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Processing stainless steel fibre reinforced NiAl matrix composites by reactive hot pressing

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Stainless steel fibres (SSF) were first wound and then plated with electrical nickel film. Alternative layers of these properly spaced Ni-coated stainless steel fibres (SSF–Ni) and aluminum foils were then stacked and diffusion bonded at 500 °C, 100 MPa in vacuum for 10 min to produce an SSF–Ni–Al precursor. Lower-temperature reactive hot pressing (RHP) at 700 °C, a process employed in this work, allowed the nickel and aluminum to react to form an NiAl matrix at a temperature approximately 500 °C lower than that accomplished by hot pressing, hot extrusion or hot isostatic pressing (HIP). RHP at 650 °C was also undertaken in order to investigate the mechanism leading to the formation of a nickel aluminide matrix composite. A dense specimen with complete NiAl reaction was obtained by hot pressing at 900 °C. The variance in microstructure that resulted from the different conditions of heat treatment and hot pressing was analysed by optical microscopy, scanning electron microscopy and X-ray diffraction (XRD).

1. Introduction

Research on new materials with higher melting points, higher strengths, lower densities and better corrosion resistances for elevated temperature applications in the aerospace industry is of tremendous interest to materials scientists at present. Among these new materials, monolithic ordered nickel aluminide intermetallics and their composites have been considered as potential candidates because of their elevated temperature properties: high melting points, high specific stiffnesses, better oxidation and creep resistances [1, 2]. Besides, the Ni and Al elements in the nickel aluminides are easier to purchase compared with Ti, Cr, Co or V for superalloys or other intermetallics. However, despite the apparent advantages of the nickel aluminide intermetallics, they have a significant drawback and that is low room-temperature damage tolerance, i.e. low-temperature brittleness [3]. Until now, this problem has remained the main reason for the unpopularity of nickel aluminides for industrial applications. Several mechanisms have been developed to explain the brittleness [4–6]. One of them is that it is difficult for the dislocations to glide due to the ordered structure of the nickel aluminide. The lack of a sufficient slip system will result in dislocation pile-ups and large stress concentrations. Furthermore, grain boundary segregation of harmful elements, such as O, S, P, Si, etc. [5], and the lack of connectivity of this boundary [6] restrict the movement of dislocations.

There are two main approaches to improving the damage tolerance of nickel aluminide intermetallics: these are intrinsic and extrinsic modifications. The first approach relies on intrinsic modification using microalloying addition of boron, chromium or other elements [7–9] to enhance the grain boundary connection strength, and to increase the density of dislocations gliding adjacent to the grain boundary. This method has not been successful for NiAl. The second approach involves extrinsic additions of ductile metallic particles or fibres to result in a crack-tip shielding phenomenon; i.e. unbroken ductile-ligaments bridging and crack deflection mechanisms [10–12]. From earlier research findings on fracture toughness, we can expect the toughness of the nickel aluminide intermetallics to be enhanced by the latter approach. This research is about the processing of nickel aluminide intermetallic composites, reinforced with uniformly distributed ductile metallic fibres, using the second approach.

Present methods for the fabrication of nickel aluminide intermetallic composites can be divided into two types: liquid and solid metallurgy. In liquid metallurgy, pressure casting or the infiltration of molten nickel aluminide into a preform is the usual method [13–15]. Generally, this method is not useful for metallic reinforcements because of drastic reactions between the molten nickel aluminide and the metallic preform at a temperature of up to 1600 °C. Besides, sustenance of the performance of the metallic preform at such a high temperature is also a problem. In solid metallurgy, fabrication is based on reactive powder metallurgy [16, 17] and selfpropagating high temperature synthesis (SHS) [18, 19] or hot pressing, hot extrusion and HIP [20–22]. High processing temperatures and pressures are necessary conditions for hot pressing, hot extrusion and HIP, so the process requires sophisticated manufacturing equipment and considerable energy. Generally, intermetallic matrix composites are fabricated at a temperature of at least 1200 °C, by either hot pressing the layers of NiAl foils and the Saphikon Al_2O_3 fibres [20] or HIP to produce the intermetallics [21, 22]. However, all these factors tend to increase the cost of production, therefore rendering the application of nickel aluminide intermetallic composites unpopular.

Work on reactive powder metallurgy and reactive hot pressing at a low temperature near the melting point of aluminum is reconsidered here. Our efforts indicate that by combining the spontaneous reaction of electrically coated nickel film on stainless steel fibres and the aluminum foils, and hot pressing them at a temperature about 500 °C lower than previously accomplished by hot pressing, hot extrusion and HIP, this would help overcome the fabrication process problem of nickel aluminide intermetallic composites reinforced with uniformly distributed metallic fibres.

2. Experimental procedure

304 stainless steel fibres with diameters of 150 μm were wound on a framework by an automatic winding machine. The spacings between the fibres were fixed by controlling the rotation speed of the framework and the movements of the stainless steel fibres. The wound fibres were then electrically nickel plated. Watt solution was chosen as the electrical plating solution, which was composed of $NiSO_4$ ($240\,g\,l^{-1}$), $NiCl_x$ ($45\,gl^{-1}$) and H_3BO_3 ($30\,g\,l^{-1}$). The pH value and the temperature of the solution were 4.0 and 50 °C, respectively. The cathode was the wound stainless steel fibres, and this was located between two anodic nickel plates. The current for the electrical plating was controlled at 6.6 A and the electrical process continued for 4 h. After the process, each fibre on the framework was coated with a uniform nickel film to form an SSF–Ni composite fibre with a diameter of 225 μm.

Alternate layers of properly spaced nickel coated stainless steel fibres and aluminum foils, 100 μm in thickness, were stacked and hot pressed at 500 °C, 100 MPa in vacuum for 10 min to obtain an SSF–Ni–Al precursor. This is the well known "diffusion bonding" process [23]. The thickness of the coated nickel film and the amount of required aluminum foils were ascertained by calculating the stoichiometric atomic ratio of NiAl, 1 : 1.

