

Reactive Thermomechanical Processing of Intermetallic Materials

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Intermetallic materials have long attracted the serious attention of scientific and industrial organizations. This is primarily due to their attractive properties, which include high-temperature oxidation and corrosion resistance, low density, and high-temperature strength. Major drawbacks that have so far restricted the application of such materials include the high energy used in their synthesis and production in the final component shape. The thermomechanical processing of intermetallic materials often requires the heating of the work piece to temperatures in excess of 1000 °C. This paper presents results from recent research into new reactive thermomechanical approaches that can produce intermetallics at operating temperatures several hundred degrees lower than those currently used. The main findings suggest that these processes may provide benefits in terms of low energy, consolidation, microstructure refinement, and homogenization.

Keywords aluminide intermetallic compounds, intermetallic materials, thermomechanical processing

1. Introduction

The properties of single-phase, dual-phase, and composite intermetallic materials have long attracted the attention of scientific and industrial organizations. The low density, high-temperature stability, and mechanical properties of aluminide intermetallic compounds in the Ni-Al, Ti-Al, and Fe-Al alloy systems are of considerable interest to a number of industrial sectors including the aerospace, automotive, and power-generation industries. Nickel aluminides are looked upon as materials that can replace existing nickel-based superalloys in demanding applications. The iron aluminides are also important as functional materials due to their good magnetic properties.^[1] Major drawbacks that have so far restricted the use of these materials are the high-energy usage in processing and difficulty in manufacturing to the final component shape.

These materials have been formed in the past by reaction synthesis. This process makes effective use of chemical energy generated during a reaction to process materials with minimal external energy inputs and in very short processing times. Well over 500 different materials have been formed using this approach, including ceramics, intermetallics, and their composites, for a vast number of diverse applications. In principle it can be claimed that this process has existed for over 100 years, however, only in the past 2-3 decades it has been under intense and detailed scientific investigation for the processing of intermetallic materials. The energy savings and economic benefits associated with this process are widely acknowledged. A great deal of work has been published on the subject with reviews by

Subrahmanyam and Vijayakumar,^[2] Yi and Moore,^[3] Murnir,^[4,5] Dunand,^[6] Moore and Feng,^[7,8] Bowen and Derby,^[9] and recently by Morsi,^[10] covering experimental and modeling aspects of reaction synthesis.

A general problem regarding reaction synthesis as a processing approach has been the formation of porosity in the final microstructure. It is, however, well known that during the reaction, the product is raised to significantly high temperatures close to, and sometimes exceeding, its melting point. Heat is then lost to the surroundings, and hence a high-temperature transient window of short duration exists in which these normally brittle materials can be very malleable and most suitable for processing. Therefore, thermomechanical processing during this period should shape and simultaneously consolidate the product. It can also provide an opportunity for microstructure refinement through dynamic recrystallization (largely unutilized in reaction synthesis). The approach also avoids the high-energy and problematic alternative of the multi-step processes of casting, hot deformation, and subsequent cold working and finishing processes. This paper presents findings from the application of thermomechanical processing during these high temperatures achieved in reaction synthesis to form FeAl and Ni₃Al. The potential of these new processes is clearly outlined.

1.1 Aluminide Intermetallics: Properties and Applications

The aluminum-containing intermetallics (i.e., aluminides) have been some of the most studied intermetallics due to their chemical and mechanical stability in addition to their potential weight savings. Nickel aluminide (Ni₃Al) possesses very good high-temperature corrosion and oxidation properties. Strength and fatigue properties can equal and exceed those for commercial superalloys, with potential weight savings. The overall properties exceed those of constructional and casting steels.^[11] A main disadvantage was the room temperature (RT) brittleness of these materials. However, it was found that by adding as little as 0.1 wt.% boron, the nickel-rich Ni₃Al can possess RT elongations of over 50% due to grain boundary strength-

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Table 1 Potential Applications of Aluminide Intermetallics

Iron Aluminides	Nickel Aluminides	Titanium Aluminides
Heating elements	Pistons and valves for automotive diesel engines	Piston heads for diesel engine
Exhaust manifolds	Rollers for steel slab heating	Turbocharger rotors
Dies for superplastic forming of titanium-based alloys	Furnace fixings in carbonizing atmospheres	Compressor cases
Regenerator discs in automotive gas turbine engines	High temperature dies and molds	Turbine blades
Components needing high temperature sulfidation and oxidation resistance	Turbine blades, turbine vanes	Turbine baffles

ble 1.^[18-20] The production of a large number of aluminide artifacts necessitates the use of thermomechanical treatment.

1.2 Reaction Synthesis of Aluminides: A Brief Summary of the Current State of Knowledge

Iron and nickel aluminides, together with a large number of other intermetallics, have been formed in the past using reaction synthesis.^[21,34] The process involves mixing elemental powders of, e.g., iron (Fe) and aluminum (Al) in the correct composition to form the desired intermetallic compound, e.g., FeAl in accordance with the phase diagram. The mixed elemental powders are pressed to form a green compact and heated to a temperature above the melting point of aluminum (typically 700 °C) at which an exothermic reaction proceeds, consuming the elemental powders and forming the intermetallic. The heat of reaction heats the compact to significantly high temperatures and is then lost by heat transfer to the surroundings. Despite its great energy-saving advantage, this method mainly results in porous intermetallic microstructures (and in some cases inhomogeneous) that may require further high temperature consolidation processes (often in excess of about 1000 °C^[22,10]).

