Effect of cold rolling and subsequent annealing on hot pressed Ni/Al laminates

K. MUMTAZ, J. ECHIGOYA, C. NAKATA
Department of Materials Science and Technology, Iwate University, Morioka, Japan
S. W. HUSAIN
Dr. A. Q. Khan Research Labs, Rawalpindi, Pakistan
E-mail: pvs.paknet2.ptc.pk

Nickel/Aluminum laminates have been prepared by hot pressing of stacked alternate layers of very thin foils of nickel and aluminium. It is found that ductile nickel layers exist in hot pressed Ni/Al laminates which are annealed up to 600 °C. However, at these temperatures Al-rich intermetallic compounds are also formed with a very thin irregular layer of pure aluminium and few voids. As the annealing temperature is increased, nickel layer is consumed and Ni-rich intermetallic compounds are formed. Cold rolling up to 8% reduction in area resulted in the cracking of the regions having intermetallic compounds with cracks perpendicular to the rolling direction. The tensile strength values are high for the laminates which contain ductile Ni layers and reach a value of 320 MPa for the specimens which are hot pressed at 420 °C. However, the laminates which are annealed at 800 °C show brittle fracture and lower tensile strength of 174 MPa. The fractographic examination indicated that Ni/Al laminates which are hot pressed at 420 °C undergo plastic deformation before breaking. On the other hand, Ni/Al laminates which are annealed at 800 °C with Ni present in the form of intermetallic compounds only, exhibit brittle intergranular fracture.

1. Introduction

Extensive attention has been paid to the microstructures and the properties of alloys in Ni-Al system due to their high temperature properties [1, 2]. Numerous ways have been attempted to process these alloys with improved properties [3, 4]. Multilayer materials are much attractive in mechanical properties as well as certain physical properties. Bulk materials with layered structures have been attempted using stacks of pure compact foils and then diffusion bonding at high temperature followed by rolling [5]. Nickel/aluminium laminates are of interest as structural materials due to their high specific strength and modulus. The major drawback associated with these laminates is the formation of brittle intermetallic compounds at temperature above about 400 °C. The four intermetallic compounds which are formed in Ni/Al system at different temperatures are $NiAl_3$, Ni_2Al_3, NiAl and Ni_3Al and these have been studied widely by different researchers [6]. These intermetallic compounds generally fail by cracking or fracturing due to interfacial decohesion. Therefore, the formation of these intermetallic compounds limit the extent of commercial use of Ni/Al laminates. If the tendency for crack formation in the intermetallic compounds of Ni and Al could be reduced, this would result in the higher tensile strength as well as higher ductility in the laminates. Therefore, with the aim to achieve good ductility and strength in the laminates, nickel/aluminum laminates have been made by hot pressing the alternately stacked foils of Ni and Al. Attempts have been made to enhance the laminate properties by producing the laminates with intermetallic compounds of Ni and Al sandwiched between high melting and ductile, pure Ni metal layers and to get intact and continuous interface between Ni and the intermetallic compounds. Higher ductility is expected in the laminates which are hot pressed, cold rolled and annealed at low temperatures. Pure metals are not susceptible to intergranular and transgranular cracking whereas intergranular brittle fracture is a common failure mode in intermetallic compounds.

The cold rolling of these laminates further decreases the thickness of layers and collapses the voids, which are then healed up in the subsequent annealing treatment. The aim of this study is to elucidate the existence of Ni layers in Ni/Al laminates and the formation of different intermetallic compounds at temperature range of 420–1000 °C. The tensile properties of these laminates are also evaluated and the behaviour of the laminates with and without ductile Ni layers is studied.

