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Some Reactive Processing Aspects of High-Temperature Aluminides Nb_3Al and RuAl

K.E. Mohamed, D. Stover, and H.P. Buchkremer

In the present investigation elemental powder mixtures with nominal composition of Nb-25at.% Al and Ru-50at.% Al corresponding to Nb_3Al and RuAl intermetallic phases were prepared. Green compacts from these mixtures were reactive sintered in vacuum or hot isostatically pressed after being encapsulated and reacted in quartz capsules.

In the absence of pressure, reactive sintering at temperatures up to 1800 °C did not lead to densification, and swelling effects were observed. Reactions of formation of Nb_3Al and RuAl were found to take place at different temperature regions characteristic for each aluminide system, with clear dependence of the reaction temperature on the heating rate of the powder mixtures. Dilatometry studies showed that swelling occurred instantaneously at temperatures corresponding to the reaction temperature and led to a volume increase of about 25% and 42% for Nb_3Al and RuAl, respectively.

The activation energy for the reactions was estimated from the differential thermal analysis. Similar data for the NiAl intermetallic phase were also obtained for comparison. The densification behavior and the phase composition of the reaction products were investigated and correlated with the heating rate, the mutual solubility of the reactants, and the amount of liquid phase available during the reaction.

Keywords hot isostatic pressing, Nb_3Al, reactive sintering, RuAl

1. Introduction

High-temperature intermetallic compounds have the potential for high specific strength and high specific elastic moduli at elevated temperatures. Those based on aluminum have the additional attractive characteristics of low density, good corrosion, and oxidation resistance. These compounds are therefore being developed and characterized for future aerospace and turbine applications.

Reactive sintering is an advantageous powder processing method that is especially suitable for producing intermetallics and intermetallic-matrix composites. Producing such high-melting-point materials by conventional melting and casting is always associated with numerous difficulties, including compositional inhomogeneities, microstructural coarsening, porosity, and cracking. Also, hot isostatic pressing (HIP) of prealloyed intermetallic powders, particularly those of high melting points, requires high pressures and temperatures and usually produces porous materials (Ref 1-5).

In reactive sintering processes, the intermetallic compound is formed through an exothermic reaction between the constituent elemental powders in the compact. As a consequence, a certain amount of heat is generated that depends on the heat of formation of the intermetallic phase. When the temperature of the compact exceeds a lower temperature eutectic in the system, or exceeds the melting point of one of the components, a temporary liquid is formed that aids in densification of the compact. Essential for the effectiveness of this temporary liquid phase, regarding densification, is the amount and duration of this phase and its distribution during the sintering cycle. These factors depend on some parameters of the system, such as the reaction temperature, the sintering temperature, the heating rate, the amount of the low-melting-point component, and the particle size ratio of the powders.

The objective of the current work was to investigate the possibility of reactive processing for two intermetallic compounds with high melting points, RuAl (2000 °C) and Nb_3Al (2060 °C), and to use differential thermal analysis (DTA) and x-ray diffraction (XRD) to analyze the synthesis reactions of these intermetallic phases. For the sake of comparison, DTA experiments were also performed on elemental powder mixtures of the NiAl intermetallic phase.

Ruthenium aluminide, RuAl, can be considered one of the most promising high-temperature intermetallics concerning strength, stiffness, toughness, and oxidation resistance (Ref 6-11). The work conducted by Fleischer and McKee (Ref 11) on arc-melted specimens of RuAl, with or without small amounts of a third element, has emphasized the role of the alloy chemistry in determining its properties. The best combination of low oxidation rates and high toughness was given by Ru-53Al-47 and Ru-51Al-48Y compositions.

Several investigations have been performed on reactive sintering of niobium and aluminum powders. However, most of these studies were related to $NbAl_3$ with almost no information regarding Nb_3Al stoichiometry. Schulze et al. (Ref 12) studied sintering of niobium with 5 at.% Al and observed that the intermetallic phase $NbAl_3$ was formed when molten aluminum contacted solid niobium particles. Others reacted niobium and aluminum powders to form porous $NbAl_3$ sponge that was later ground into powder (Ref 2, 3).

The work on reactive sintering of $NbAl_3$ (Ref 13-15) showed that typical densities obtained after reactive sintering were between 85 and 95% of theoretical. Near-full density was obtained after reactive hot pressing. These investigations emphasized the role of certain processing parameters, such as the particle size ratio of niobium and aluminum, initial

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stoichiometry, heating rate, and sintering temperature, during reactive processing of this intermetallic compound.

2. Experimental Approach

The starting elemental powders were –40 mesh niobium powder (<350 μm), ruthenium (6 μm average particle size), Inco 123 Ni (3–7 μm) and gas-atomized aluminum powder of 20 μm average particle size. The elemental powders were mixed in the ratio of 91.2 wt% Nb to 8.8 wt% Al, 78.93 wt% Ru to 21.07 wt% Al, and 69 wt% Ni to 31 wt% Al. These mixtures correspond to the stoichiometric compositions of the intermetallic compounds Nb_3Al, RuAl, and NiAl, respectively. The three powder mixtures were individually blended in a turbula mixer for about 6 h. Green compacts were made by putting the powder mixture in a polyurethane rubber die, sealing, and cold isostatic pressing at 300 MPa.

For reactive sintering, green compacts were heated in vacuum from room temperature at a specified heating rate to the sintering temperature, then held for 2 h after which the furnace was turned off. For Nb_3Al green compacts this cycle included additional hold time for 10 h at 500 °C for degassing. To make a reactive HIP experiment, the green compact was wrapped in molybdenum foil and encapsulated under vacuum in a quartz or tantalum capsule. The quartz capsules were heated at 0.25 K/s to slightly above the reaction temperature, held for 30 min, and then heated to a higher sintering temperature as pressure was applied gradually. At the reaction temperature the pressure was about 3 to 5 MPa. At about 1400 °C, the pressure was increased to 20 MPa and held for 10 min after which it was raised to the planned value for the HIP cycle. For the tantalum capsule, a heating rate of 0.17 K/s was applied to slightly above the reaction temperature. The temperature was raised to 1600 °C. The pressure was about 3 MPa up to about 800 °C and it was then gradually increased to 180 MPa.