A number of pressure-assisted reactive processes have emerged to combat the problem of porosity,^[10] including reaction hot pressing (RHP), reaction hot isostatic pressing (RHIP),^[23] high-pressure reaction synthesis (HPRS),^[24] and reaction infiltration (RI).^[25,26] The application of the more traditional thermomechanical processes such as extrusion and forging during the high temperatures achieved in reaction synthesis have surprisingly been so far ignored. Their application has been limited to use as prereaction consolidation processes of elemental powders such as the extrusion of elemental powders followed by heat treatment and diffusion to produce the intermetallic (often resulting in unwanted mixture of phases and Kirkendall porosities due to unbalanced diffusivities of the elemental species) or as a post reaction synthesis consolidation process by heating the already reacted and cooled compact again to an elevated temperature and applying deformation (clearly an energy intensive and extra processing step). Also recently, hot extrusion reaction synthesis (HERS) was used to extrude elemental powder pellets at temperatures as low as 300 °C and allow the heat of deformation during extrusion to raise the temperature of the extruding material to the reaction temperature with the view of achieving simultaneous reaction and consolidation. This method, however, also resulted in porous microstructures because the reaction always occurred after the material had already exited the extrusion die, thus bypassing the consolidation stage of extrusion due to unfavorable reaction kinetics.^[27,28]

Figure 2 shows a real-time temperature-time profile for an

FeAl compact that we formed by reaction synthesis. As previously mentioned, since the intermetallic product can be heated to very high temperatures during the reaction, there exists a transient high-temperature processing window of short duration where the material can be soft and most suitable for shaping/processing. This high-temperature processing window has not so far been effectively used in the thermomechanical processing of intermetallics.^[10] Figure 2 shows that the processing window is on the scale of seconds for our small specimens; however, for larger specimens it can be on the scale of minutes due to a lower surface area/volume ratio and therefore reduced heat losses.

2. Experimental Procedure

Elemental powders of iron [Atlantic Equipment Engineers (AEE), Bergenfield, NJ], aluminum [Var Lac-oid Chemical Co, Inc. (V.L.O.), Bergenfield, NJ], and nickel (INCO, Inco123, Saddle Brook, NJ) were turbula mixed (GIENMILLS INC., Clifton, NJ) for 30 min in the composition of FeAl (40 at.% Al) and Ni₃Al (25 at.% Al). The mixed powders were then compacted into cylinders with an aspect ratio of 1:1, with diameters of 11.3 and 14mm for the FeAl and Ni₃Al compositions, respectively. All green densities used in reactive thermomechanical processing were 76-79% of theoretical green density (TGD). The compacts were then vacuum degassed at 300 °C for 12 h to remove any low boiling point impurities on the powder surface that may evolve during the reaction and cause unwanted porosity. The degassed pellets were then reaction processed. Table 2 summarizes the relevant process/material combinations used in our experiments. For the reaction forging experiments, each pellet was placed between two quartz glass plates connected to two vertical rams. A K-type (Chromel-Alumel) thermocouple was imbedded into the specimen at half height, and the whole arrangement was surrounded by a vertical tube furnace and heated (at a rate of about 10 °C/min) to the ignition temperature under an argon atmosphere. A computer-controlled setup with rapid-response sensors was used to allow the control of the onset of deformation at a specified temperature along the temperature profile (Fig. 1) as registered through the thermocouple. Graphite was used as the lubricant. In addition to the reaction-forged compacts, specimens were reaction synthesized using the same arrangement/forging setup without actually applying any deformation (i.e., 0% strain).

These experiments were carried out to compare reaction-unforged or reaction-synthesized microstructures with the reaction-forged ones and to determine the reproducibility of the combustion temperature (maximum temperature achieved during the reaction). The experiments verified that the combustion

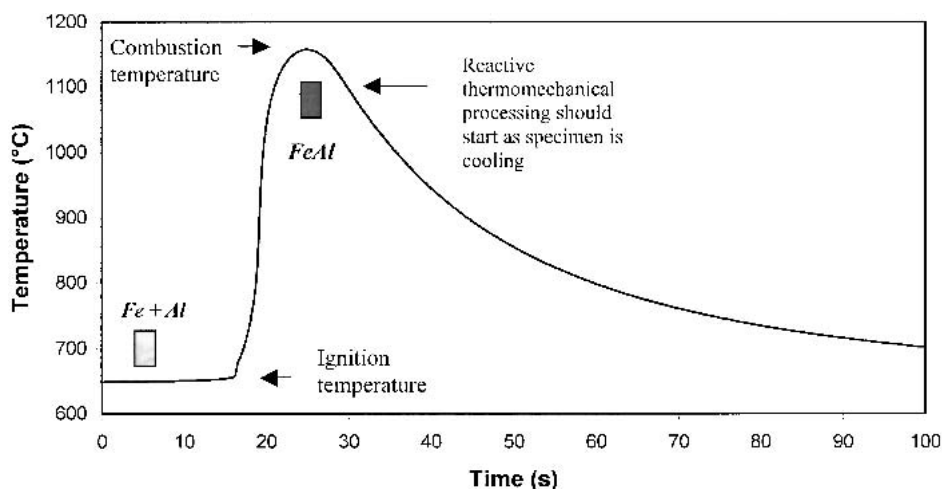


Fig. 2 Temperature-time profile for reaction-synthesized FeAl (actual data), also showing where thermomechanical processing should be applied