What we wanted, however, was not the SSF–Ni–Al precursor but the SSF–NiAl composite. The following process, reactive hot pressing, was thus employed; which enabled the nickel and aluminum to react to form a nickel aluminide intermetallic matrix. The SSF–Ni–Al precursor was first embedded in the centre of a mould filled with Al_2O_3 powder. Cold compacting with a pressure of 100 MPa was next applied for 10 min to densify the Al_2O_3 powders. Hot pressing with this design has a similar effect to that of HIP and the design reduces aluminum expulsion from the periphery of the specimen. Schematic designs of the fibre winding, electrical nickel plating, diffusion bonding and reactive hot pressing are illustrated in Fig. 1.

Two reactive hot pressing processes under different conditions were tried in order to investigate the mechanism leading to the formation of the NiAl intermetallic matrix. The first process involved hot pressing the SSF–Ni–Al precursors at 650 °C, 150 MPa for 5, 10, 15 and 20 h, respectively. Local aluminum adjacent to the nickel melted, due to the presence of heat released during the exothermic reaction. The reaction was investigated employing different hot pressing times. In the second process, the specimens were heated to 700 °C and maintained at this temperature for 1 h,

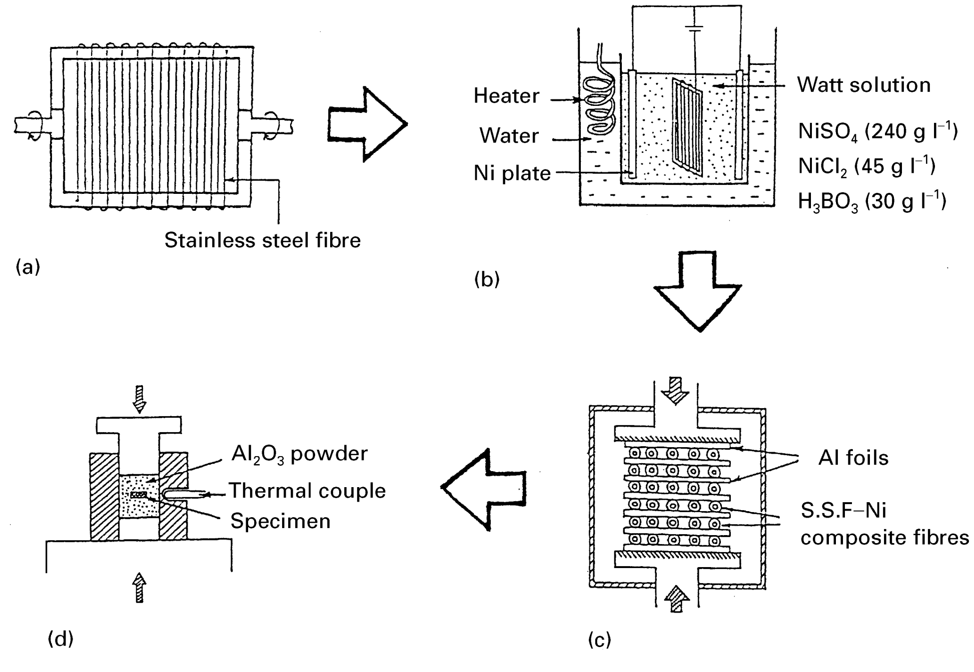

Figure 1 Schematic design of (a) the fibre winding, (b) electrical nickel plating, (c) diffusion bonding, and (d) reactive hot pressing.

during which the nickel adjacent to the aluminum dissolved into liquid aluminum and a slurry semisolid phase began to form. The increase in the viscosity inhibited the expulsion of aluminum during application of pressure, and the pressure was increased in a series of steps to 100 MPa (30 MPa for 1 h, 50 MPa for 2 h and 100 MPa for 4 h). The processing temperature of 700 °C, one that was about 500 °C lower than that which was previously accomplished by hot pressing the layers of NiAl foils and Saphikon Al_2O_3 fibres [20], hot extrusion [21] or by hot isostatic pressing [22], was used to produce the intermetallic composite.

Higher temperature hot pressing at 900 °C, 70 MPa for 2 h was necessary to allow complete NiAl reaction to occur and to derive a denser specimen. To understand the reaction between nickel and aluminum, different heat treatment and hot pressing conditions for the SSF–Ni–Al precursor were tried. The variance of the microstructure exposed to such varied conditions of heat treatment and hot pressing was analysed by optical microscopy, scanning electron microscopy and XRD.

3. Results and discussion

To obtain a densified SSF–NiAl intermetallic matrix composite, four investigative procedures, electrical nickel plating, diffusion bonding, reactive hot pressing and higher-temperature hot pressing, were undertaken in the order listed. The results and mechanisms for each step are discussed below.

3.1. SSF–Ni composite fibres

Fig. 2a shows the surface morphology of the as-received stainless steel fibre. The surface of the fibre is smooth. Fibres of 150 μm diameter were electrically plated with a uniform nickel film to form an SSF–Ni composite fibres (Fig. 2b) with an average diameter of 225 μm. A noticeable uniform film with a slightly rough surface was a common phenomenon and the many small particles that deposited on the surface of the fibre were due to the large current used and fast deposition rate. Inhomogeneous plating occurred only on the fibres around the framework because a non-uniform electrical field resulted from the edge effect. The thickness of most of the nickel film was well controlled. Only uniformly coated fibres of the same diameter in the centre of the framework were chosen for the next procedure.