The exothermic reaction for the three powder mixtures (Nb_3Al, RuAl, and NiAl) was analyzed by DTA at various heating rates ranging from 0.05 to 0.5 K/s. A dilatometer facility attached to the HIP machine was used to measure the dimensional changes during the synthesis reaction of the Nb_3Al and RuAl green compacts.

The microstructure was evaluated by optical and scanning electron microscopy, and the phases were identified by XRD.

3. Results and Discussion

3.1 *Heating Rate and the Reaction Temperature*

Figure 1 shows the DTA traces for elemental powder mixture samples corresponding to NiAl, RuAl, and Nb_3Al phases. These samples were heated in vacuum with heating rates varying from 0.05 to 0.5 K/s.

Within the experimental conditions investigated, the Ni-Al system showed only one exothermic peak (Fig. 1a), indicating the reaction between nickel and aluminum powders. The temperature of onset of the reaction (first deviation from the baseline) was about 625 °C and was nearly independent of the heating rate. The reaction peak temperature, on the other hand, showed a slight dependence on the heating rate, increasing from 630 °C at 0.08 K/s to about 645 °C at 0.42 K/s.

For the Ru-Al system (Fig. 1b), two exothermic peaks were observed when the sample was heated at a rate of 0.17 K/s or lower. Gradual change to the one-peak behavior was observed upon increasing the heating rate. Unlike the Ni-Al system, marked dependence of the initiation temperature and the peak temperature of the reaction was observed for the Ru-Al system, with the initiation temperature increasing from 600 to 650 °C and the peak temperature increasing from 620 to 665 °C when the heating rate was increased from 0.08 to 0.42 K/s. The second peak for the two-peak traces reached the same temperature for 0.08 and 0.17 K/s heating rates.

Samples of the Nb-Al system (Fig. 1c), heated at rates varying from 0.05 to 0.5 K/s, showed different behavior with respect to Ni-Al and Ru-Al systems. Regardless of the heating rate used, an endothermic peak was observed at about 660 °C, corresponding to the melting of aluminum, followed by an exothermic peak at higher temperatures. The onset temperature of the reaction, as well as the reaction strength (indicated by the

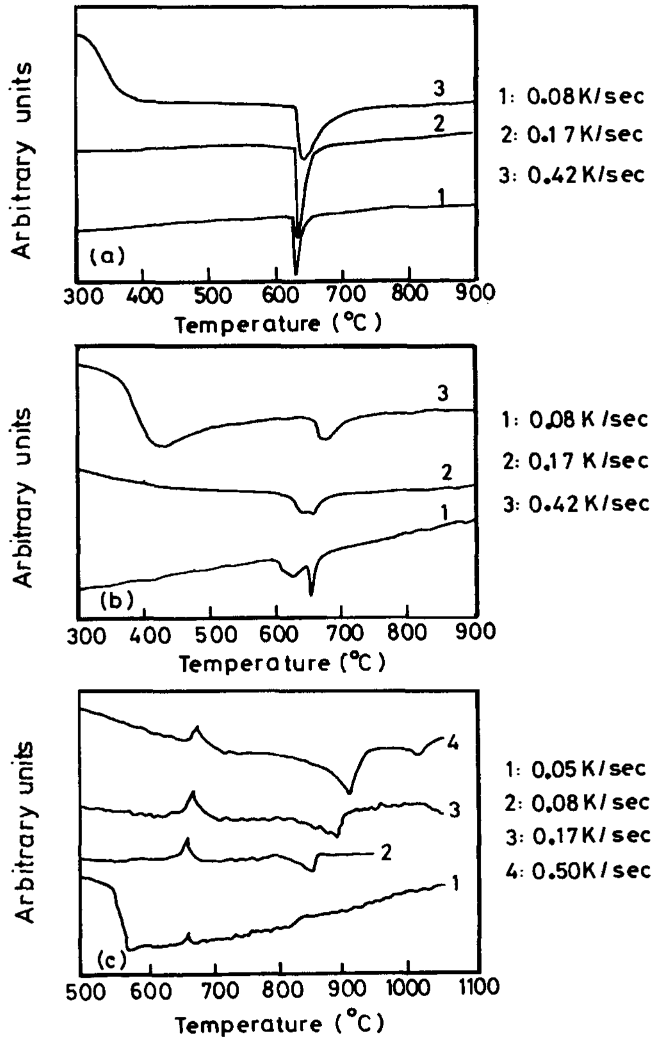

Fig. 1 Differential thermal analysis traces at different heating rates for elemental powder mixtures of (a) NiAl, (b) RuAl, and (c) Nb_3Al

peak size), increased with faster heating rates. For the lowest heating rate, 0.05 K/s, there was only a slight indication of a reaction taking place at about 815 °C. The initiation temperature of the reaction increased from 825 to 885 °C and the reaction peak temperature increased from 855 to 910 °C as the heating rate was increased from 0.08 to 0.5 K/s.

For the Ni-Al system and the Ru-Al system the initiation temperatures of the reaction lie below the lowest eutectic of the Ni-Al system (640 °C) and below the melting point of aluminum for the Ru-Al system. Thus, the reactions were initiated in the solid state. The temperature increase of a Ni-Al compact during the exothermic reaction was found to be a function of the heating rate and the aluminum content (Ref 16). For a heating rate of 0.08 K/s and 30 at.% Al, the increase in temperature was about 800 °C (Ref 16). In the present study, with about 50 at.% Al and higher heating rates, a much higher increase in the actual temperature of the powder sample was expected. Although the reaction between nickel and aluminum powders started in the solid state, it was completed in the presence of a liquid phase. The work of Philpot and Munir (Ref 16) showed that the Ni-Al system exhibited two exothermic peaks for heating rates equal or less than 0.03 K/s for the smaller aluminum contents. This indicates that the two-peak behavior is related to the strength of the reaction through dependence on the reaction temperature and the amount of liquid formed. Thus, for the Ni-Al samples in the present work, a two-peak behavior should not be expected.