Table 2 Summary of the Materials and Their Initial Elemental Particle Sizes to Which Each Type of Reactive Process Was Applied

Process/Material		
Reaction Synthesis	Reaction Forging	Reaction Extrusion
FeAl, <45 μm Fe and < 45 μm Al	FeAl, <45 μm Fe and <45 μm Al	FeAl, <45 μm Fe and 1-6 μm Al Ni ₃ Al, <45 μm Ni and <45 μm Al

temperature was reproducible to within ± 25 °C (more than 10 specimens examined). The reaction extrusion experiments involved heating the green pellet in the extrusion chamber to the ignition temperature. A thermocouple monitored the temperature of the pellet through the hole in the extrusion die. After the reaction, the extrusion was then carried out at the selected temperature along the temperature-time profile and at an extrusion speed of 6-7 mm/s and an extrusion ratio of 10:1. Extrusion onset temperature was 1160 °C for the FeAl composition and 1180 °C for Ni₃Al. Glass was used as the lubricant. All specimens were sectioned centrally along the forging and extrusion axes for microstructural characterization. Microstructural and compositional analysis were conducted using scanning electron microscopy (SEM)/energy dispersive spectroscopy (EDS). Phase analysis was carried out using x-ray diffraction (XRD)(Scintag PADV automated microprocessors control x-ray powder diffractometer, Sunnyvale, CA). To reveal the microstructure, polished specimens were etched using an etchant of composition (25 ml acetic acid + 15 ml HNO₃ + 15ml HCl + 5 ml H₂O for the FeAl and 30 ml H₂O, 10 ml HCl, and 10 ml HNO₃ for the Ni₃Al specimens). Bulk density was measured using the Archimedes principle. Grain size measurements were conducted using the linear intercept method.

3. Results and Discussion

3.1 Reactive Thermomechanical Processing and Effect of Green Density

The formation of FeAl and Ni₃Al from elemental powders occurs via first the formation of other intermediate transitional

phases. However, the full conversion to these aluminides would have already been complete during the cooling stage as the specimen is cooling from the combustion temperature.^[29] This was recently experimentally confirmed using time-resolved x-ray diffraction (TRXRD) applied to phase detection during combustion synthesis of FeAl and mechanically activated self propagating high temperature synthesis (SHS) of FeAl.^[30] Therefore, it is a logical conclusion that the forging or extrusion should be performed as the specimen is cooling from the combustion temperature and not before reaching it (where a number of intermediate phases may still be present). This, of course, assumes prior optimization of a number of processing parameters so that the elemental powder would have transformed to almost 100% intermetallic prior to the forging/extrusion stroke. There are examples where this may not be the case; for instance, if the iron (the higher melting point element) particle size is too large an incomplete reaction will result, leaving excess iron and a mixture of phases in the final product. This can also occur if the green density is not properly optimized. To exemplify this, our initial experiments focused on using fine iron powders and we pressed the Fe + Al powder mixture to 67% and 77% TGD. Each pellet was then reaction synthesized using the same arrangement used for reaction forging but without applying any load during the reaction.

The reaction for Fe + Al compacts was found to initiate at a temperature of 653 °C (± 0.5 °C), i.e., close to the eutectic temperature of 652 °C, and the enthalpy of formation heated the compact to the peak combustion temperature. Similar ignition temperatures have been recently reported for the reaction synthesis of iron aluminides.^[34] It was also observed that the heating rate increased significantly when the temperature

reached exactly 660 °C (the melting point of aluminum). The combustion temperature can give useful indication as to whether the reaction went to completion, e.g., $\text{Fe} + \text{Al} \rightarrow \text{FeAl}$ (assuming the compact does not undergo any other exothermic reactions during reaction synthesis such as oxidation). Usually the higher the combustion temperature, the more complete a reaction. The average combustion temperature for our FeAl specimens was 1174 ± 25 °C. Equation 1 can be used with the relevant thermodynamic data^[31-33] to estimate the maximum possible adiabatic temperature (T_{ad}). For FeAl (40 at.% Al) the adiabatic temperature was found to be 1209 °C.