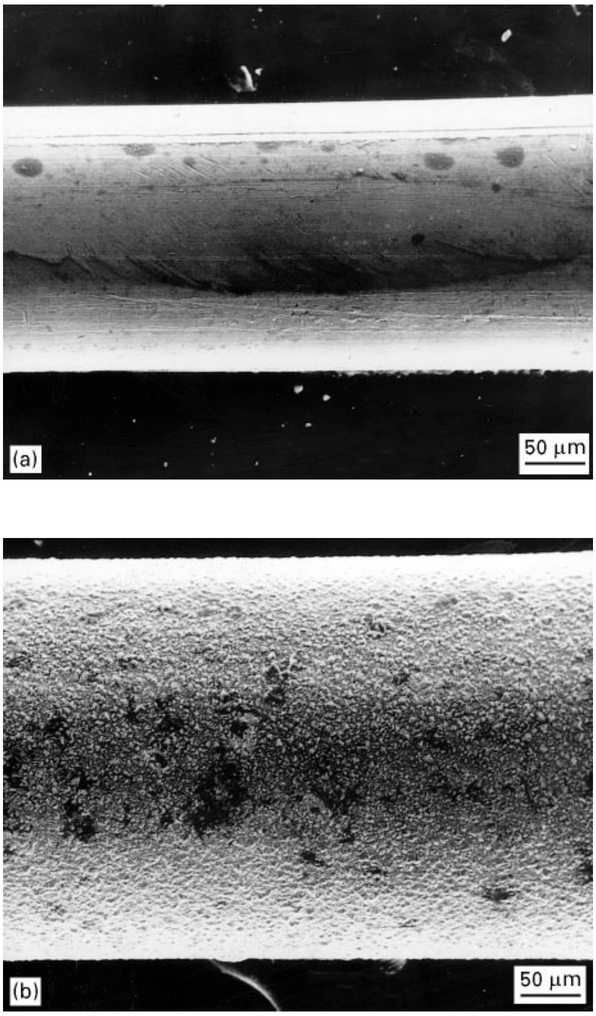

Figure 2 Surface morphologies of (a) as-received stainless steel fibre and (b) electrical Ni-coated stainless steel fibre.

3.2. SSF–Ni–Al precursor

"Diffusion bonding", a well developed process for producing fibres reinforced aluminum matrix composites [23] is also adopted in this experiment. An SSF–Ni–Al precursor with a relatively uniform distribution of stainless steel fibres in the matrix obtained by diffusion bonding is presented in Fig. 3a. Fig. 3b shows the small interfacial reaction between the coated nickel film and the aluminum when processing at the temperature of 500 °C for a short time. The small particles on the rough surfaces of the coated SSF–Ni composite fibres provide an advantage for diffusion bonding. During the deformation of aluminum foils under pressure, the oxided Al_2O_3 films on the surfaces of the aluminum foils were broken easily by shear forces due to the rough surfaces of the coated SSF–Ni composite fibres and because fresh aluminum would then have been in close contact with the nickel film. No interfacial voids then existed and immediate reaction occurred in the following reactive hot pressing procedure.

Kesapradist *et al.* [24] have suggested that the Al_2O_3 fibre reinforced Ni_3Al matrix composite can be produced by vacuum hot pressing of layered nickel-plated aluminum foils and Al_2O_3 fibres. It is mentioned that complete infiltration of the Al_2O_3 fibres by the nickel at a raised temperature of 800 °C and a pressure of 55 MPa remained impeded where the fibres bundled together and that a non-uniform distribution of the fibres in the Ni_3Al matrix also occurred. Our work, however, indicated that the previous uniform nickel coating on fibres, associated with the complete infiltration of aluminum at a low temperature of 500 °C by diffusion bonding, overcame the processing problem of embedding the fibres uniformly into the matrix.

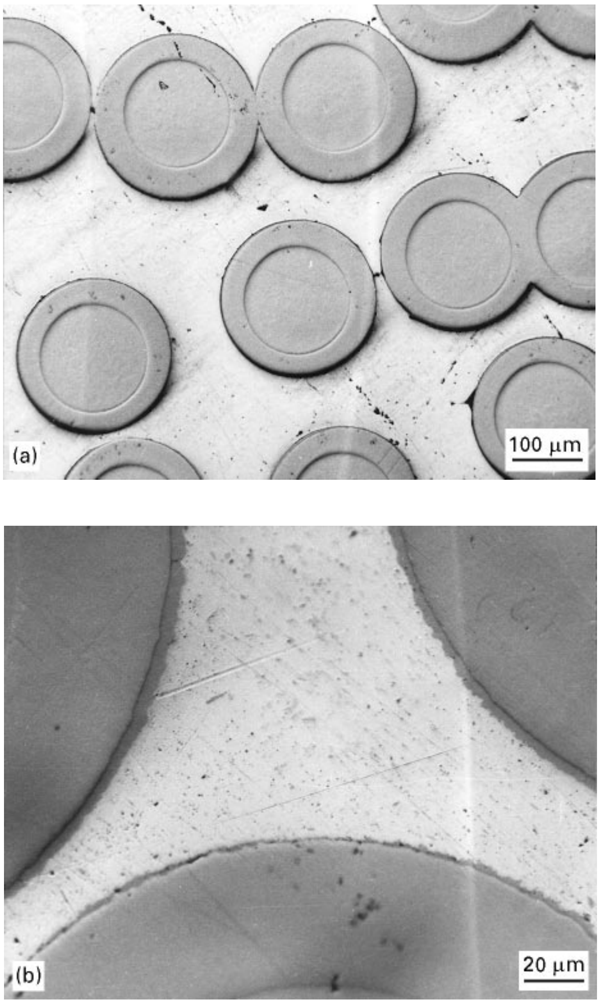

Figure 3 Microstructures of the SSF–Ni–Al precursor obtained by diffusion bonding at 500 °C, 100 MPa for 10 min show (a) lower magnification (b) small interfacial reaction found at higher magnification.