The appearance of two exothermic peaks in the present work for the Ru-Al system at 0.08 and 0.17 K/s and the transition to the one-peak behavior at 0.25 K/s can also be interpreted in terms of an increase in the strength of the reaction with increased heating rate. It is therefore possible to suggest that when the heat generated by the exothermic reaction initiated in the solid state is high enough for the formation of a liquid phase in the system, the reaction becomes more rapid and the exothermic behavior changes to the one-peak regime. This is supported by the fact that the peak temperature of the reaction (when only one peak exists) lies at about 665 °C where aluminum melts, and the fact that the actual temperature of the sample is definitely higher due to the heat generated during the reaction.

The existence of an endothermic peak corresponding to the melting of aluminum before the exothermic reaction starts in the Nb-Al system suggests that the reaction in this case can only take place between solid niobium particles and liquid aluminum. This is confirmed by the results of Murray et al. (Ref 13) and German et al. (Ref 14) during reactive synthesis of $NbAl_3$. In their investigations, although they used niobium powder of much finer particle size (<44 μm) than we have used in the present work (<350 μm), they found that the reaction between niobium and aluminum is initiated at temperatures higher than the melting point of aluminum. It seems that this behavior is characteristic of the Nb-Al system rather than an effect of niobium particle size.

3.2 *X-Ray Diffraction Analysis*

X-ray diffraction analyses were performed on the powder mixture samples after they had been heated through their reaction temperatures. The results are given in Fig. 2. For the Ni-Al samples heated at 0.25 K/s, sharp NiAl peaks were observed (Fig. 2a). No peaks for nickel, aluminum, or other phases could be seen. Also, for the Ru-Al samples heated at 0.25 K/s, the primary phase formed was RuAl, with little traces of unreacted ruthenium (Fig. 2b). In contrast, when Ru-Al samples were heated at a slower rate (0.08 K/s, where two exothermic peaks were observed), two phases were formed: $RuAl_2$ and RuAl, as shown in Fig. 2(c). From the relative intensities in Fig. 2(c), it is clear that the amount of RuAl is higher than that of $RuAl_2$. An x-ray diffractogram of the Nb-Al sample heated at 0.25 K/s (Fig. 2d) exhibited a wide spectrum of phases, although it showed only one exothermic peak after melting of aluminum. The major x-ray peaks observed are those of $NbAl_3$ and unreacted niobium, while the minor phases are Nb_2Al and Nb_3Al.

From these observations we anticipate that for the Ni-Al and Ru-Al samples heated at 0.25 K/s, where only one exothermic peak was observed by DTA, the main reaction products are NiAl and RuAl. For the Ru-Al sample, heated at a rate of 0.08 K/s, where two exothermic peaks were observed (see Fig. 1b), the first exothermic reaction was initiated and reached its peak before the formation of a liquid phase. When the heat from the sample generated by the exothermic reaction superimposed on heating from the furnace is sufficiently high to exceed the melting point of aluminum, the liquid phase formed in the system enhances the reaction rate and the second phase forms. Thus, the two-phase reaction product in this case (Fig. 2c) is in agreement with the two-peak regime observed for this sample. The relatively higher amount of RuAl phase compared to $RuAl_2$ indicates that the major portion of ruthenium and aluminum powders in this sample reacted through the liquid phase reaction corresponding to the second exothermic peak.

X-ray analysis of the Nb-Al sample (Fig. 2d) showed the presence of three different niobium aluminide compounds in addition to unreacted niobium, although it was heated at the same heating rate (0.25 K/s) as the Ru-Al and Ni-Al samples, which gave a more or less single-phase reaction product. The high reaction temperature of the Nb-Al system and the polyphase reaction product with one exothermic peak indicate that solid-state interdiffusion processes, as well as diffusion in molten aluminum before the exothermic reaction takes place, play a significant role in this case. The small amount of liquid available for the Nb_3Al stoichiometry (8.8 wt% Al) and the high reaction temperature of this system slow down the reaction and can explain the formation of a polyphase reaction product in the Nb-Al sample. The presence of unreacted niobium in the reaction product may be explained by the formation of large amounts of the high-aluminum phase $NbAl_3$, which consumes a considerable portion of aluminum necessary for complete reaction with niobium. Unreacted niobium can also result from the coarse niobium powder particle size used in the present work. With large niobium powder size an intermetallic phase layer can form between niobium and aluminum, which can hinder further diffusion and reaction, leading to incomplete reaction with niobium. A similar effect was found for iron powders during in situ microfusion processes in the Fe-Al system (Ref 17).

3.3 *Activation Energy of the Reactions*

In Fig. 3 the logarithm of the heating rate is plotted versus the inverse absolute values of the initiation temperature (Fig.