$$-\Delta H_{f,298}(\text{FeAl}) + \int_{298}^{T_i} C_p(\text{Fe} + \text{Al})dT = \int_{298}^{T_{\text{ad}}} C_p(\text{FeAl})dT, \quad (\text{Eq 1})$$

where

$\Delta H_{f,298}(\text{FeAl})$ = enthalpy of formation of FeAl at 298 K

$C_p(\text{Fe} + \text{Al})$ = specific heat capacity of powder compact

$C_p(\text{FeAl})$ = specific heat capacity of FeAl (40 at.% Al)

T_i = ignition temperature

It is not uncommon for the combustion temperatures to be lower than the thermodynamically predicted adiabatic temperature due to the inherent “adiabatic” assumption that is seldom realized experimentally. Our lower combustion temperatures may be attributed to heat losses at the tooling interfaces in addition to flowing argon. The temperature is also in good agreement with the reported combustion temperature of 1220 °C for Fe-50 at.%Al,^[34] which has a slightly greater enthalpy of formation.^[17] The 67% TGD specimen, however, exhibited an extremely low combustion temperature (797 °C). It is well known that the reaction synthesis process occurs by first the formation of a transient liquid (eutectic/aluminum), which then spreads by capillary action throughout the compact encapsulating the higher melting point particles (i.e., iron). As the reaction front locally progresses through diffusion, iron aluminide is precipitated in its wake. The spreading of the aluminum phase through efficient capillary action is essential in ensuring proper Al/Fe interaction leading to a complete reaction and consolidation through capillary forces. At high porosity levels (as observed for the 67% TGD specimen), the number of contact points between elemental powders is low, and in addition, the capillary forces are also low due to the excessive porosity. This appears to have resulted in an incomplete reaction. Similar findings have previously been reported for the reaction synthesis of iron aluminides and nickel aluminides with low initial green densities.^[34,35] SEM micrographs in the backscattered mode revealed a highly inhomogeneous microstructure for the 67% TGD specimens. The 77% TGD specimen microstructures were, however, predominantly homogeneous FeAl except for very few regions that were slightly iron-rich FeAl. Figure 4 shows micrographs for the green compact microstructures and both material microstructures after reaction synthesis.

XRD was also carried out on both specimens. It was determined that the 67% TGD specimen contained a mixture of phases including FeAl, Fe_3Al , FeAl_2 , Fe_2Al_5 , and unreacted iron, in addition to a small number of unidentified peaks, while the 77% TGD specimen appeared single phase FeAl (because iron diffraction peaks appear at positions close to those of the FeAl, the findings were also confirmed using the compositional analysis facility in the SEM). To achieve enough deformability necessary for crack-free forging, temperatures in the range of 950-1150 °C are usually required.^[36] We therefore did not consider forging temperatures below this range in our experiments. Since the combustion temperature of the 67% TGD specimen was approximately 800 °C, forging was not carried out on this specimen because its combustion temperature fell below the optimum temperature range for hot working in addition to the fact that a number of phases (due to an incomplete reaction), would have been present immediately prior to the forging stroke which may have favored cracking.

3.2 Microstructural Homogenization

The severe deformation associated with extrusion should be expected to have a homogenization effect on an extruding in-

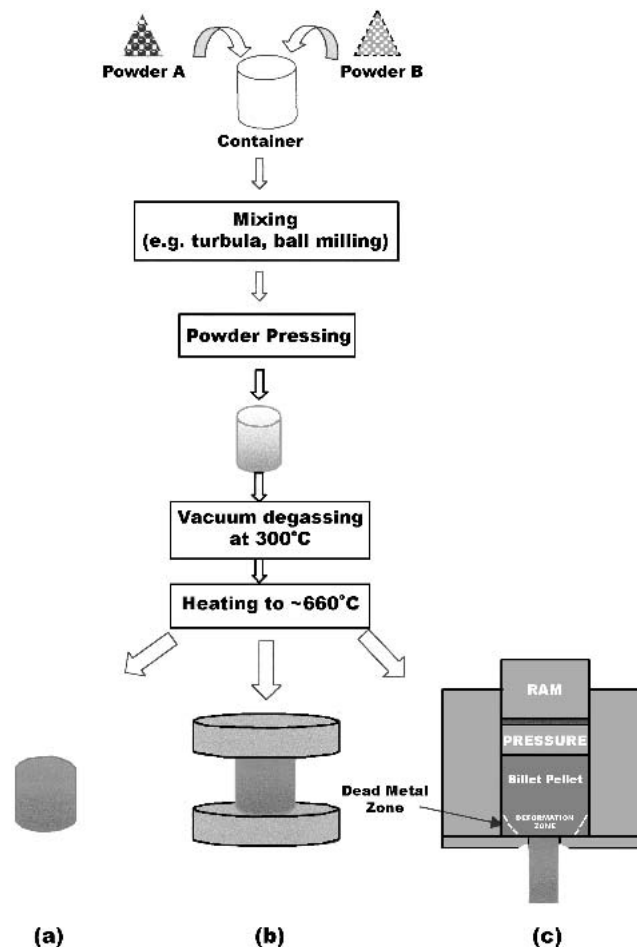


Fig. 3 Processing steps in (a) reaction synthesis, (b) reaction forging, (c) reaction extrusion