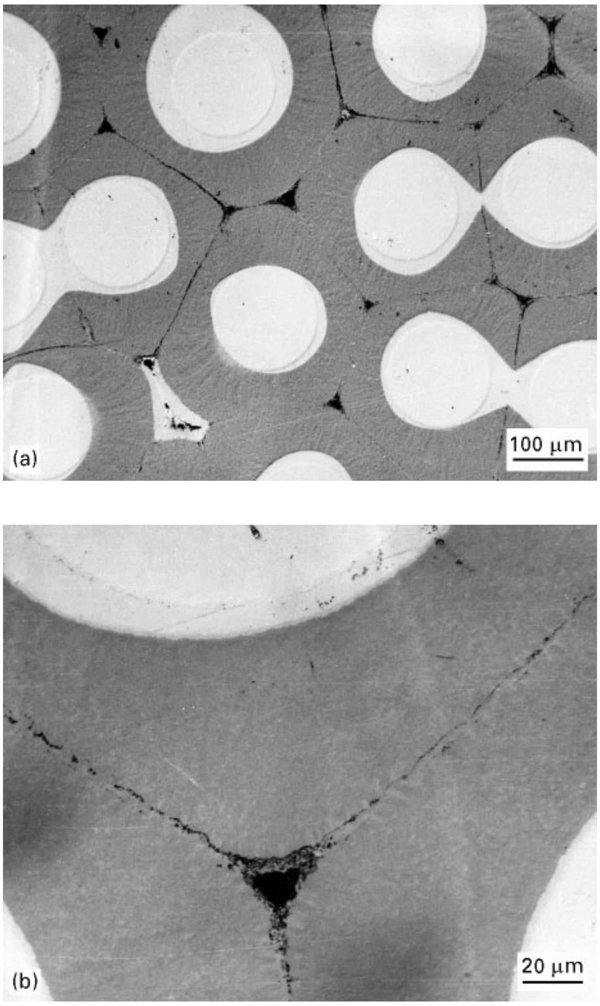

Figure 4 Microstructure of the nickel aluminide matrix composite after RHP at 650 °C, 150 MPa for 20 h showing (a) hexagonal structure and (b) an obvious tubular pore.

3.3. Reactive hot pressing at 650 °C

An intermetallic matrix composite with hexagonal structure was obtained after processing at 650 °C for 20 h (Fig. 4a) The formation of hexagonal reaction zones was due to reaction between the nickel and the aluminum, and to the existence of reaction zone boundaries, tubular pores and several phases in the matrix as shown in Fig. 4b. The reaction zones were uniform and they continued to expand with increasing reaction time until they made contact with each other. From the XRD patterns, it is known that the aluminum was almost consumed and the matrix was composed of several phases, including a high proportion of NiAl, Ni_2Al_3 and some $NiAl_3$, Ni_3Al (Fig. 5).

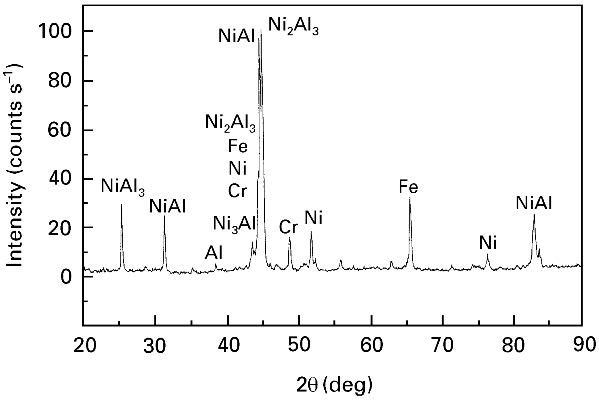

Figure 5 XRD pattern of the nickel aluminide matrix composite after RHP at 650 °C, 150 MPa for 20 h.

Hot pressing below the melting point of aluminum prevented the expulsion of the aluminum from the periphery of the specimen due to the higher viscosity of the solid aluminum. Spontaneous reaction of the aluminum and the nickel into nickel aluminides at the interface began immediately when the temperature was raised to 600 °C. A small amount of transient liquid aluminum, that formed locally at the interface between the nickel and the aluminum, was attributed to the heat released during the exothermic reaction and which then accelerated the reaction. The interdiffusion coefficient of aluminum is larger than that of nickel in the Ni–Al system at this temperature of 650 °C [25]. Thus, the more rapid diffusion of aluminum atoms into nickel than that of nickel atoms into aluminum resulted in the lesser formation of nickel aluminides in the aluminum but much more in the nickel, and lead to expansion of the reaction zones.

The reaction zones acted as diffusion barriers and reduced the reaction rate. As the aluminum was consumed continuously, layers of oxides and contaminants on the surfaces of the aluminum foils moved with the movement of the reaction zone–aluminum interface due to the Kirkendall effect. This behaviour resembles the marker observation of the well known Kirkendall diffusion couple [26] and Darken's analysis [27]. Pores also formed in the centre of the aluminum phase due to the Kirkendall effect, associated with volume shrinkage during the formation of the intermetallic phase. Before the aluminum was consumed completely, these pores were closed immediately by the plastic flow of aluminum under this temperature and pressure as soon as they were formed. Eventually, as expansion of the reaction zones and consumption of the aluminum occurred, layers of oxides and contaminants moved towards and existed with some small voids, resulting in boundaries between reaction zones. The hexagonal reaction zones constructed the matrix and looked like the grain structure. Similar results were reported in [19] in which it was suggested that oxides, contaminants and voids existed in the reaction zone boundaries. These boundaries affected the tensile properties seriously and the cracks propagating along the interfaces further reduced the strength drastically compared with typical NiAl intermetallics [1, 2].

Tubular pores, parallel to the fibres, appeared at reaction zone junctions. As described above, pores formed in the centre of the aluminum phase as the reaction zones expanded and they were closed up immediately by plastic flow of the other aluminum nearby until the reaction zones had expanded to make contact with each other. When the reaction zones had made contact with each other, they formed a stereo skeleton and the aluminum at the reaction zone junctions became isolated. By this time, the new pores forming inside the aluminum phase could not be eliminated by the plastic flow of aluminum or nickel aluminide due to the lack of deformation of the rigid nickel aluminide skeleton under 650 °C, 150 MPa condition. As consumption of aluminum and volume shrinkage continued during reaction, the pores expanded to be observed as tubular pores.

3.4. Reactive hot pressing at 700 °C

Hot pressing at a temperature above the melting point of aluminum accelerated the reaction and the aluminum was consumed in a short time. A nickel aluminide intermetallic matrix composite reinforced with stainless steel fibres was obtained, as shown in Fig. 6a. Reaction zone boundaries and tubular pores, however, did not exist. As shown in Fig. 6b, only some small pores were found in the matrix. The cracks beneath the fibre, seen also in Fig. 6b, were formed when preparing the metallurgraphical specimen. The XRD pattern demonstrated that the phases existing in the intermetallic matrix were mostly NiAl, Ni_2Al_3 and a small amount of $NiAl_3$, Ni_3Al, (Fig. 7).