2. Experimental procedure

The materials used for laminate formation were nickel and aluminum foils, both 99.5% pure, 0.01 mm thick, and purchased from The Nilaco Corporation, Japan. Ni and Al foils were cut to size of 0.01 mm thickness × 4 mm width × 8 mm length. About 400 foils of Ni and Al were alternately stacked in a graphite holder. For laminate formation, the stacked foils were hot pressed at temperature of 420 °C with load of 50 kilogram for 30 minutes in vacuum (pressure 4.5×10^{-5} torr). The Ni/Al laminates were then cold rolled to 8% reduction in area. After cold rolling, the laminates were annealed at different temperatures (500 °C to 1000 °C) for 30 minutes in Ulvac Infrared Image furnace. Fig. 1 schematically shows the manufacturing process of Ni/Al laminates. Some unrolled specimens were also prepared. Tensile samples were prepared by Spark wire cut machine (Fine Sodick Wire Cut EDM). The geometry of the tensile specimens with a 2 mm gauge length and 1 mm width is shown in Fig. 2. The specimen was stretched with a constant strain rate (0.5 mm/minute) by a tensile testing machine (AGS-1kND, Shimadzu Type SBL-1kN). A scanning electron microscope (SEM), electron probe X-ray micro analyzers (EPMA) equipped with wavelength dispersive spectrometer (WDS) were used to study the microstructures and the formation of intermetallic compound in Ni/Al laminates in as hot pressed, cold rolled, and subsequently annealed conditions. Various phases were identified by the chemical composition, i.e., by the Ni/Al ratio. Microfractographic examination of tensile tested specimens was also carried out.

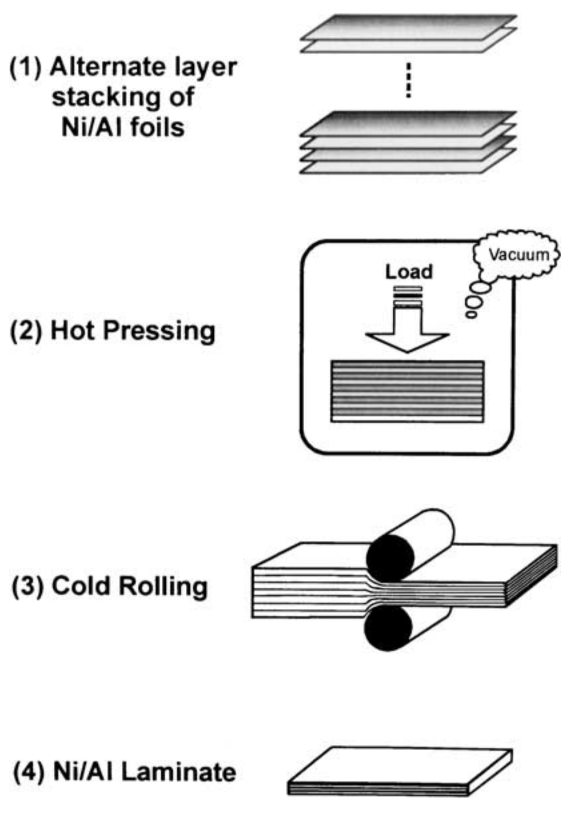


Figure 1 Schematic representation of the manufacturing process of Ni/Al laminates.

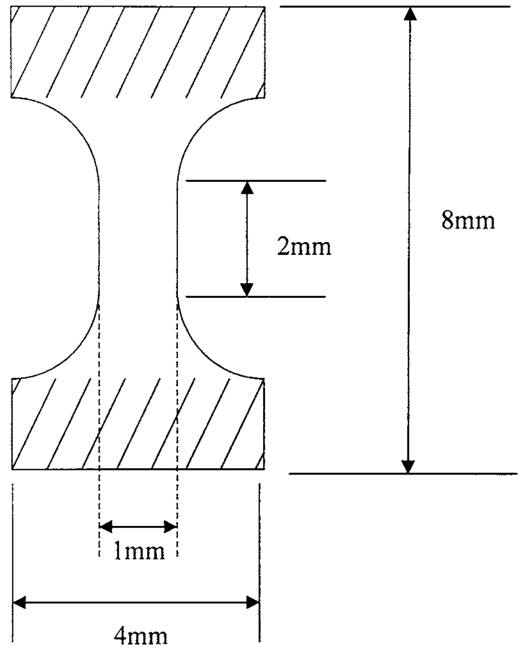


Figure 2 The geometry of the tensile specimens of the laminates.

3. Results and discussion

Fig. 3 clearly shows the presence of about 6 μm intact continuous layers of Ni metal in Ni/Al laminates which are hot pressed at 420 °C. The presence of Al layers (black region) was also indicated by EPMA analyses. It is found that the interfacial reaction was initiated in the Ni/Al laminates at temperature of 420 °C with the initial formation of Ni_2Al_3 and subsequently $NiAl_3$ intermetallic compounds were formed, both of which appear as light and dark layers respectively. Ni_2Al_3 layers of about 3.2 μm which are formed at the Ni-interface are also quite continuous. All the layers are intact and continuous but voids are formed in the center of intermetallic compound layers and also few in Ni layers.