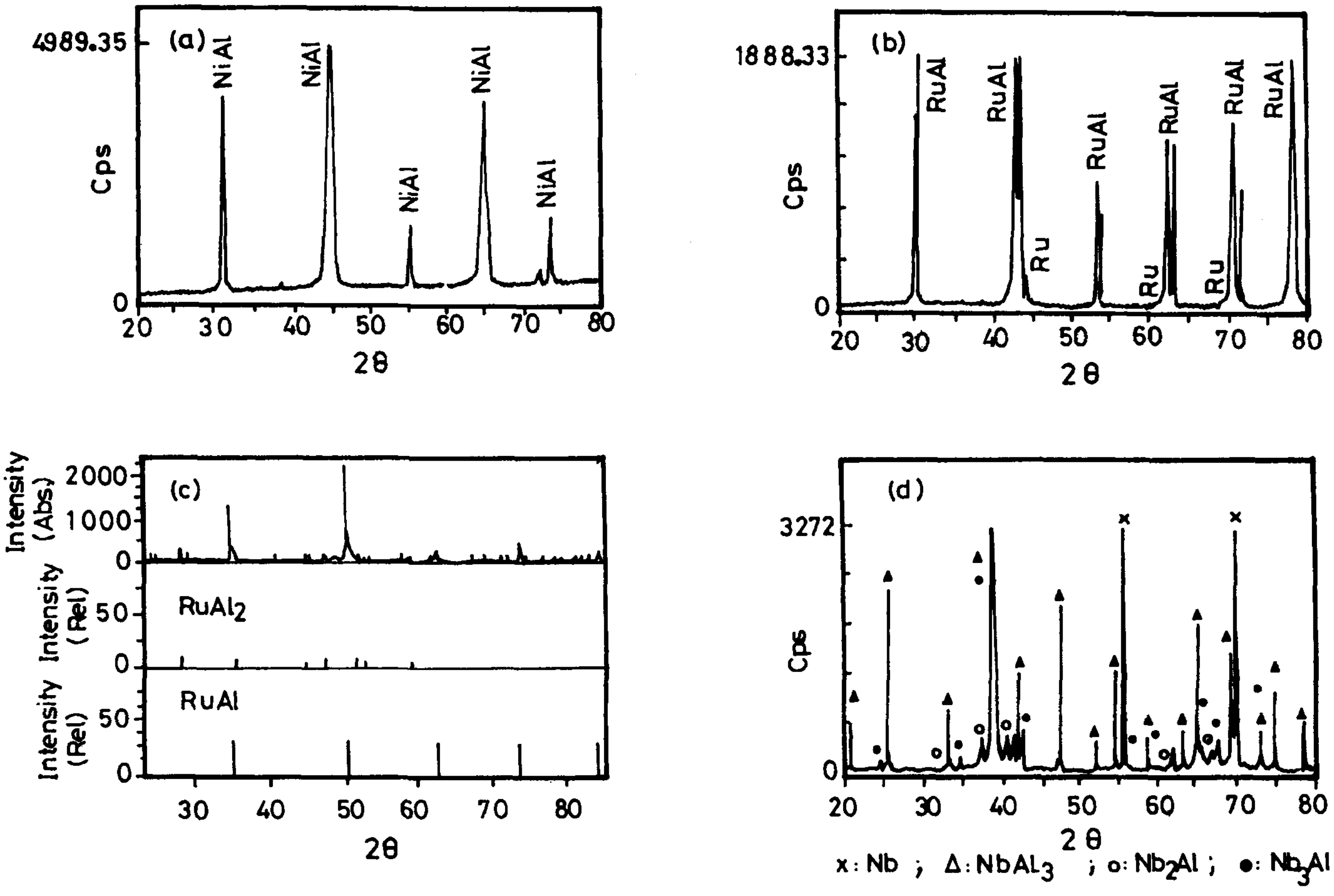

Fig. 2 X-ray diffraction patterns of elemental powder mixtures heated through their reaction temperatures. (a) Ni-Al, 0.25 K/s. (b) Ru-Al, 0.25 K/s. (c) Ru-Al, 0.08 K/s. (d) Nb-Al, 0.25 K/s

3a) and the reaction peak temperature (Fig. 3b). A linear relationship was found and the associated activation energies are given in Table 1.

The high activation energy calculated for the Ni-Al sample compared to that for the Ru-Al and Nb-Al samples can be attributed to the fact that a sufficient amount of liquid formed rapidly in the early stage of the reaction throughout the whole range investigated of the heating rates, thus enhancing the reaction and leading to near independence of the initiation temperature from the heating rate. The low values of activation energy for the reaction initiation (234 and 202 kJ/mol, in the case of the Ru-Al and Nb-Al samples) are typical of the chemical reaction between metals initiated in the solid state. As discussed above, for the Ru-Al samples it was found that the reaction was initiated and reached its first peak, in the two-peak regime, before the formation of liquid, while for the second peak or the only peak (in the one-peak regime) a liquid existed in the system during the reaction. This can explain the relatively large increase in the activation energy value from 234 to 470 kJ/mol for the Ru-Al samples when the reaction peak temperature is considered. For the Nb-Al samples, because the reaction between niobium and aluminum takes place at temperatures higher than the melting point of aluminum, a liquid phase is expected to be present in the system during the reaction. However, because of the small volume fraction of liquid for the Nb_3Al stoichiometry (23%) and the big temperature difference between the melting point of aluminum and the onset of the synthesis reaction, a considerable amount of the liquid phase is consumed through interdiffusion processes and vaporization before the start of the reaction. Thus, a very limited amount of liquid is expected to be present during the initiation of the reaction, and a strong dependence of the initiation and peak temperatures of the reaction on the heating rate is expected. This explains the very slight difference in the activation energy values for the reaction initiation and reaction peak given in Table 1 for the Nb-Al system.

3.4 *Densification Behavior of RuAl and Nb_3Al*

3.4.1 Reactive Sintering

Green compacts of the elemental powder mixtures of RuAl and Nb_3Al were heated at different heating rates ranging from 0.05 to 0.5 K/s to sintering temperatures varying from 900 to 1800 °C in vacuum, held for 2 h, and furnace cooled. The specimens expanded during sintering and no densification was observed. The degree of swelling was higher in the RuAl samples compared to the Nb_3Al samples. Representative microstructures of specimens sintered at 1800 °C are shown in Fig. 4. The specimens were porous and contained some dense areas. Coarse isolated pores were observed for the Nb_3Al sample, while a coarse porosity network was observed for the RuAl sample, indicating the higher degree of expansion.