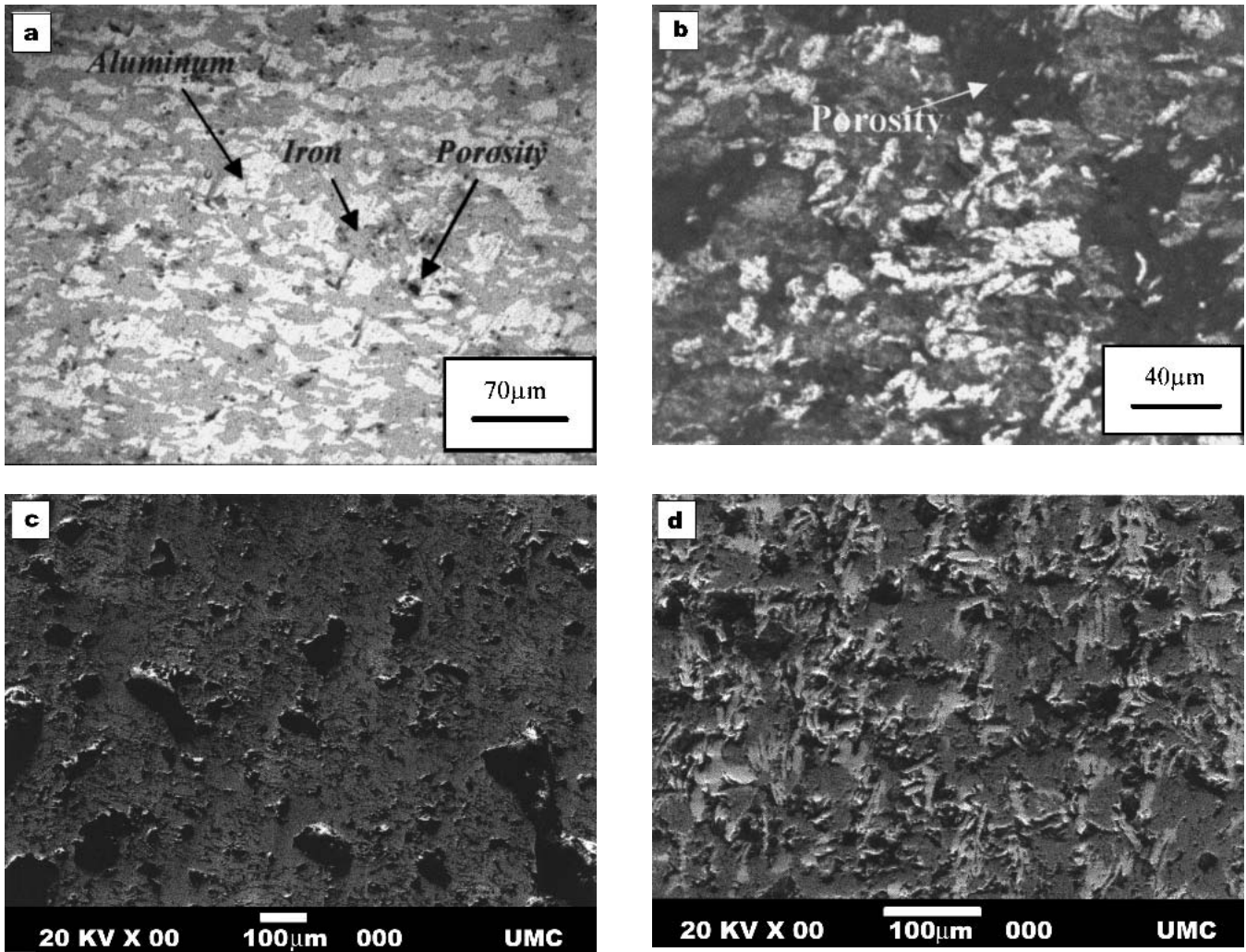


Fig. 4 Light micrograph from the cross section of Fe-Al green compact with (a) 77% TGD and (b) 67% TGD showing the microstructure prior to the reaction, and electron micrographs in the back-scattered mode showing (c) predominantly homogeneous microstructures in the 77% TGD specimens and (d) inhomogeneities in the 67% TGD specimens after the reaction

homogeneous material. To demonstrate this, we applied the extrusion pressure to a reacting pellet having an inhomogeneous microstructure. As the reacted specimen is extruded through the die the material undergoes a high level of deformation and diffusion distances decrease within the extruding material. This facilitates the homogenization of inhomogeneous regions within the microstructure. Figure 5 shows light micrographs taken from different regions within the extrudate. Some regions are taken from inside the discard (i.e., the part of the pellet left behind) having not gone through the severe deformation. The microstructure appears to homogenize as the material is extruded through the die.

For a more detailed examination of the material composition, Fig. 6 shows the reaction-extruded specimen with the discard, and a backscattered image of the microstructure within the extruded material. EDS analysis on the discard revealed a mixture of phases ranging in composition from 12-48 at.% Al (balance Fe), showing the degree of inhomogeneity prior to extrusion. Upon extrusion, EDS analysis on the extruded ma-

terial showed that this compositional range had in fact decreased to be 32-42 at.% Al (balance Fe), clearly showing a homogenization effect (Fig. 5).