The Ni/Al laminates were then cold rolled and annealed at different temperatures to examine the inter-diffusion and the formation of different intermetallic compounds. Fig. 4 shows that the specimens, which are hot pressed at 420 °C and cold rolled to 8% reduction in area, contain continuous Ni layers of thickness about 5 μm. However, cracking is observed in Ni_2Al_3 and $NiAl_3$ intermetallic compounds. These cracks are perpendicular to the rolling direction.

After cold rolling the specimens were hard. The annealing of such laminates induces softening. The annealed specimens show that the Ni layers are gradually consumed. Fig. 5 shows the 500 °C annealed Ni/Al laminates. In this case NiAl is the dominant intermetallic while the thickness of $NiAl_3$ and Ni_2Al_3 intermetallic layers is too small to be noted.

Fig. 6 shows the sample annealed at 700 °C. During annealing at these higher temperatures several changes occurred to a varying extent. Most prominent change was the entire diffusion of Ni layers and the formation of Ni-rich intermetallic compound of Ni_3Al (white layer).

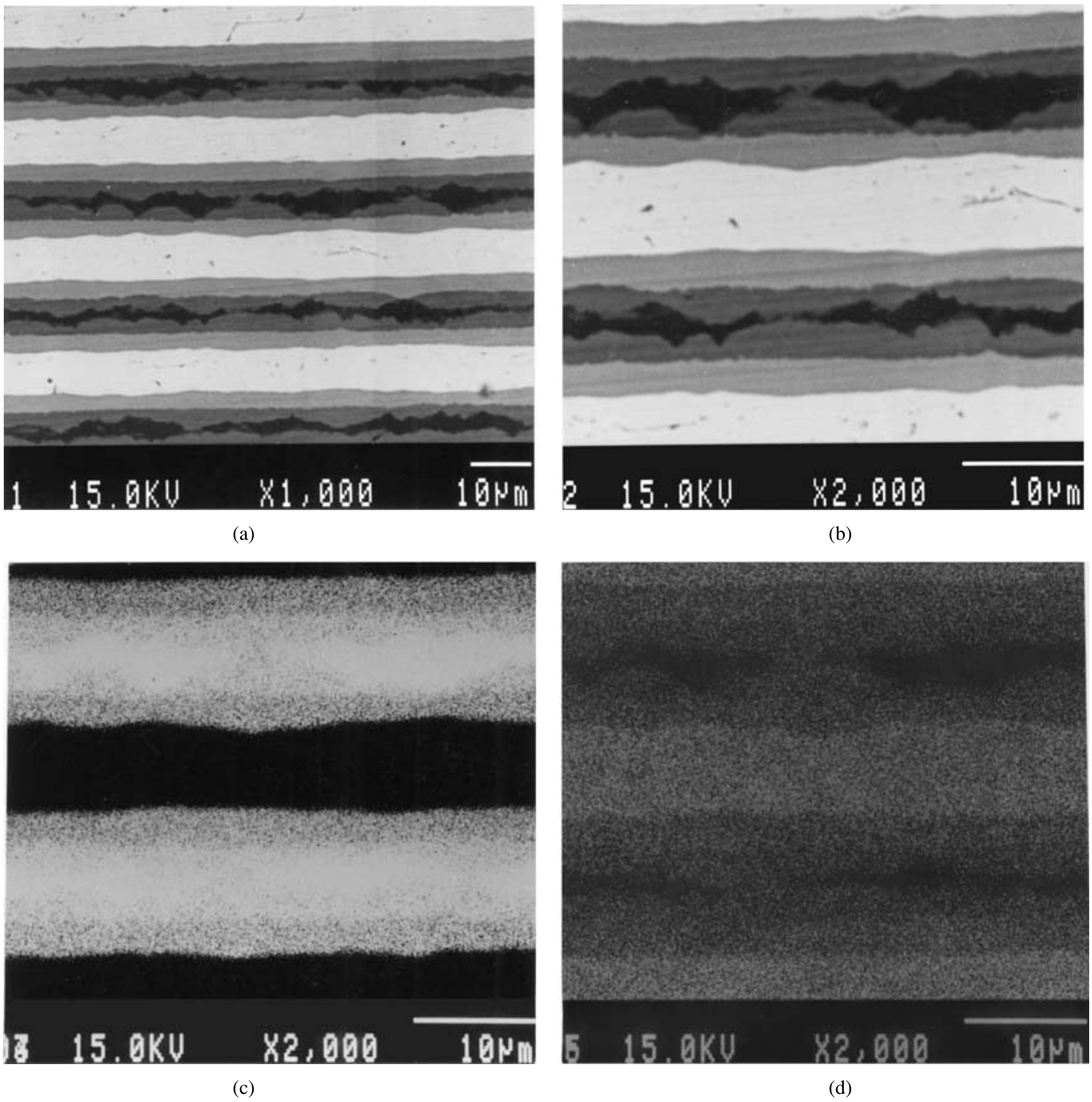


Figure 3 The microstructural and chemical information obtained from the sample hot pressed at 420 °C. (a) and (b) show microstructure at lower and higher magnifications. The white layer is Ni while the black layer is Al. The gray layers are the intermetallics, light gray being Ni_2Al_3 and dark gray $NiAl_3$ (c) x-ray mapping using Al kα_1 (d) x-ray mapping using Ni kα.

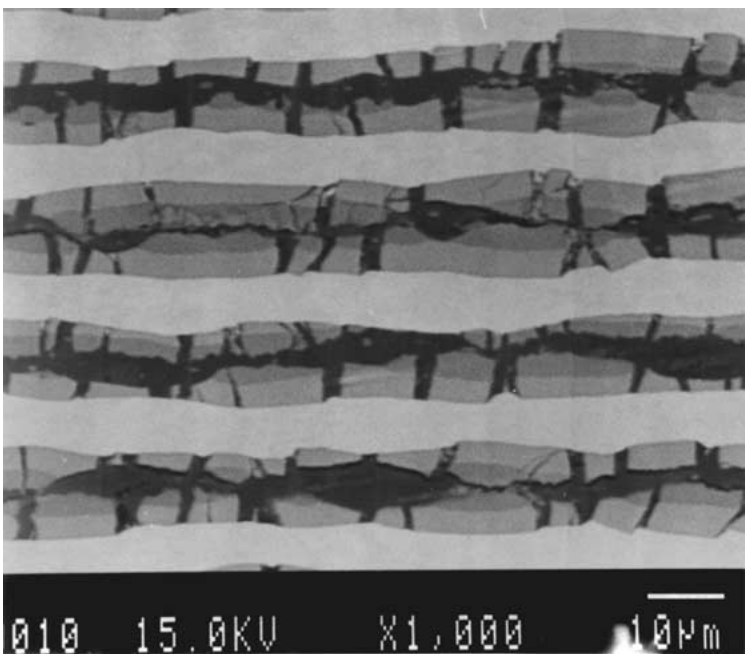


Figure 4 The microstructural information obtained from the sample hot pressed at 420 °C and then cold rolled to 8% reduction in area. Note that the intermetallic compound layers show cracks perpendicular to the rolling direction.

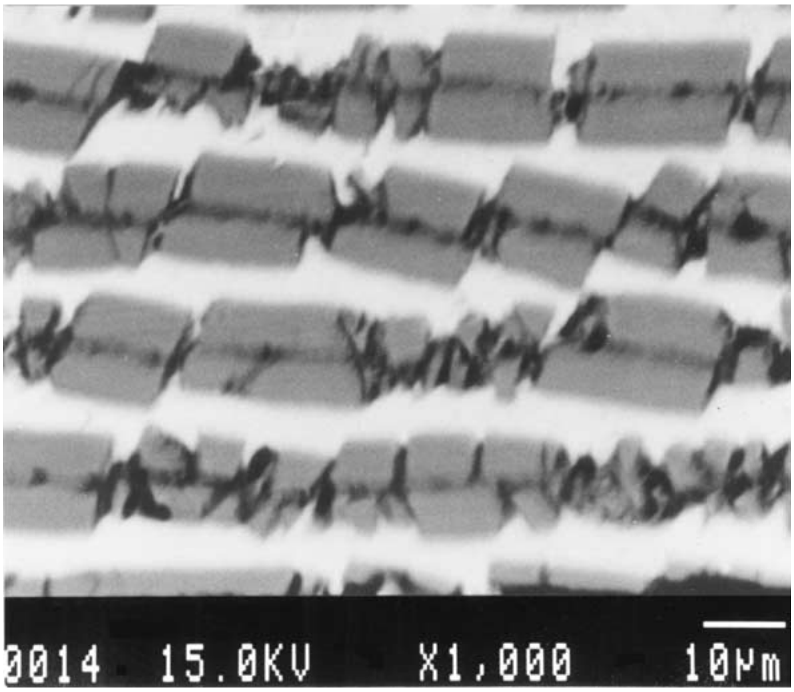


Figure 5 The microstructure of the sample hot pressed at 420 °C, cold rolled to 8% reduction in area and then annealed at 500 °C. Here, NiAl is the dominant intermetallic while the thickness of $NiAl_3$ and Ni_2Al_3 intermetallic layers is too small to be noted.