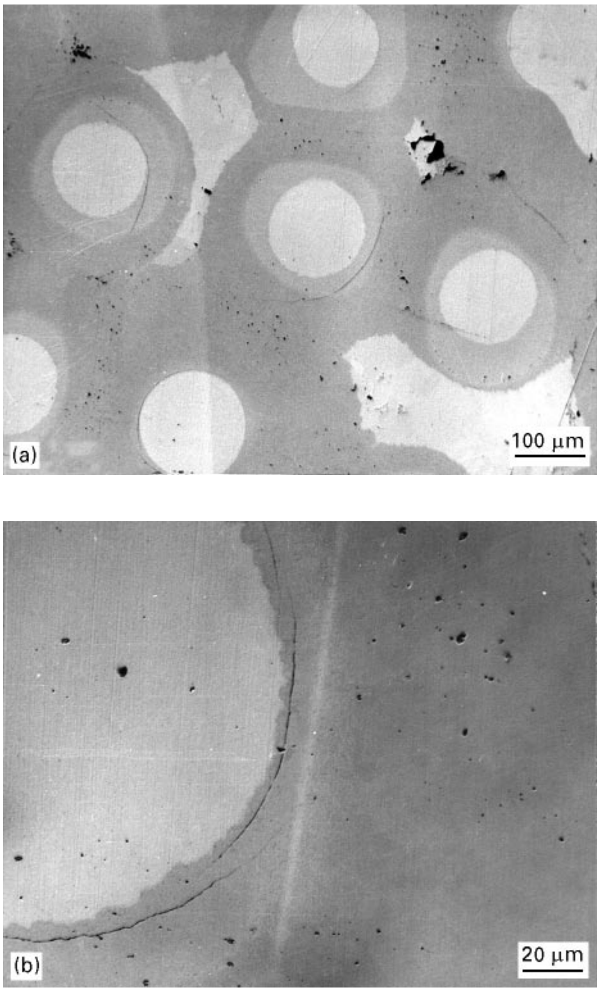

Figure 6 Microstructure of the nickel aluminide matrix composite after RHP at 700 °C with increasing pressure (without pressure for 1 h, 30 MPa for 1 h, 50 MPa for 2 h and 100 MPa for 4 h) at (a) lower magnification and (b) showing some small pores in the matrix.

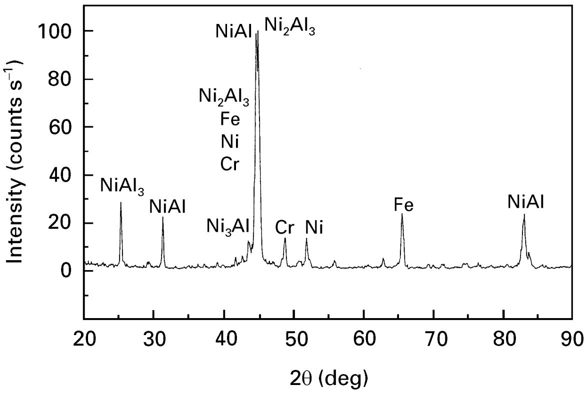

Figure 7 XRD pattern of the nickel aluminide matrix composite after RHP at 700 °C with increasing pressure (without pressure for 1 h, 30 MPa for 1 h, 50 MPa for 2 h and 100 MPa for 4 h).

Liquid aluminum was expelled from the periphery of the specimen when the pressure was applied immediately. Holding the specimen at 700 °C for 1 h allowed nickel, adjacent to aluminum, to dissolve into liquid aluminum rapidly and form a slurry semisolid phase (composed of liquid aluminum and solid nickel aluminides, $NiAl_3$ and Ni_2Al_3). The formation of solid phases in liquid aluminum raised the viscosity and minimized the leakage of aluminum. Pressure, which was applied in a series of steps to 100 MPa, closed up the voids that formed during the reaction.

Reaction zone boundaries, as shown by RHP at 650 °C, did not exist. Whether the transient liquid phase formed or not during RHP at 650 °C, the oxides and the contaminants remained on the surfaces of the aluminum foils. But at 700 °C, all of the aluminum foils melted. Because dissolution of the nickel into liquid aluminum is faster than into solid aluminum, a large amount of $NiAl_3$ and Ni_2Al_3 precipitated in the liquid aluminum while supersaturation occurred. Oxides and contaminants were then dispersed from the original surfaces of the aluminum foils and never concentrated together. Any pore that formed during the reaction was eliminated immediately by applying pressure due to the facile flow of the semisolid phase. After the aluminum had been consumed, Ni_2Al_3 and $NiAl_3$ became the dominant phases in this region. The nickel aluminide matrix formed by precipitation of $NiAl_3$ and Ni_2Al_3 phases, not by expansion of the reaction zones. The two original surfaces did not move towards each other, and did not result in the appearance of reaction zone boundaries as seen by RHP at 650 °C.

Though these pores continued to exist, they became fewer in number and smaller in size. Because the nickel dissolved fast into the liquid aluminum at 700 °C, a large amount of solid $NiAl_3$ and Ni_2Al_3 precipitated uniformly in the liquid aluminum. The pores formed due to the Kirkendall effect and the volume shrinkage was also distributed uniformly in the semisolid phase, and could be eliminated easily. Because the pores did not concentrate at a local site, as shown by RHP at 650 °C, tubular pores were eliminated and the amount of pores was much reduced. The reaction mechanisms, during heat treatment, RHP at 650 and 700 °C, are illustrated in Fig. 8.

