

## The Effect of Hot Rolling on Room Temperature Ductility of a NiAl Intermetallic Compound

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**Abstract.** A Ni<sub>57</sub>Fe<sub>18</sub>Al<sub>25</sub> alloy is produced by vacuum induction melting under an argon atmosphere and gravity cast into a copper chill mould. Ingots of 2 x 10 x 50 mm are re-heated at 1100 °C for 24 hours and hot rolled until a 25 % reduction is reached. Immediately, hot rolled plates are heat treated at 1100 °C for 1 hour and water cooled to room temperature. Microstructures of as cast, hot rolled and hot rolled plus heat treated specimens are characterized using scanning and transmission electron microscopy and x-ray diffraction. Specimens in the as-cast and hot rolled conditions show the presence of equiaxial and partially elongated β-grains, respectively, with the presence of a thin γ-phase decorating the grain boundaries. Hot rolled plus heat treated specimens have partially recrystallized β-grains with γ-phase and (Ni,Fe)<sub>3</sub>Al at the grain boundaries. The hot rolled specimens have a room temperature ductility of approximately 5 % in tensile testing. This enhancement in ductility is attributed to the modification of the β-grains by coexistence with a γ-phase along the grain boundaries.

### Introduction

The NiAl intermetallic compound has been identified as a potential candidate for high temperature structural applications because it has high melting temperature, low density, good oxidation resistance, attractive Young's modulus, high thermal conductivity, metal-like properties and cheap raw materials [1-3]. However, the main problems, which have hindered its industrial application, are poor ductility and low fracture toughness at room temperature, particularly because of grain boundary embrittlement [4]. In order to improve the tensile room temperature ductility of the β-phase, research has focused on the effect of additions of microalloying and macroalloying elements, controlling the microstructure through processing, and fiber strengthening [5]. It has been mentioned that brittleness is not an inherent feature of the β-phase. Single crystal and polycrystalline samples with a very fine grain size can exhibit a fair ductility [6]. Recently, it has been reported that addition of alloying elements, such as cobalt, chromium, copper and iron to NiAl (B2)-base alloys causes a positive effect on the hot workability and tensile ductility. Values up to 10.2 % of tensile elongation at room temperature for a hot rolled plus heat treated NiAlCo alloy have been reported. This increase in room temperature tensile ductility has been attributed to the formation of a γ-phase that decorates the grain boundaries of the β-phase [7]. This work reports results of copper chill cast ingots, hot rolled plates and hot rolled plus heat treated plates, in terms of the effect of Fe additions on room temperature ductility of NiAl (B2)-base alloy.

### Experimental Procedure

A  $\text{Ni}_{57}\text{Fe}_{18}\text{Al}_{25}$  alloy was prepared using an Inductotherm vacuum induction melting furnace under an argon atmosphere and gravity cast in copper chill moulds. The resulting ingots with dimensions of  $2 \times 10 \times 50$  mm were heat treated in a Thermolyne resistance furnace at  $1100^\circ\text{C}$  for 24 hours and hot rolled in a 2-Hi Fenn Mill until a 25 % reduction in thickness was achieved. Then, the hot rolled specimens were heat treated at  $1100^\circ\text{C}$  for 1 hour and immediately water cooled to room temperature. The microstructures of the as-cast, hot rolled and hot rolled plus heat treated specimens were characterized using a Stereoscan 440 scanning electron microscope (SEM), a Jeol 2100 scanning transmission electron microscope (STEM) and a Siemens D-5000 X-ray diffractometer. Evaluation of the mechanical properties of as-cast, hot rolled and hot rolled plus heat treated samples were carried out in an Instron 1125 testing machine at a crosshead speed of  $0.05$  mm/min.

### Results and Discussion

Fig. 1a shows part of the ternary Ni-Al-Fe phase diagram. A circle within the  $\beta + \gamma$  region indicates the position of the alloy investigated in the present work. The average chemical composition of the alloy was  $57.8 \pm 1.2$  at. % Ni,  $24.7 \pm 1.3$  at. % Al and  $17.5 \pm 1.5$  at. % Fe.