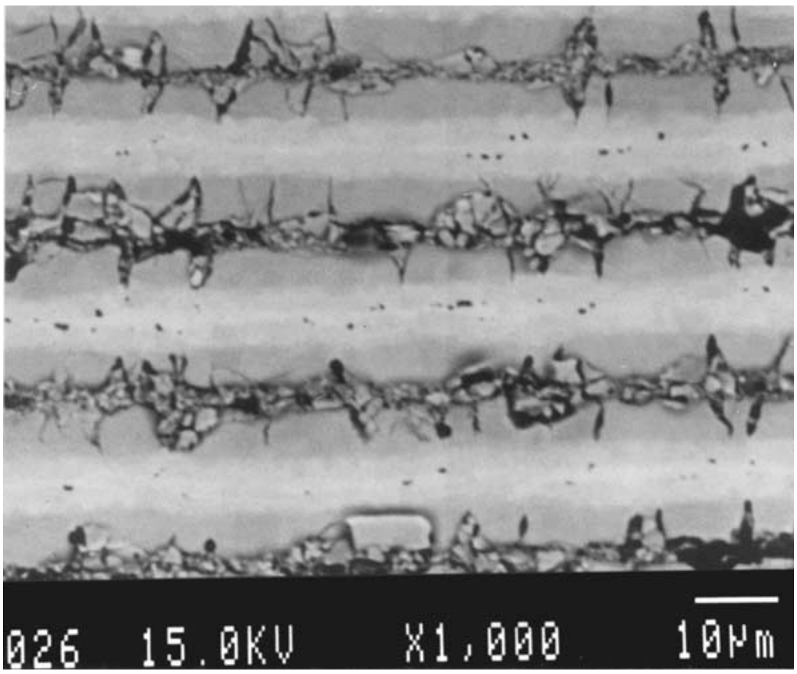


Figure 6 The microstructure of the sample hot pressed, cold rolled and then annealed at 700 °C. The Ni layer has now vanished. The Ni-rich intermetallic compound Ni_3Al (white layer) can be noted. This is in addition to the presence of other intermetallics NiAl, $NiAl_3$ and Ni_2Al_3.

This is in addition to the presence of other intermetallics NiAl, $NiAl_3$ and Ni_2Al_3, in accordance with the Ni/Al phase equilibria. Some voids are also noted in the center of Ni_3Al layer. The crack density is further reduced. As depicted in Fig. 7, the phases present in the samples annealed at 800 °C are similar to those in the samples annealed at 700 °C and the density of cracks as well as the voids is further reduced. It is also noted that the thickness of Ni_3Al (white layer) is decreased. The growth of NiAl continues at the expense of Ni_3Al as the annealing temperature is further increased to 900 °C, as shown in Fig. 8. This is accompanied by decrease