3.3 Consolidation

The reaction-forged FeAl specimen appeared sound and crack-free with good surface finish. Also, it was clear that forging was effective in consolidation; pore closure was clearly apparent upon forging. Figure 7^[37] shows the backscattered micrographs of the 77% TGD specimens (from both reaction synthesized and reaction forged specimens). EDS analysis on the reaction-forged specimen confirmed that the bulk predominant composition was FeAl [40.5 Al (± 2) at.%]. The percent porosity of the reaction-synthesized material as measured by the Archimedes method was 15.2%. Upon reaction forging, this was reduced to 5.5%. The experimental setup used was an open-die forging setup; it should be expected that under closed-

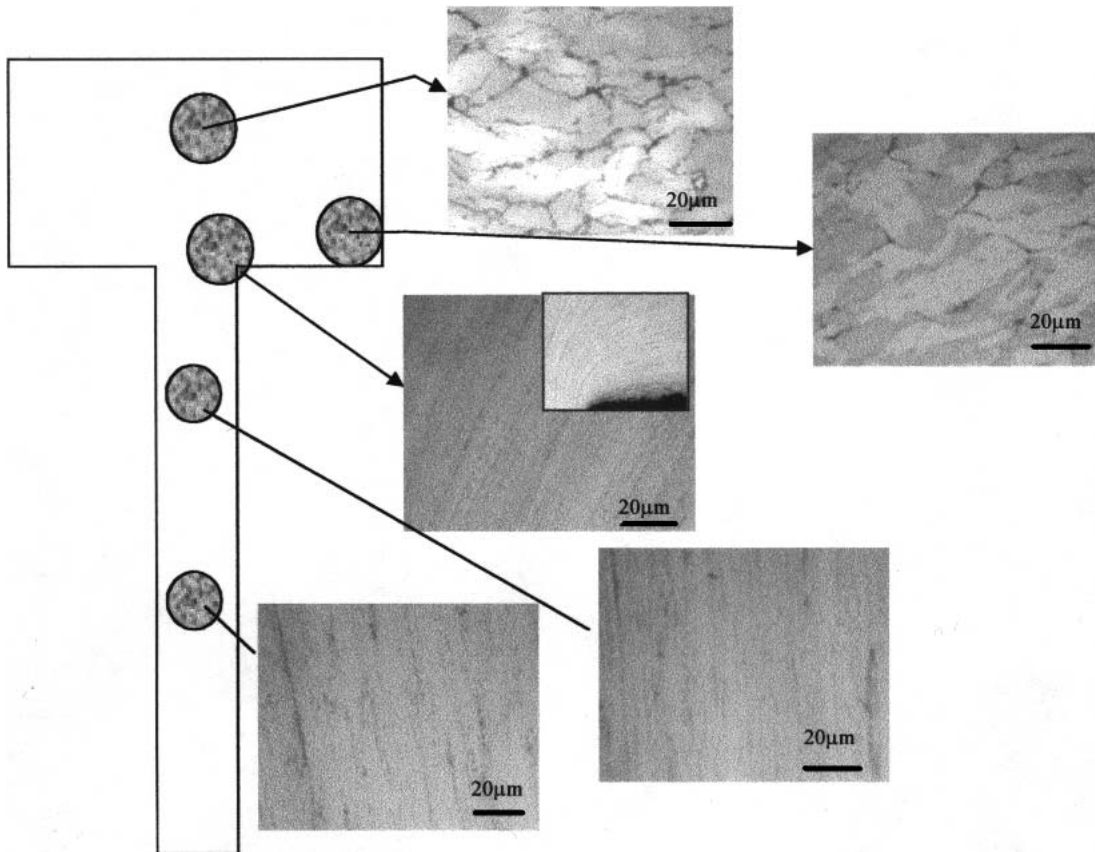


Fig. 5 Light micrographs showing microstructural homogenization due to extrusion

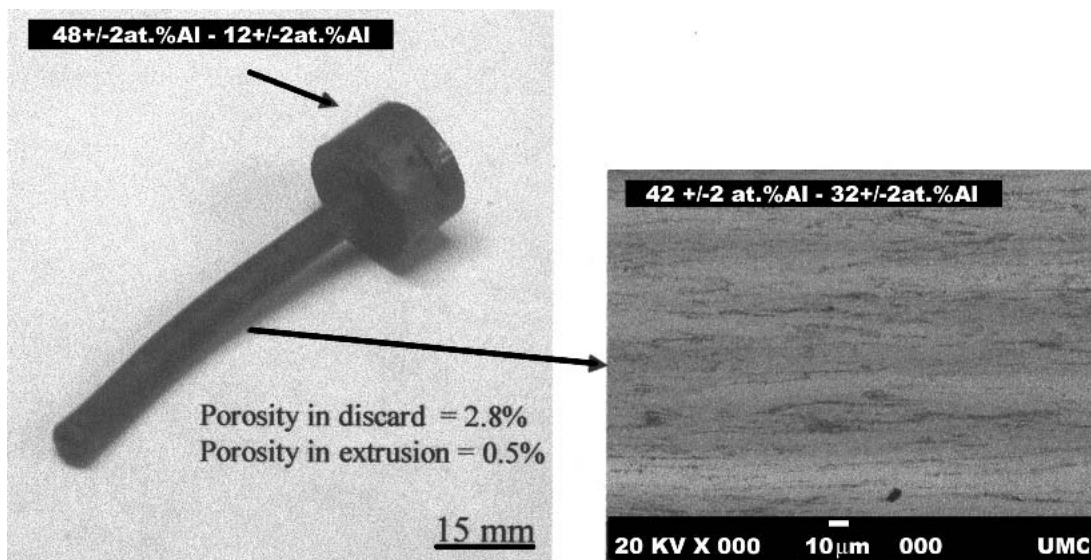


Fig. 6 FeAl specimen reaction extruded at 1160 °C and backscattered SEM micrograph of a microstructure within the extruded material

die forging configurations where higher pressures can be applied, or in extrusion, that pore-free microstructures will result. In fact, this was the case when FeAl and Ni₃Al were reaction extruded; almost pore-free microstructures resulted with an extrusion ratio of 10:1 (Fig. 5 and 6).

3.4 Recrystallization and Microstructure Refinement

