $TiAl_3$ and, second, by forging samples where conditions are different in terms of phase constitution.

A high heating rate in the range of $100\ ^{\circ}C/min$ to $300\ ^{\circ}C/min$ was achieved by high-frequency induction heating. A powder mixture of $Ti-46.6Al-1.4Mn-2Mo$ was induction heat treated to $1000\ ^{\circ}C$ and held for 10 minutes. The resulting sample contained virtually no $TiAl_3$ and the major phase was Ti_3Al . Forging the sample in this condition reduced pores significantly. This result indicates the possibility of an efficient processing of $Ti-46.6Al-1.4Mn-2Mo$ without prolonged holding at a high temperature.

Since the $TiAl_3$ phase can also be reduced by extended

holding above the reaction finish temperature, a series of forging experiments was done at different holding times to check the porosity. Samples were heated to $1000\ ^{\circ}C$ at a rate of $5\ ^{\circ}C/min^{-1}$, held for different amounts of time, and then forged. Three holding times were selected: 30, 160, and 300 minutes. In the microscopic examination of open pores, the porosity in the forged samples decreased by about 60 pct in the latter two extended holding conditions.

IV. DISCUSSION

The source of pores in a reactively sintered $Ti-46.6Al-1.4Mn-2Mo$ alloy appears to be the transient phase, a representative one being $TiAl_3$. It must be noted, however, that the route of pore formation is based on the sequence of the transient phase formation. Therefore, it is of interest to schematically model the sequence of alloying reaction in the powder mixture.

During heating of the powder mixture, pure elemental particles of Al and Ti react in solid form. The extent of this solid reaction, however, is dependent on the rate of heating. Slow heating provides for more time of reaction, resulting in a larger amount of transient phases. The compound form of the transient phases is mainly $TiAl_3$. Melting of Al , which did not participate in the solid reaction, accelerates the sintering reaction. Either the solid reaction or the liquid-solid reaction results in the transient phase, and this reaction occurs before the main exothermic reaction between pure Ti and Al .

When the temperature reaches the reaction initiation temperature (T_{ig}), the reaction between the elemental powders occurs simultaneously with the reaction between the solid reaction products and Ti . When this reaction takes place, the diffusion route of Al is through the transient phases toward pure Ti . This results in Kirkendall porosity since Ti , being the minor element in the compound $TiAl_3$, diffuses slowly.^[20-23] The sequence of alloying reaction during reactive sintering is depicted in Figure 9 as a schematic model.

Since the source of the porosity during reactive sintering of the powder mixture of $Ti-46.6Al-1.4Mn-2Mo$ composition is the transient phase, two possible solutions exist in industrial production of this alloy. The first method is to reduce the amount of transient phases by fast heating. Fast heating reduces the extent of the solid reaction prior to the main exothermic reaction. The second method is to hold the alloy at a temperature above the reaction peak for a long enough time to transform the transient phases to stable $TiAl$. A sufficient heat input will reduce the amount of transient phase, thus minimizing the porosity during the next stage of processing, namely, hot extrusion or hot forging.

For high-temperature applications of the present experimental alloy, a concern arises regarding the presence of unstable beta (β) phase. In contrast to Mo -containing β titanium alloys of good strength and high-temperature formability,^[24,25,26] the β phase in the present experimental alloy was observed as islands in the as-extruded condition, an example being shown in Figure 10(a). This phase was found in the areas enriched with Mo . During homogenization, however, the β phase disappeared when the annealing temperature was sufficiently high or the cooling rate was low. For example, the β phase existed in the sample homogenized at $1200\ ^{\circ}C$ but not in the one homogenized

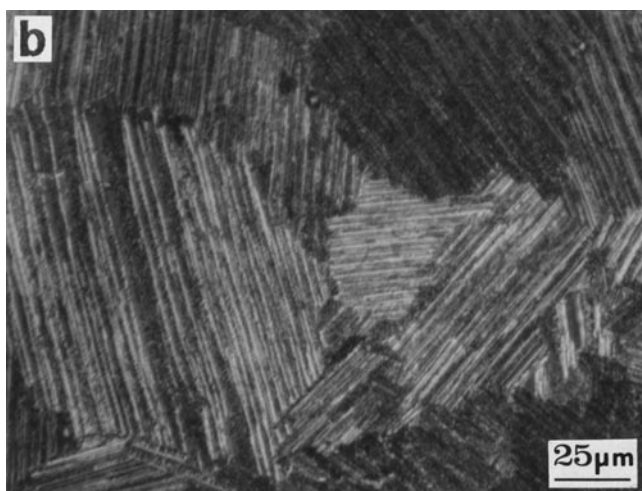
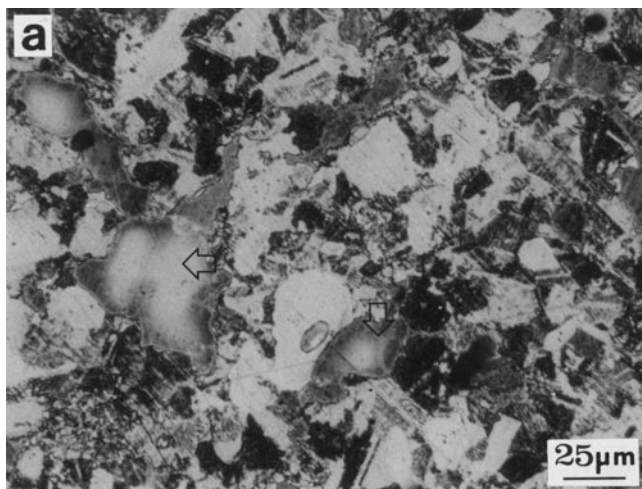


Fig. 10—Behavior of β phase in experimental Ti-46.6Al-1.4Mn-2Mo alloy produced from elemental powder mixture: (a) β phase (marked by arrows) in as-extruded (1230 °C) condition and (b) absence of the β phase in a specimen heat treated (1400 °C 1 h/cooled at 400 min⁻¹) to give the fully lamellar microstructure.

at 1300 °C. Since the heat treatment for the fully lamellar structure involves high-temperature exposure, the β phase is usually absent in the final heat-treated condition. To ensure a complete removal of the β phase prior to the heat treatment, the alloys should be annealed at a sufficiently high temperature. Excessive exposure to the high temperature, however, causes general grain growth and lamellar coarsening. Therefore, these two factors—the amount of β phase and the grain structure—must be optimized. An example of such a microstructure is shown in Figure 10(b).

V. CONCLUSIONS

From the present study on the experimental Ti-46.6Al-1.4Mn-2Mo alloy processed by EPM, it was concluded that the existence of porosity during reactive sintering results from the presence of transient phases such as TiAl₃. Since these are the source of Al in Kirkendall diffusion toward elemental Ti particles, the transient phases result in voids. The porosity, therefore, can be minimized by the reduction of such phases. Two possible remedies were offered: first, a

fast heating rate to the temperature for consolidation and, second, a sufficient amount of soaking at a temperature above the reaction peak. A forging experiment and an induction heating experiment designed according to this concept verified the proposed hypothesis. The Mo-rich β phase in this experimental alloy, which is of concern for high-temperature application, could be controlled by heat treatment.

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