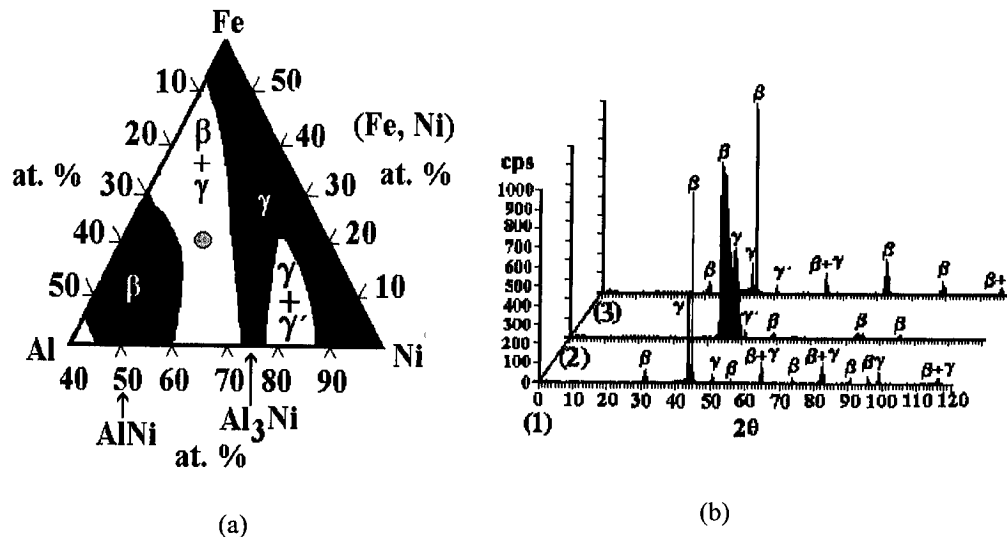


Figure 1. (a) A portion of the ternary NiAlFe phase diagram. The alloy studied in this work is indicated by a gray circle in the  $\beta + \gamma$  phase field, (b) X-ray diffraction patterns of (1) as-cast ingots, (2) hot rolled plates and (3) hot rolled plus heat treated plates.

Figure 1b shows X-ray diffraction patterns obtained from as-cast, hot rolled and hot rolled plus heat treated specimens. All samples show the presence of a main phase, identified as the  $\beta$ -phase, with a cubic lattice parameter  $a = 2.8650 \text{ \AA}$ . The  $\gamma$ -phase was also detected. In addition to these two phases, in the hot rolled plus heat treated specimens, the  $\gamma'$ -phase was positively identified.

During observations of the as-cast ingots in the SEM, an equiaxed  $\beta$ -dendrite microstructure with the  $\gamma$ -phase in interdendritic regions was observed (Figs. 2a and 2b). Equiaxed grains showed a maximum size of  $45 \pm 4 \mu\text{m}$  and the interdendritic  $\gamma$ -phase showed a maximum width of  $2 \mu\text{m}$ . Bands of deformed  $\beta$ -phase showed a width between 10 to  $12 \mu\text{m}$  and, along deformed  $\beta$ -grains, the  $\gamma$ -phase showed a maximum width of  $0.8 \mu\text{m}$ , as shown in Figs. 2c and 2d. Hot rolled plus heat