A dilatometer facility installed on the HIP machine was used to measure the dimensional change of the green compacts during reactive sintering. Heating in vacuum was not possible when the dilatometer was used and the specimens were heated at 0.08 K/s in 2 MPa argon atmosphere. Specimen expansion and temperature against time are shown in Fig. 5 for Ru-Al and Nb-Al compacts. For both materials, no dimensional change was seen up to the reaction temperature. Sudden expansion was observed at about 590 °C for the Ru-Al compact and at about 750 °C for the Nb-Al compact, corresponding to the start of the exothermic reaction for both systems. These temperatures are slightly lower than the initiation temperatures obtained from DTA analysis for the same heating rate. The delayed initiation of the reactions observed during DTA experiments is attributed to the fact that loose powder samples were used, whereas compacts prepared by cold isostatic pressing at 300 MPa were used for dilatometry investigations. The volume increase due to the observed dimensional change was about 42% for the Ru-Al compact and about 25% for the Nb-Al compact. As the temperature was increased very little dimensional change was observed, implying that no densification takes place during the high-temperature solid-state diffusion processes.

During reactive sintering, the densification process relies on the presence of a temporary liquid phase. The density and quality of the microstructure depend strongly on the amount of the liquid phase available in the system, its distribution, and its duration. The processing conditions that influence the reaction will also alter these liquid parameters. These processing conditions include the heating rate, the particle size and particle size ratio of reacting species, the green density of the compact, and the sintering atmosphere (Ref 18-27).

In the present investigation, the Ru-Al samples exhibited a high degree of swelling, reaching about 42 vol%, and showed a coarse porosity network (Fig. 4a). The volume fraction of the liquid phase for the RuAl stoichiometry was about 55%. This is comparable with the 60% liquid phase for the NiAl, which showed good reactive sintered density (Ref 28, 29). The enthalpy of formation of RuAl is about 124 kJ/mol (Ref 30), similar to that of NiAl, reported to be about 118 kJ/mol (Ref 31). Also, the Ni_3Al stoichiometry, with about 34 vol% liquid phase and a heat of formation of 153 kJ/mol, showed good reactive sintered density (Ref 20, 27). The green density of the Ru-Al compact was about 75 to 77% of theoretical and was suitable for good distribution of the liquid phase. The mean particle size of ruthenium and aluminum powders used in the present work is about 6 and 20 μm; however, agglomeration of ruthenium during mixing is likely to occur and can alter the actual particle size ratio of ruthenium and aluminum in the compact. Thus, although some processing conditions (including the particle size range, the amount of liquid phase, and the enthalpy of formation) were similar for the Ru-Al and Ni-Al systems, the latter system showed better reactive sintered density, while the Ru-Al system exhibited a high degree of swelling.

Several reasons can be suggested to explain the observed swelling during reactive sintering of Ru-Al: (a) the widely different diffusion rates of ruthenium and aluminum; (b) the formation of RuAl phase, which has greatly different lattice parameters compared to those of ruthenium and aluminum; (c) nonuniform distribution of the liquid phase due to an unsuitable powder particle size ratio of ruthenium and aluminum; and (d) very limited solubility of aluminum in ruthenium (2 at.%) at the reaction temperature. It is not clear, however, at the present time, which one of these reasons plays the major role for the poor densification behavior observed.

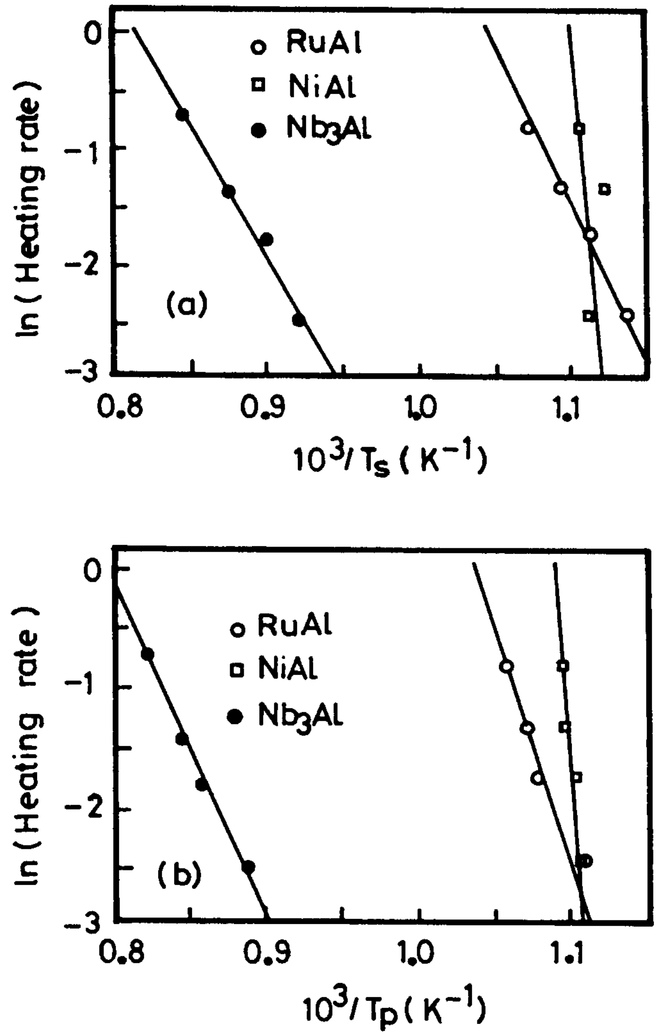

Fig. 3 The relationship between the initiation temperature (T_S), the peak temperature (T_P), and the heating rate

Table 1 Activation energy (Q) calculated from the plots in Fig. 3

Powder mixture	Q, kJ/mol, onset of reaction	Q, kJ/mol, reaction peak
NiAl	1078	1293
RuAl	234	470
Nb_3Al	202	234