3.5. Higher-temperature hot pressing at 900 °C

In order to obtain a specimen with good mechanical properties and a stable structure at elevated temperature, complete NiAl reaction between the several nickel aluminide phases must be achieved and the densification must be much enhanced. Higher-temperature hot pressing at 900 °C, 70 MPa for 2 h (still a lower temperature than previously accomplished by hot pressing and HIP [20–22]), allows complete reaction to occur and a denser specimen to be obtained in a shorter time. In the specimens after RHP at 650 °C and hot pressing at 900 °C, pores at local reaction zone junctions became smaller, but yet remained

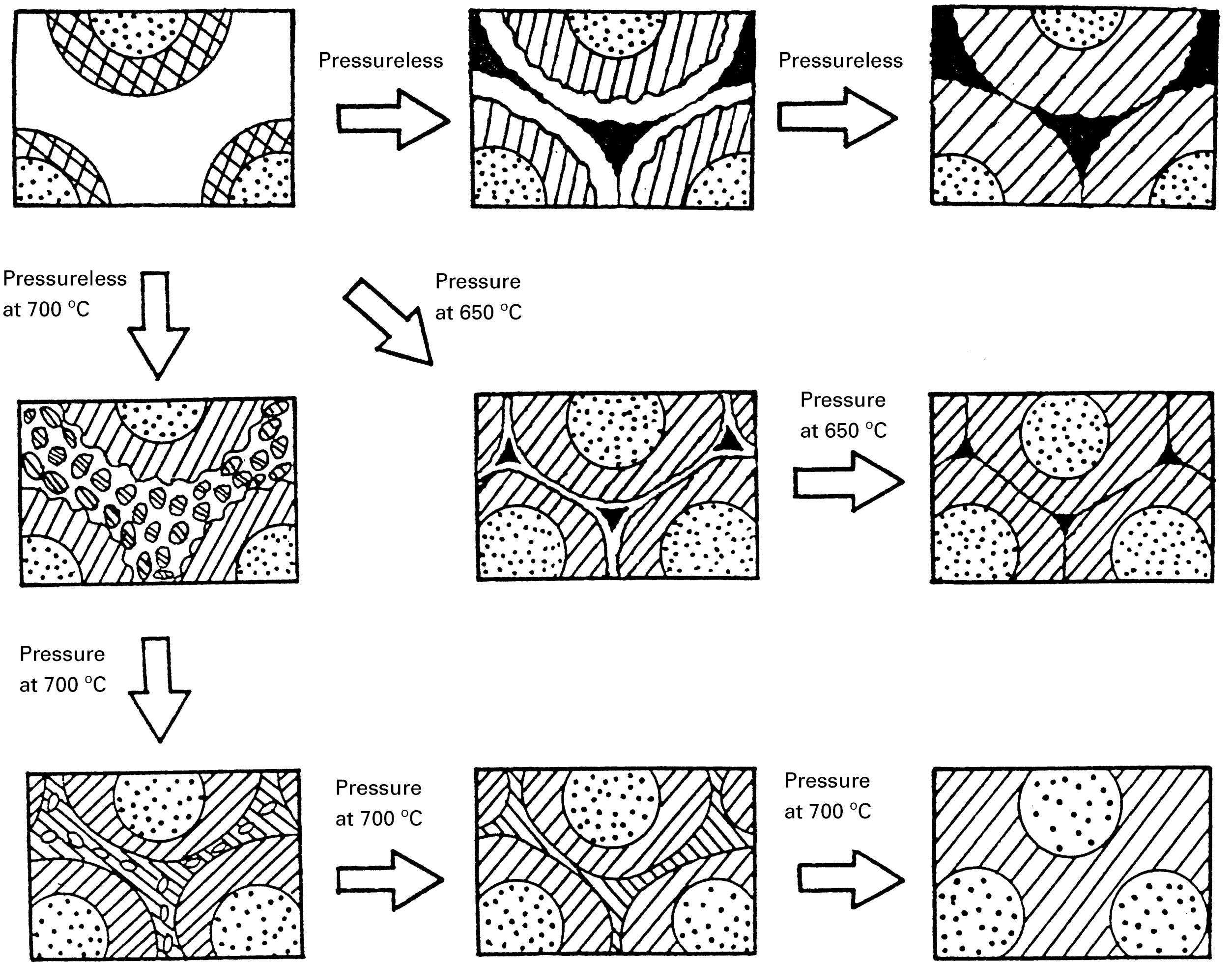

Figure 8 Illustration of the reaction mechanisms during heat treatment, RHP at 650 and at 700 °C: (⁙) stainless steel fibre, (▩) nickel, (□) aluminum, (▨ ▧) nickel aluminide, (■) pore.

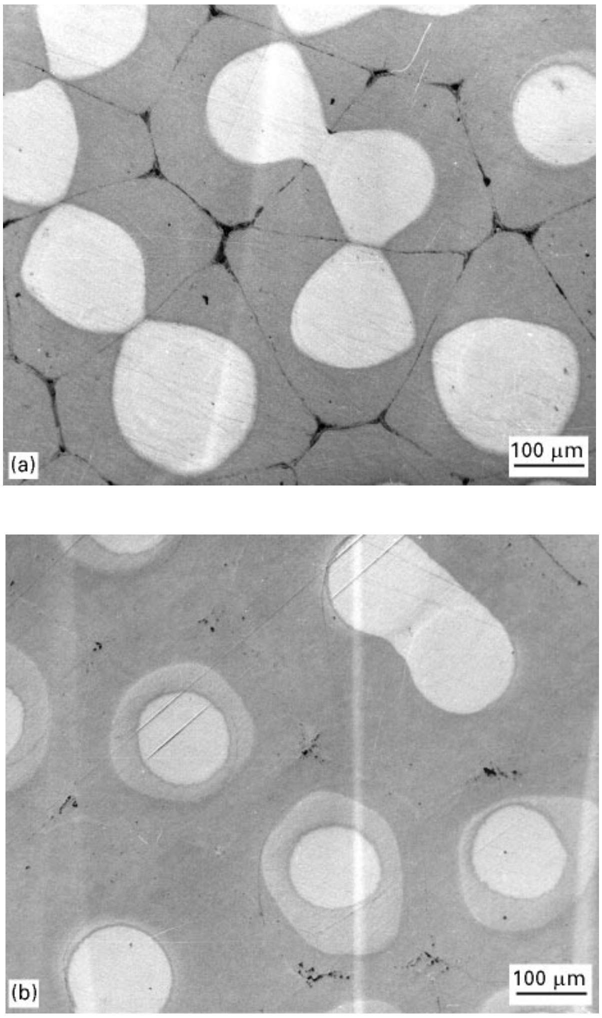

Figure 9 Microstructure of the NiAl matrix composites after RHP at (a) 650 and (b) 700 °C, both followed by higher-temperature hot pressing at 900 °C for 2 h.

inside because of slight plastic deformation of the rigid nickel aluminide skeleton, continuous pore formation and volume shrinkage (Fig. 9a). Reaction zone boundaries were still observed. By contrast, pores were eliminated in the specimens after RHP at 700 °C and hot pressing at 900 °C (Fig. 9b) and the matrix was composed of NiAl phase from the XRD analysis (not shown here). Consequently, a nearly poreless NiAl matrix composite toughened with the stainless steel fibres were obtained successfully. Combining the high stiffness of NiAl and the large ductility of stainless steel fibres, excellent tensile behaviour of the composite associated with a large increase in fracture toughness can be expected.