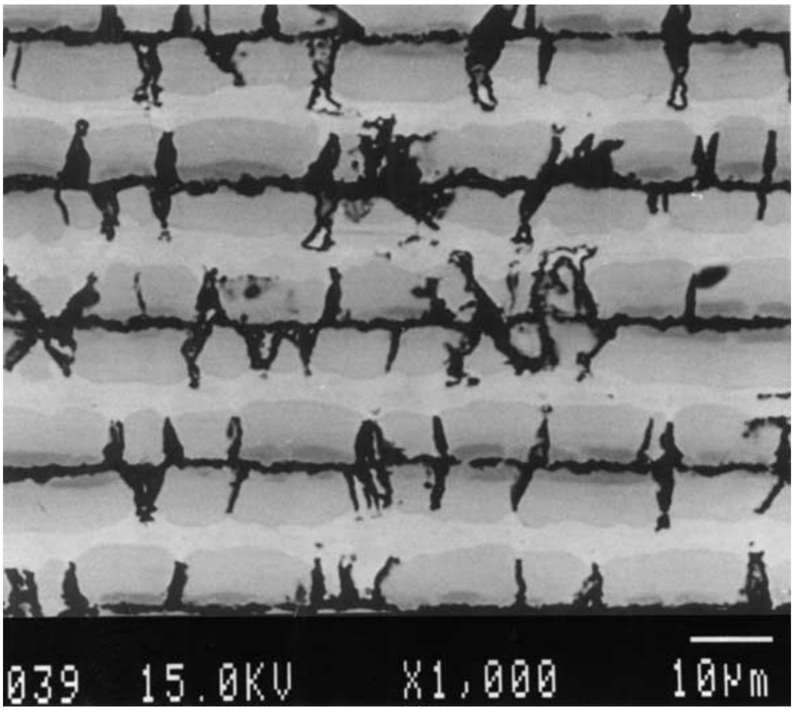


Figure 7 The microstructure of the sample hot pressed, cold rolled and then annealed at 800 °C. The density of cracks as well as the voids is further reduced. The thickness of Ni_3Al (white layer) is decreased and the growth of NiAl (light gray) continues at the expense of Ni_3Al.

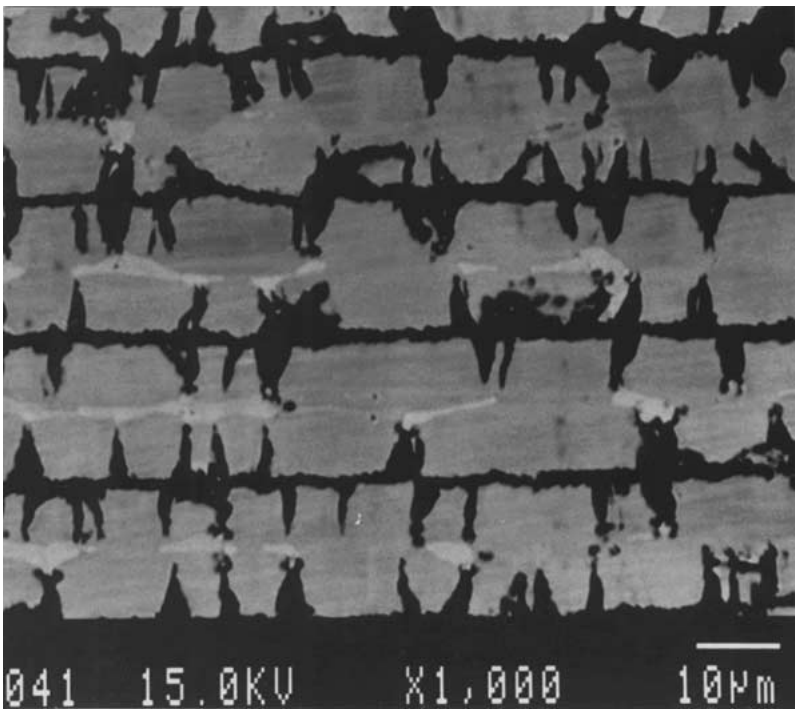


Figure 8 The microstructure of the sample hot pressed, cold rolled and then annealed at 900 °C. The growth of NiAl continues at the expense of Ni_3Al.

in crack density and voids. Similar trend is noted in samples annealed at 1000 °C. As evident in Fig. 9, the samples annealed at 1000 °C start developing the equiaxed structure and the layered structure is getting less dominant.

As far as the mechanical properties are concerned, the laminates which contain ductile Ni layers show high strength. As shown in Fig. 10, the tensile strength values for such laminates are high and reach a value of 320 MPa for the specimens which are hot pressed at