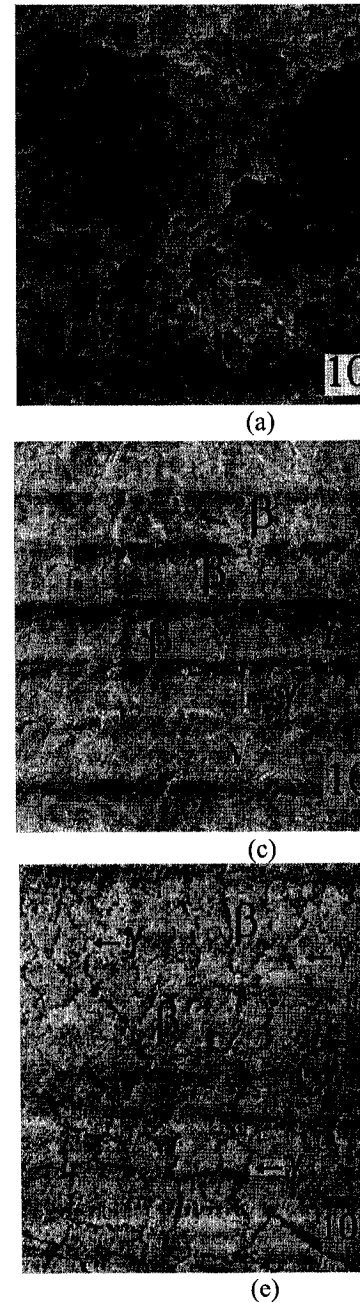


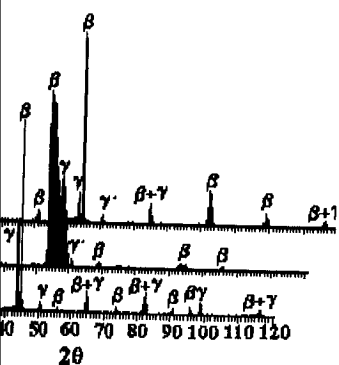
Figure 2. (a), (b) As-cast, (c), (d) hot rolled plus heat treated specimens observed in Ni-Fe-Al alloys. Arr

treated specimens showed partial grain boundaries (Fig. 2e), and also

Table 1 shows the EDX analysis of as-cast ingots, hot rolled plates

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(b)

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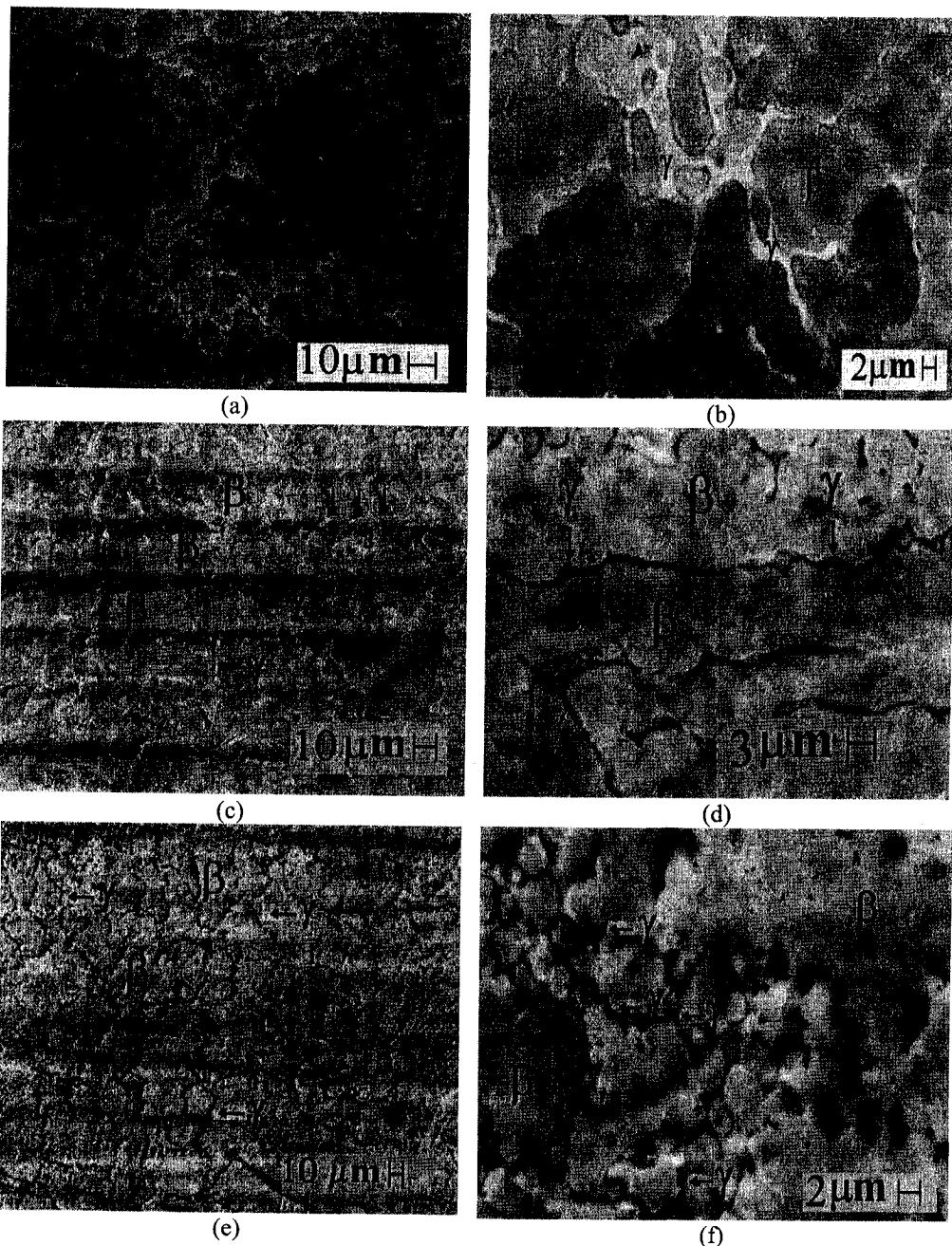