The Nb-Al samples exhibited less swelling, 25 vol%, than RuAl. This is also emphasized by comparing the micrographs of Fig. 4. The Nb_3Al stoichiometry has about 23 vol% liquid phase at the melting point of aluminum, which is partially consumed by evaporation and interdiffusion processes during

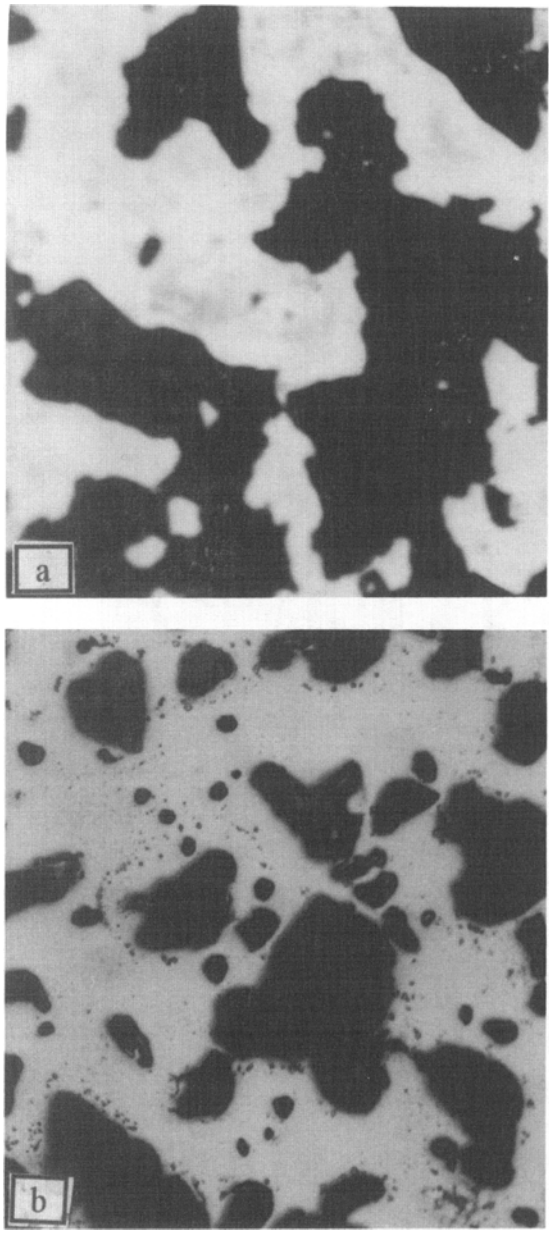

Fig. 4 Microstructure of sintered elemental powder compacts of (a) RuAl and (b) Nb_3Al. ×200

heating to the reaction temperature. Thus, the volume fraction of the liquid phase available during the reaction is less than 23% . This is much lower than the volume fraction of the liquid phase available for systems that show good reactive sintered density, for example, 60% for NiAl (Ref 28, 29), 34% for Ni_3Al (Ref 20), and 74% for $NbAl_3$ (Ref 13). As mentioned above, the importance of the amount of liquid phase for good densification during reactive sintering is strongly stressed in the literature. A decrease in the aluminum content from 13 to 11 wt% in the Ni_3Al stoichiometry increased the sintered porosity from 3.5% to about 14% (Ref 20). It can be concluded, therefore, that the main reason for the swelling observed during reactive sintering of Nb_3Al is the insufficient amount of liquid available in this system, as well as its poor distribution due to the coarse niobium powder used (<350 μm). In Ref 13, the success achieved during reactive sintering of $NbAl_3$ phase was mainly due to a high volume fraction of liquid and the use of fine niobium powder (44 μm). This minimizes the role of solubility of aluminum in niobium (about 5 at.% at the reaction temperature) and the difference in diffusion rates of these elements as possible reasons for the poor densification observed during reactive sintering of Nb_3Al.

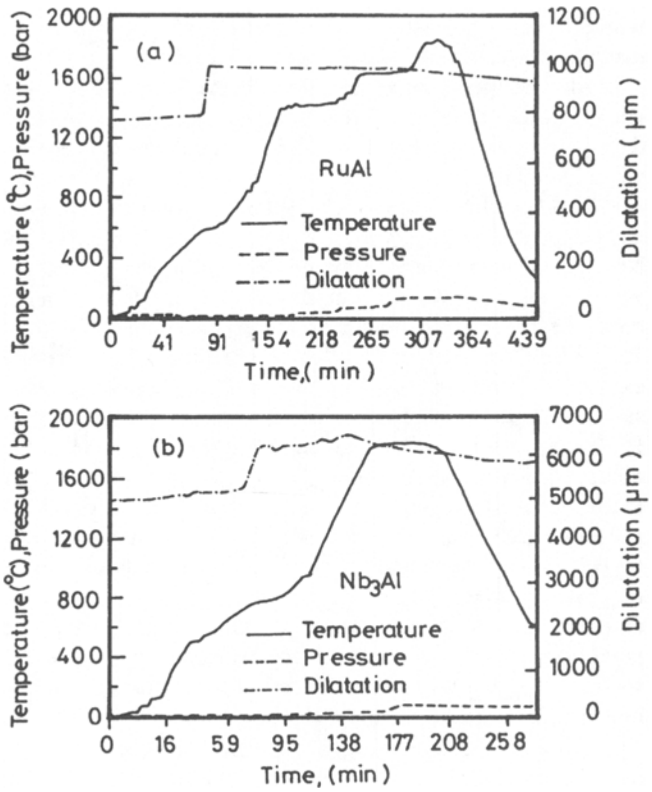

Fig. 5 Plot of temperature, pressure, and linear expansion against time during dilatometry experiments for (a) RuAl and (b) Nb_3Al

3.4.2 Effect of Applying Pressure

Additional driving force for densification can be provided by applying external pressure during the reactive sintering process. For this purpose, green compacts of RuAl and Nb_3Al elemental powder mixtures were wrapped in molybdenum foil, vacuum encapsulated in quartz or tantalum capsules, and hot isostatically pressed at different temperatures and pressures. Quartz and tantalum capsules were used to enable application of pressure at high sintering temperatures. Sample microstructures of RuAl reactive hot isostatically pressed at different conditions are shown in Fig. 6. Although the material is not fully dense, a significant increase in density is evident compared to the pressureless reactive sintered RuAl (Fig. 4a).