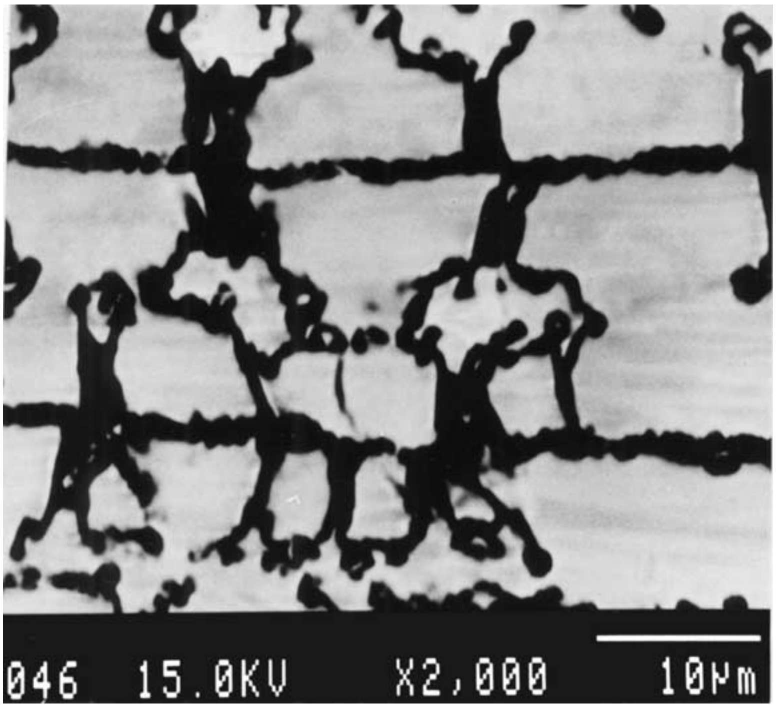


Figure 9 The microstructure of the sample hot pressed, cold rolled and then annealed at 1000 °C. The dominant phase is NiAl and the equiaxed structure starts developing.

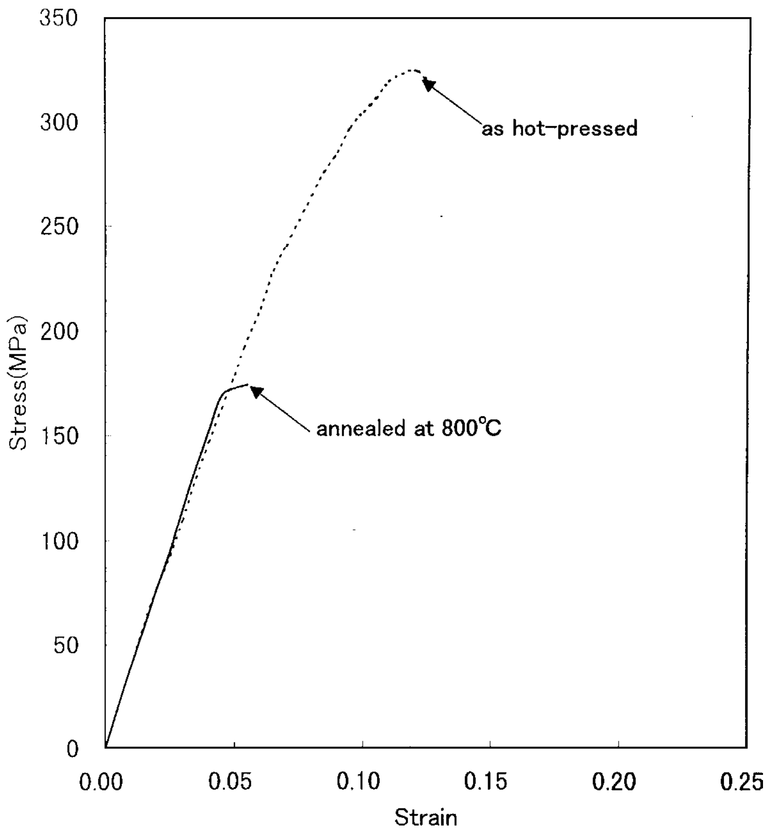
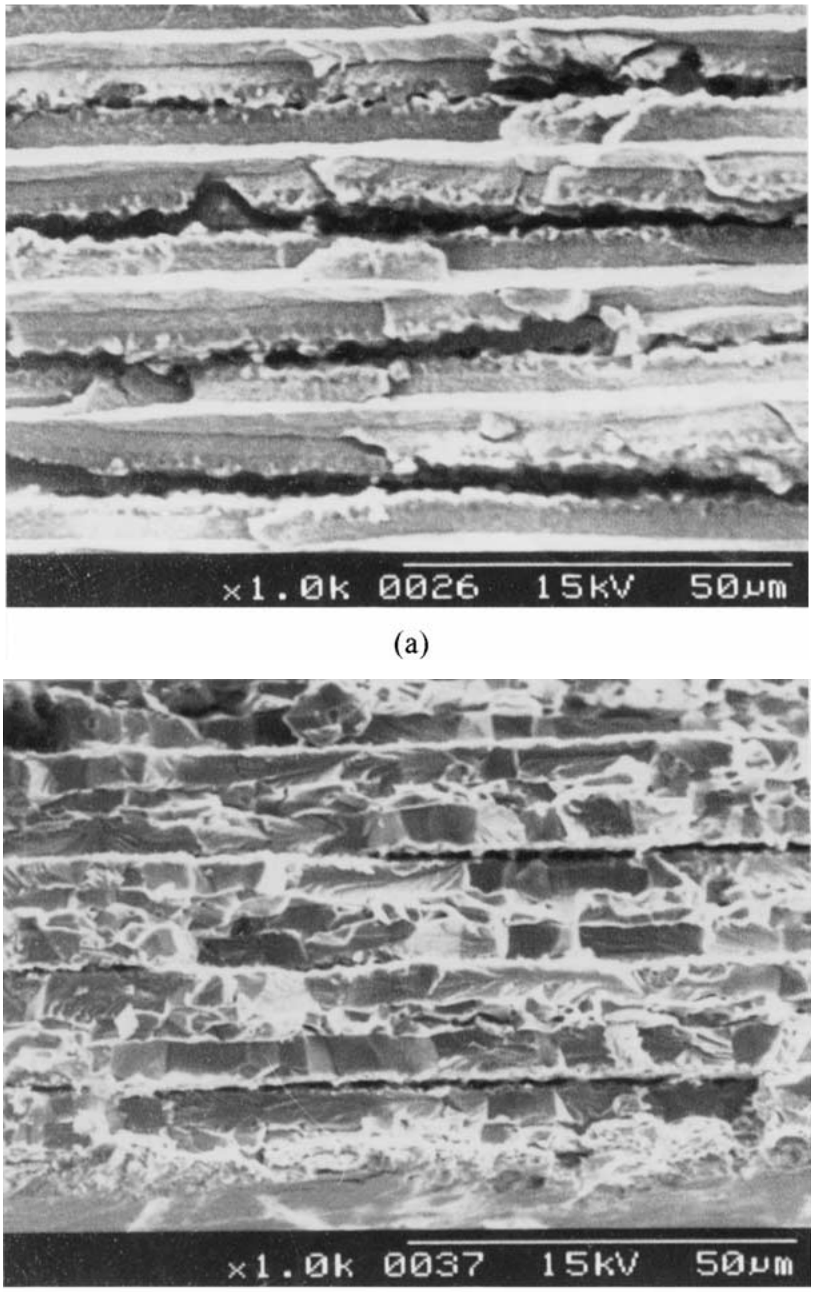