After etching the polished reaction-forged specimens, clear indication of subgrain structure was evident (Fig. 8).^[37] Similar findings were recently reported for FeAl alloy that had under-

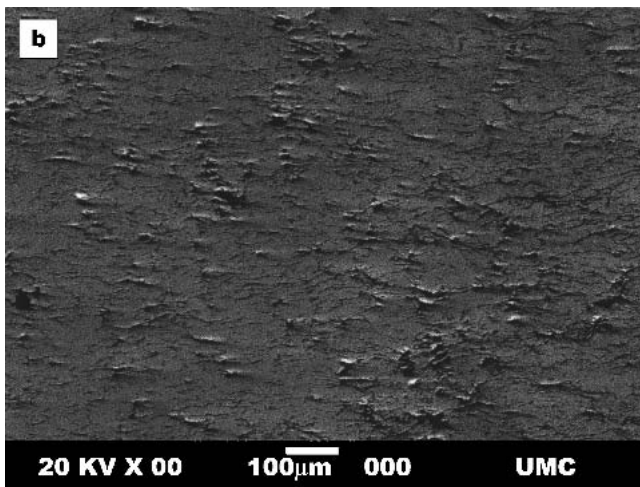
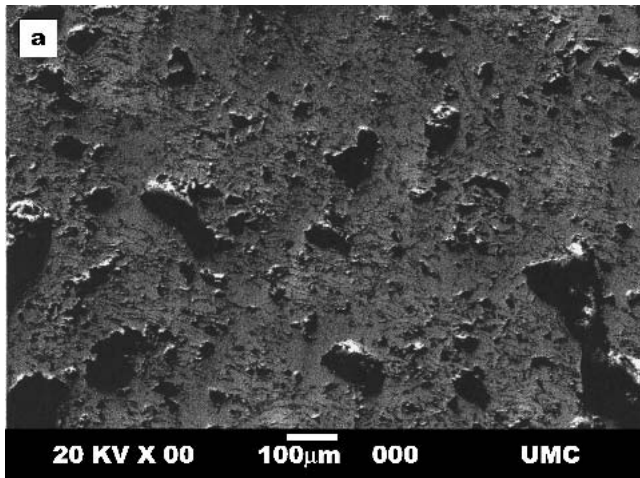


Fig. 7 Backscattered SEM micrographs of 77% TGD (a) reaction synthesized and (b) reaction forged specimens

gone conventional forging to 45% height reduction, at 1000 °C and 1 s⁻¹ strain rate, which revealed subgrains 2-5 μm in size.^[38] Our reaction synthesized FeAl specimen had an average grain size of 13.2 μm compared with an average size of 6.5 μm for the reaction-forged material.

Figure 9 shows a reaction synthesized (reacted inside the extrusion chamber without extruding) and a reaction extruded Ni₃Al clearly showing consolidation and grain refinement through dynamic recrystallization. The average grain size was reduced from 28.4 ± 1.7 μm to 4.5 ± 2.2 μm upon reaction extrusion.

Both reaction forging and reaction extrusion had the effect of increasing the Rockwell hardness of the material compared with the reaction-synthesized counterpart. This was mainly due to reduction in porosity, grain refinement, and the presence of subgrain structure. For example, the reaction synthesized Ni₃Al had a Rockwell hardness (A-scale) of 53 ± 2.9 HRA compared with 77 ± 3.4 HRA for the reaction extruded material.