Figure 2. (a), (b) As-cast, (c), (d) hot rolled and (e), (f) hot rolled plus heat treated microstructure observed in Ni-Fe-Al alloys. Arrows in Fig. 2c show the presence of hot bands.

treated specimens showed partially recrystallized  $\beta$ -grains with continuous  $\gamma$ -phase formed at the grain boundaries (Fig. 2e), and also the presence of spherical particles of  $\gamma'$ -phase (Fig. 2f).

Table 1 shows the EDX analyses made at the grain boundaries of the  $\beta$  and  $\gamma$  phases observed in as-cast ingots, hot rolled plates and hot rolled plus heat treated plates. As can be observed, the

amount of Ni and Al in the  $\beta$ -phase remains almost constant; the Fe content does not change. With respect to the  $\gamma$ -phase (as compared with the  $\beta$ -phase), it is richer in Ni and Fe, and has less Al. Ishida et al. [7] reported that, at elevated temperature, the  $\beta/\gamma$  equilibrium becomes stable by the addition of elements such as Co, Cr, Cu, Fe and Mn as shown in Fig. 3. This condition was obtained in the plates hot rolled at 1100 °C, where a two phase structure of  $\beta$ -phase matrix accompanied by a  $\gamma$ -phase was obtained by alloying NiAl with Fe.

Table 1. EDX-microanalysis performed in phases present in ingots, plates and heat treated plates.

Condition	Phase	Ni [at. %]	Al [at. %]	Fe [at. %]
Cast	$\beta$	54.6	27.3	18.1
	$\gamma$	61.0	15.2	23.8
Hot rolled	$\beta$	54.3	27.6	18.1
	$\gamma$	58.6	15.6	25.8
Hot rolled + heat treated	$\beta$	55.7	26.2	18.1
	$\gamma$	58.8	13.0	28.2
	$\gamma'$	55.5	18.8	25.7

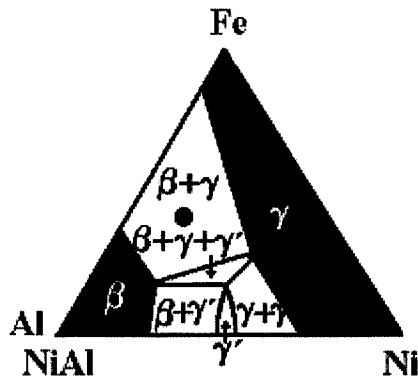


Figure 3. Part of the ternary Ni-Al-Fe phase diagram at elevated temperature.