It is important to note that a change in HIP conditions or capsule material was not accompanied by large changes in density. Moreover, the quartz capsules were found to be cracked after the HIP cycle.

From the dilatometry results (Fig. 5a) it was concluded that the volume of RuAl compact increased suddenly by about 42% at the reaction temperature, which was found to be about 600

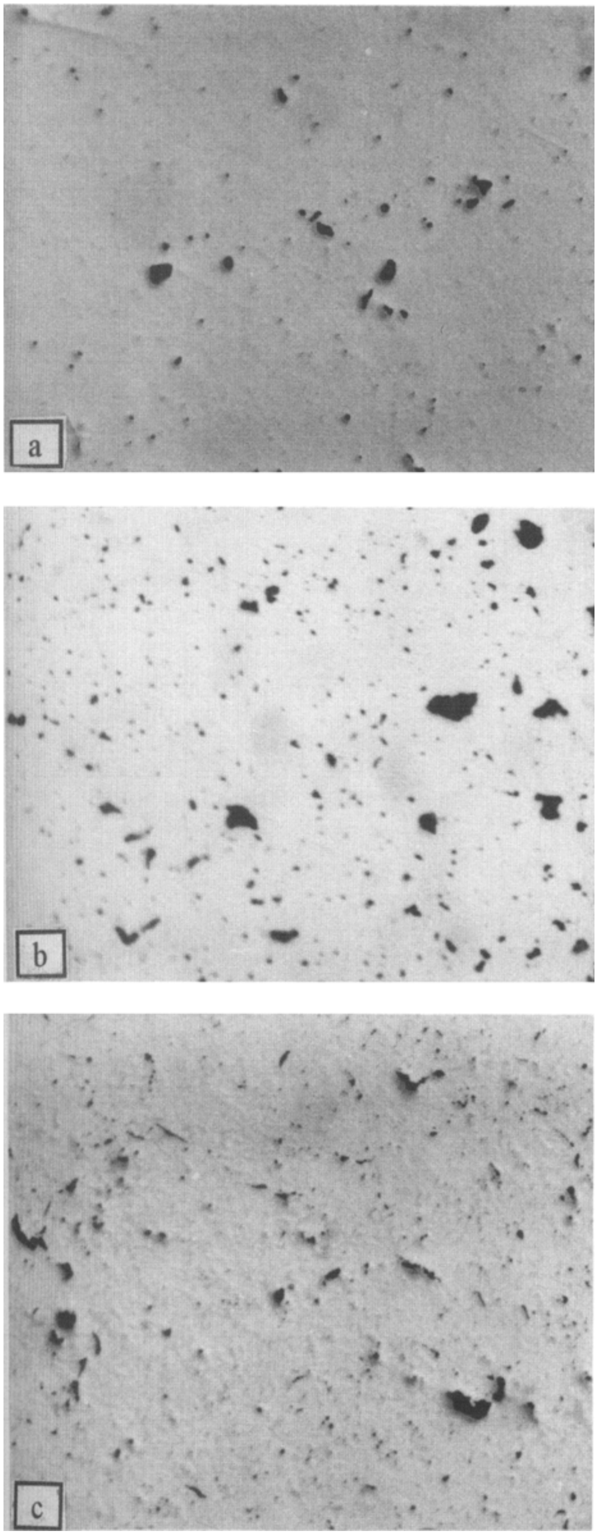

Fig. 6 Microstructure of reactive hot isostatically pressed elemental powder compacts of RuAl. (a) 1800 °C, 180 MPa, quartz capsule. (b) 1750 °C, 320 MPa, quartz capsule. (c) 1650 °C, 180 MPa, tantalum capsule. ×200

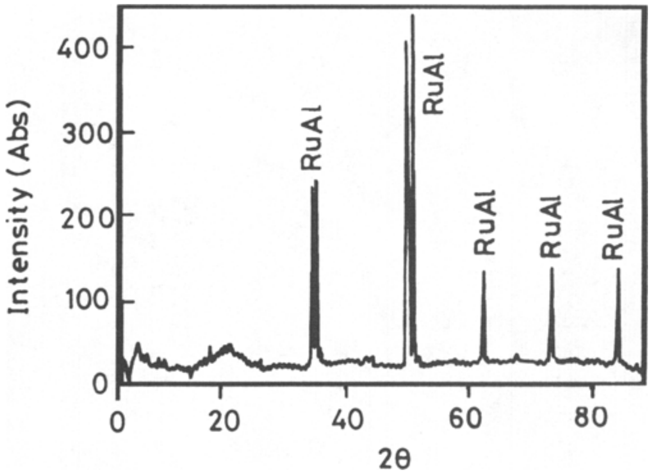

Fig. 7 X-ray diffraction pattern of reactive hot isostatically pressed RuAl

°C, depending on the heating rate. This is well below the softening point of quartz, and in this temperature range the quartz capsule cannot expand to accommodate this large volume increase of the RuAl compact. Hence, capsule cracking takes place. From the HIP cycle, the maximum pressure at the reaction time just before capsule cracking is about 3 MPa. Tantalum has a very high melting point (2996 °C), and a tantalum capsule can collapse around the RuAl compact and effectively transmit the applied pressure only at temperatures over 1000 °C. Thus, none of the capsule materials used was suitable for transmitting the desired pressure at the reaction time where liquid is available in the system. This can explain the incomplete densification after reactive HIP of RuAl. The XRD pattern of this material (Fig. 7) indicates that the RuAl intermetallic phase was formed with no residual ruthenium as it was seen after heating the loose powder through the reaction temperature (Fig. 2b).