4. Conclusions

One of the important outcomes of our experiments is that iron and nickel aluminides can be successfully shaped during

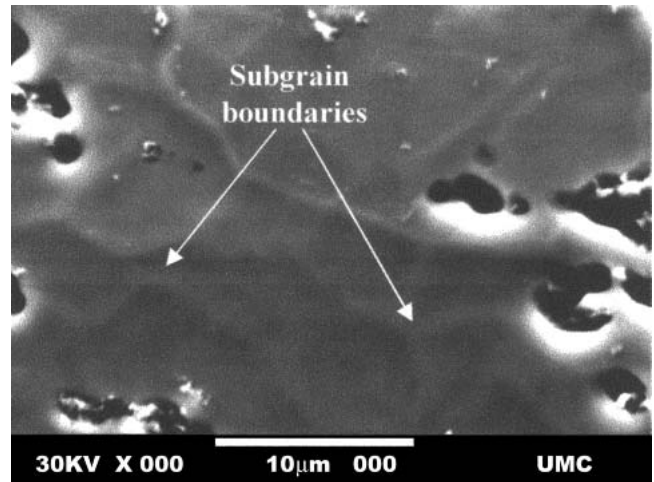


Fig. 8 SEM micrographs of polished and etched microstructures of reaction-forged (secondary electron image) specimen

the high temperatures achieved in reaction synthesis. A number of general conclusions can also be drawn:

- 1) Reaction forging (open-die configuration) can reduce reaction-induced porosities; however reaction extrusion can almost eliminate them.
- 2) Microstructural refinement was found to be possible through dynamic recrystallization. This should open the door to important new research in the field of reaction synthesis.
- 3) Reaction forging and reaction extrusion are one-step processes compared with the two-step process of reaction synthesis followed by additionally heating the reacted compact to the forging/extrusion temperature and then applying forging/extrusion as a second processing step to consolidate and shape the material. This, coupled with the low operating temperatures used, also makes this process more environmentally friendly.
- 4) Both extrusion and forging have been shown to be possible during the high temperatures achieved in the reaction synthesis of iron and nickel aluminides. It is believed that this can further be extended to other systems.
- 5) This processing approach is carried out by heating the compact to temperatures several hundred degrees lower than required for conventional thermomechanical processing of these materials with potential energy savings.

A potential issue that may face reactive thermomechanical processing is establishing a mechanism by which one can uniformly heat very large compacts to the ignition temperature. This would be necessary in producing homogenous microstructures/properties throughout the volume of the large compact. However, the homogenization feature of extrusion may help to reduce/eliminate this if properly optimized. The industrial prospects for reactive thermomechanical processing are very promising. However, there still remains important research that needs to be conducted to fully optimize and model these processes.

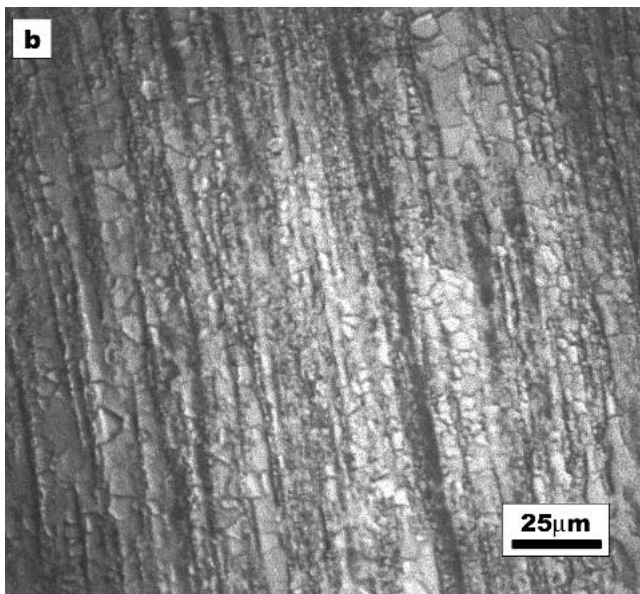
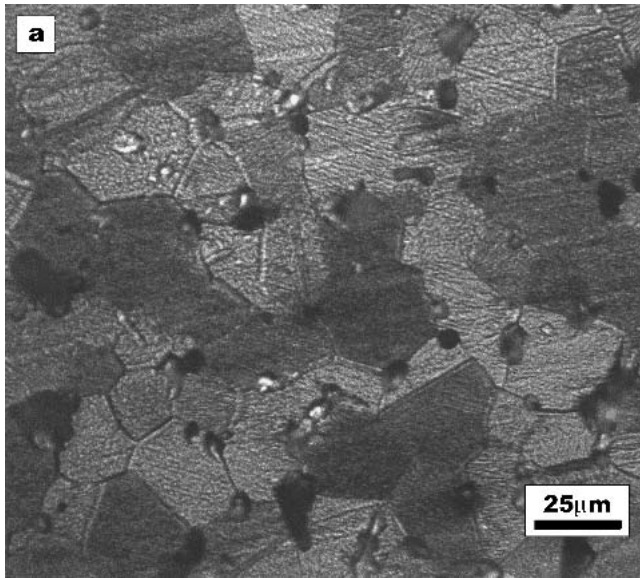


Fig. 9 Light micrographs of as-etched Ni₃Al produced using (a) reaction synthesis (28.4 μm ± 1.7 average grain size), and (b) reaction extrusion (4.5 ± 2.2 μm average grain size)

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