Figure 10 Stress-Strain curve obtained from the tensile test of hot pressed samples and the samples hot pressed, cold rolled and then annealed at 800 °C.

420 °C. However, the laminates which are annealed at higher temperatures show brittle fracture and lower tensile strength. For example, the laminates annealed at 800 °C show tensile strength of 174 MPa only. The fractographs shown in Fig. 11, indicate that Ni/Al laminates which are hot pressed at 420 °C undergo plastic deformation before breaking. These laminates contain layers of pure Ni. On the other hand, Ni/Al laminates which are annealed at 800 °C with Ni present in the form of intermetallic compounds only, exhibit brittle intergranular fracture after only a little plastic deformation.



(a)

(b)

Figure 11 The fractographs obtained from the samples broken in the tensile test of (a) hot pressed samples and (b) the samples hot pressed, cold rolled and then annealed at 800 °C.

Acknowledgements
We thank Mr. K-Nonaka for his valuable help in the EPMA examination of the specimens. The financial support provided by the Iwate University, Morioka, Japan is gratefully acknowledge.

References

1. WESTBROOK, *Trans. AIME* **209** (1957) 898.
2. THORNTON, R. G. DAVIES and T. L. JOHNSTON, *Metall. Trans.* **1** (1970) 207.
3. GANG LI, ZHONG-GUANG WANG, CHANG-XU SHI and V. OVCHARENKO, "Strength of Materials," edited by Oikawa *et al.* (The Japan Institute of Metals, 1994).
4. AHMED, Ph.D. Dissertation, University of Connecticut, 1985.
5. SHIOMI KIKUCHI, HIDEYUKI KUWAHARA and SYUNJI URAI, "Strength of Materials," edited by Oikawa *et al.* (The Japan Institute of Metals, 1994).
6. PHILPOT, Z. A. MUNIR and J. B. HOLT, *J. Mater. Sci.* **22** (1987) 207.

Received 31 January 2000
and accepted 13 January 2001