Reactive HIP of Nb_3Al elemental powder compact was only tried in quartz capsules. Typical micrographs are given in Fig. 8. Results similar to those found for the RuAl compact were obtained: the densification process was greatly enhanced but full densification could not be obtained. Also, in this case the quartz capsule was cracked after the HIP cycle. The XRD pattern of reactive HIP Nb-Al compact given in Fig. 9 shows that the structure consisted primarily of Nb_2Al and Nb_3Al, with the majority of peaks belonging to Nb_3Al phase. This structure is more homogeneous than that obtained after heating the loose powder (Fig. 2d).

Thus, the reactive HIP structure (Fig. 6 and 8) is more dense compared to the pressureless reactive sintered material (Fig. 4), and the phase composition is more homogeneous compared to that obtained after heating the loose mixed powders. It seems likely that some of the process parameters that hindered densification during pressureless reactive sintering are improved during the HIP process. For RuAl, these parameters may include diffusion rates as well as liquid phase distribution. For Nb_3Al, improvement in the liquid phase distribution could be the main reason for improved densification. These parameters, together with the cold isostatic pressure used to form the green compacts, are the most probable reasons for the more homogeneous reaction products compared to those obtained after heating the loose mixed powders to slightly above the reaction temperature.

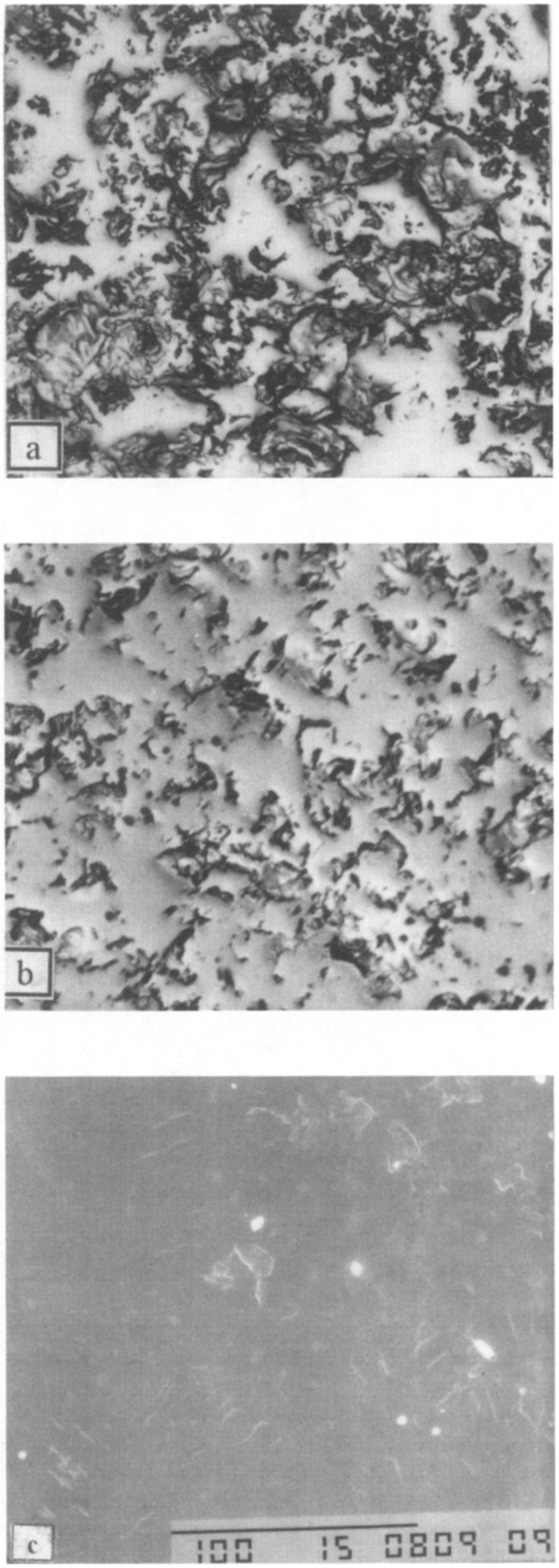

Fig. 8 Microstructure of reactive hot isostatically pressed elemental powder compacts of Nb_3Al. (a) 1750 °C, 100 MPa. (b) 1750 °C, 150 MPa. (c) Scanning electron micrograph of (b). ×200

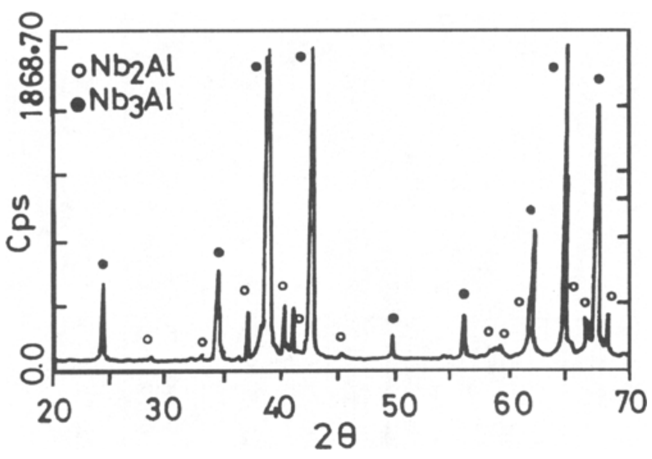

Fig. 9 X-ray diffraction pattern of reactive hot isostatically pressed Nb_3Al

4. Suggestions for Further Work

The effect of other processing parameters should be investigated, such as the particle size range and the particle size ratio of reacting species, as well as the effect of alloy composition on the densification behavior. Also, other capsule materials that enable application of pressure at the reaction time during reactive HIP of these intermetallics must be tried.

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