3.6. Porosity

Four factors related to pore formation during sintering of TiNi shape memory alloys have been mentioned by Zhang *et al.* [28]. These are: (1) primary pores, (2) capillary phenomenon, (3) the Kirkendall effect and (4) volume change during formation of alloys. These four factors are introduced and compared with those of our experimental results.

1. Primary pores were not observed in the SSF–Ni–Al precursor, as shown in Fig. 3, because of the complete infiltration of aluminum. Thus, the pores that appeared in SSF–NiAl specimens were not the remnants of primary pores.

2. During liquid phase sintering, the transient liquid spread along the small spaces between the powders due to the capillary force and pores formed at the sites where the liquid flowed out. In our experiment, however, the situation with regard to powder sintering is different. All the spaces between the SSF–Ni composite fibres were full of aluminum, and there was no small space for the liquid to flow into either during RHP at 650 or 700 °C.

Pores that resulted from the capillary phenomenon did not appear in our experiment. Therefore, these two factors are not prerequisites for pore formation. The dominant factors for pore formation are the following third and fourth factors.

3. Pore formation due to the Kirkendall effect is a common phenomenon in diffusion couples. In RHP at 650 °C, fast diffusion of aluminum into nickel in the Ni–Al system [17, 25] led to pore formation in the centre of the aluminum phase due to the Kirkendall effect. Evidence for this is provided by the pores existing at reaction zone junctions. Though pressure was applied, these pores could not be eliminated if the reaction zones were in contact with each other. By contrast, in RHP at 700 °C, pores also formed in the aluminum phase due to the Kirkendall effect, but they were eliminated immediately by applying pressure on the semisolid phase, no pore were found in this specimen. After the aluminum was consumed, whether during RHP at 650 or 700 °C, faster diffusion of the nickel into Ni_2Al_3 in the Ni–Ni_2Al_3 system [17, 25] resulted in pore formation in the nickel phase, but not in the Ni_2Al_3 phase. This result can be obtained in the 1200 °C heat treated specimen without pressure after RHP at 650 °C (Fig. 10). Pores several micrometers in size formed in the nickel phase around the stainless steel fibres due to the Kirkendall effect. These pores were eliminated gradually under pressure because of plastic deformation of the nickel phase under pressure.

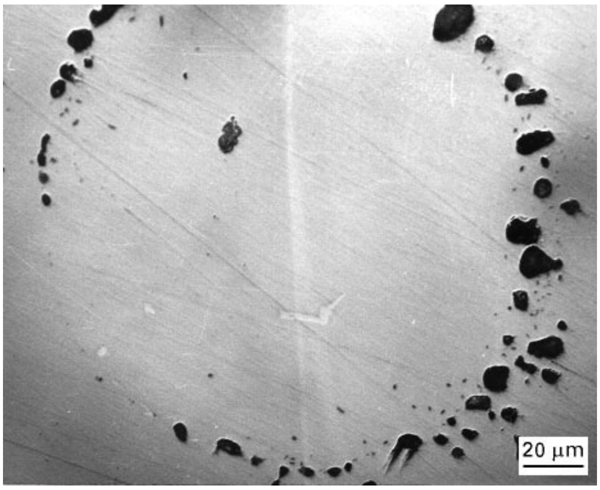

Figure 10 Pore formation around the stainless steel fibre due to the Kirkendall effect after RHP at 650 °C and heat treatment at 1200 °C for 2 h.

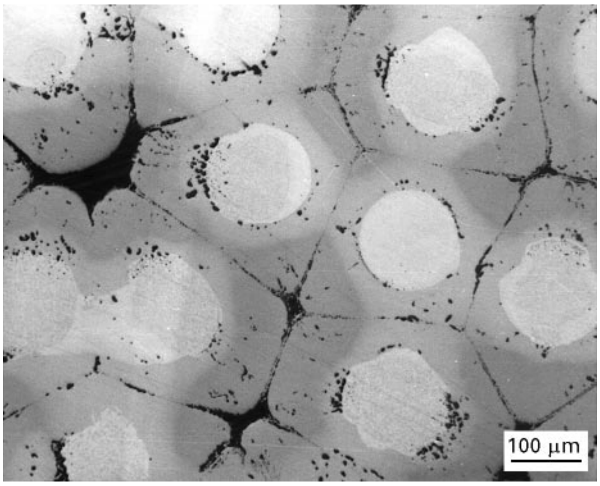

Figure 11 Microstructure of the NiAl matrix composite with a large amount of volume shrinkage after RHP at 650 °C, followed by heat treatment at 1200 °C for 2 h.

4. A volume change always occurs in non-ideal substitutional solid solution in which different kinds of atoms arrange in a long-range order pattern due to a large enthalpy decrease ($\Delta H < 0$). The volume shrinkage leads to a process problem during powder sintering of these elements. The volume changes, ΔV, during the formation of nickel aluminide are

$$3Al + 2Ni \rightarrow Ni_2Al_3 \quad \Delta V = -3.7\%$$

$$Ni_2Al_3 + Ni \rightarrow 3NiAl \quad \Delta V = -9.8\%$$

These changes were calculated from the atomic weight and the density of the aluminum, 26.98, 2.7; nickel, 58.69, 8.9; Ni_2Al_3, 198.32, 4.77; and NiAl, 85.68 g mol^{-1}, 5.913 g cm^{-3}, respectively. The formation of many large pores is expected due to volume shrinkage after complete reaction between nickel and aluminum. After RHP at 650 °C, pores existed at the reaction zone junctions as described previously and they could not be eliminated if the reaction zones had made contact with each other, even though pressure was applied. Continuous expansion of the pores, as measured by volume shrinkage, was found in the specimen after RHP at 650 °C and heat treatment at 1200 °C (Fig. 11). Several nickel aluminide phases had reacted completely and formed a homogeneous NiAl matrix. Increasing the heat treatment time exacerbated the volume shrinkage, the pores expanded in size and no sintering phenomenon occurred.

3.7. Various conditions of heat treatment and hot pressing

After heat treatment of the SSF–Ni–Al precursor at 600 or 650 °C for 48 h, complete reaction of nickel and aluminum to nickel aluminide was not realized and the aluminum still partly remained because the temperature was too low for the atoms to diffuse quickly. Some pores also formed in the aluminum phase due to the Kirkendall effect and volume shrinkage. In heat treatment at 700 or 800 °C for 8 h, the reactions were more serious. Many pores and cracks formed along the reaction zone boundaries and junctions in the specimens. After heat treatment at 700 or 800 °C, pressure was useless to densify the porous specimens due to the rigidity of the nickel aluminum skeleton. If pressure was applied to the SSF–Ni–Al precursor immediately after the temperature was raised to 700 °C, without holding the specimen at this temperature, expulsion of liquid aluminum was inevitable.

4. Conclusions

1. Stainless steel fibres were electrically plated with a uniform nickel film to form SSF–Ni composite fibres. The SSF–Ni–Al precursor was produced by diffusion bonding, which involved layering of SSF–Ni composite fibres with aluminum foils. Stainless steel fibres were distributed uniformly in the matrix. Previous nickel coating on the fibres overcame the problem of embedding fibres into the nickel.

2. The NiAl intermetallic matrix composite was produced successfully by RHP at 700 °C with pressure undertaken in a series of steps. The temperature at which the composite was fabricated was 500 °C lower than previously accomplished by hot pressing or HIP. A dense NiAl matrix composite reinforced with stainless steel fibres was obtained by the following hot pressing process at 900 °C.

3. Reaction zone boundaries existed in the specimen after RHP at 650 °C. They found due to the consumption of aluminum, layers of oxides and contaminants on the surfaces of the aluminum foils. Tubular pores formed due to the Kirkendall effect, associated with volume shrinkage, and they could not be eliminated after the reaction zones had made contact with each other because of lack of deformation of the rigid nickel aluminide skeleton. Both reaction zone boundaries and tubular pores were eliminated by RHP at 700 °C due to rapid dissolution of a large amount of nickel into liquid aluminum and uniform precipitation of Ni_2Al_3 and $NiAl_3$ in the liquid.

Acknowledgements

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References

1. D. B. MIRACLE, *Acta. Metall. Mater.* **41** (1993) 649.
2. R. D. NOEBE, R. R. BOWMAN and M. V. NATHAL, *Inter. Mater. Rev.* **38** (1993) 193.
3. K. S. KUMAR, S. K. MANNAN and R. K. VISWANADHAM, *Acta. Metall. Mater.* **40** (1992) 1201.
4. E. M. SCHULSON, *Int. J. Powder Metall.* **23** (1987) 25.
5. C. T. LIU, *Scripta Metall. Mater.* **25** (1991) 1231.
6. E. P. GEORGE, C. L. WHITE and J. A. HORTON, *ibid.* **25** (1991) 1259.
7. T. K. CHAKI, *Mater. Sci. Eng.* **A190** (1995) 109.
8. C. T. LIU and J. A. HORTON Jr, *ibid.* **A192/193** (1995) 170.
9. K. AOKI, K. ISHIKAWA and T. MASUMOTO, *ibid.* **A192/93** (1995) 316.
10. V. C. NARDONE, *Metall. Trans.* **23A** (1992) 563.
11. W. O. SOBOYEJO and S. M. L. SASTRY, *Mater. Sci. Eng.* **A171** (1993) 95.

12. K. T. V. RAO, G. R. ODETTE and R. O. RITCHIE, *Acta. Metall. Mater.* **42** (1994) 893.
13. S. NOURBAKHSH, H. MARGOLIN and F. L. LIANG, *Metall. Trans.* **20A** (1989) 2159.
14. S. NOURBAKHSH, O. SAHIN, W. H. RHEE and H. MARGOLIN, *ibid.* **22A** (1991) 3059.
15. D.C. DUNAND, J. L. SOMMER and A. MORTENSEN, *ibid.* **24A** (1993) 2161.
16. WEN-CHIH CHIOU and CHEN-TI HU, *ibid.* **25A** (1994) 985.
17. D. C. DUNAND, *J. Mater. Sci.* **29** (1994) 4056.
18. J.-P. LEBRAT and A. VARMA, *Combust. Sci. Technol.* **88** (1992) 211.
19. D. E. ALMAN, C. P. DOGAN, J. A. HAWK and J. C. RAWERS, *Mater. Sci. Eng.* **A192/193** (1995) 624.
20. J. H. SCHNEIBEL, E. P. GEORGE, C. G. MCKAMEY, E. K. OHRINER, M. L. SANTELLA and C. A. CARMICHAEL, *J. Mater. Res.* **6** (1991) 1673.
21. R. M. GERMAN and A. BOSE, *Mater. Sci. Eng.* **A107** (1989) 107.
22. D. E. ALMAN and N. S. STOLOFF, *Int. J. Powder Metall.* **27** (1991) 29.
23. A. VARSCHAVSKY and P. TAMAYO, *J. Mater. Sci.* **4** (1969) 653.
24. J. KESAPRADIST, K. ONO and K. FUKAURA, *Mater. Sci. Eng.* **A153** (1992) 641.
25. M.M.P. JANSSEN and G.D. RIECK, *Trans. AIME* **239** (1967) 1372.
26. A. SMIGELSKAS and E. KIRKENDALL, *ibid.* **171** (1947) 130.
27. L. DARKEN, *ibid.* **174** (1948) 184.
28. NING ZHANG, P. B. KHOSROVABADI, J. H. LINDENHOVIUS and B. H. KOLSTER, *Mater. Sci. Eng.* **A150** (1992) 263.

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