For the hot rolled plus heat treated plates,  $\gamma'$ -phase precipitated at grain boundaries. Precipitation of this  $\gamma'$ -phase occurred in regions of  $\gamma$ -phase, where the increase in Fe content gave the conditions for the precipitation of this phase at 1100 °C.

Figure 4 shows the tensile tests results at room temperature for the as-cast and hot rolled specimens. As can be observed, the stress strain curve for the as-cast specimens has a maximum tensile strength of ~600 MPa and an elongation of ~2.86 %. The hot rolled specimens have a plateau at ~270 MPa. The tensile strength reaches a value of ~570 MPa. The percentage elongation to fracture of the hot rolled specimens was ~5.0 %. This improvement of elongation for the hot rolled specimens was attributed to the presence of fragmented secondary dendrite arms of  $\beta$ -phase, which are surrounded by the  $\gamma$ -phase (see Fig. 5). The hot rolled plus heat treated specimens have zero ductility due to the presence of the  $\gamma'$ -(Ni, Fe)<sub>3</sub>Al precipitates at  $\beta$ -phase.

Stress (MPa)

Figure 4. Stress-strain

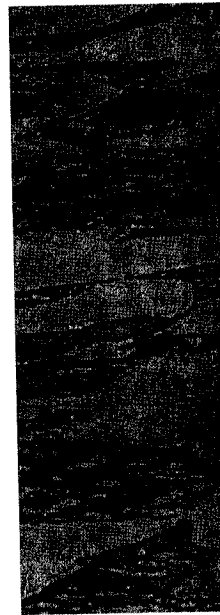


Figure 5. A micro

### Conclusions

- 1.- The tensile ductility at room temperature was improved. This was higher for the hot rolled specimens with elongated  $\beta$ -grains with  $\gamma$ -phase.
- 2.- The improvement in ductility was attributed to the presence of fragmented secondary dendrite arms of  $\beta$ -phase, which are surrounded by the  $\gamma$ -phase.

the Fe content does not change. With an increase in Ni and Fe, and less Al, the equilibrium becomes stable by the formation of  $\beta$ -phase matrix accompanied by a

plates and heat treated plates.

Al [at. %]	Fe [at. %]
27.3	18.1
25.2	23.8
27.6	18.1
25.6	25.8
26.2	18.1
23.0	28.2
28.8	25.7

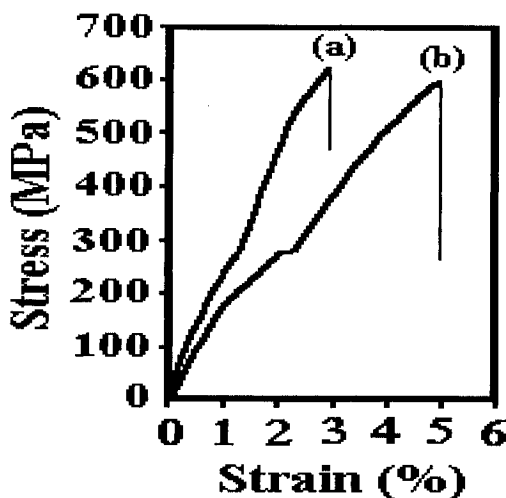


Figure 4. Stress-strain curves obtained in NiFeAl intermetallic compounds in the (a) as cast and (b) rolled conditions.

**Ni**

at elevated temperature.

at grain boundaries. Precipitation of  $\beta$ -phase in Fe content gave the conditions

for the as-cast and hot rolled specimens. The as-cast specimens have a maximum yield strength of approximately 100 MPa. The hot rolled specimens have a yield strength of approximately 180 MPa. The percentage elongation to failure for the hot rolled specimens is approximately 5%. The improvement of elongation for the hot rolled specimens is attributed to the fragmentation of dendrite arms of  $\beta$ -phase, which are fragmented into small particles of  $\beta$ -phase, plus heat treated specimens have a yield strength of approximately 180 MPa at  $\beta$ -phase.

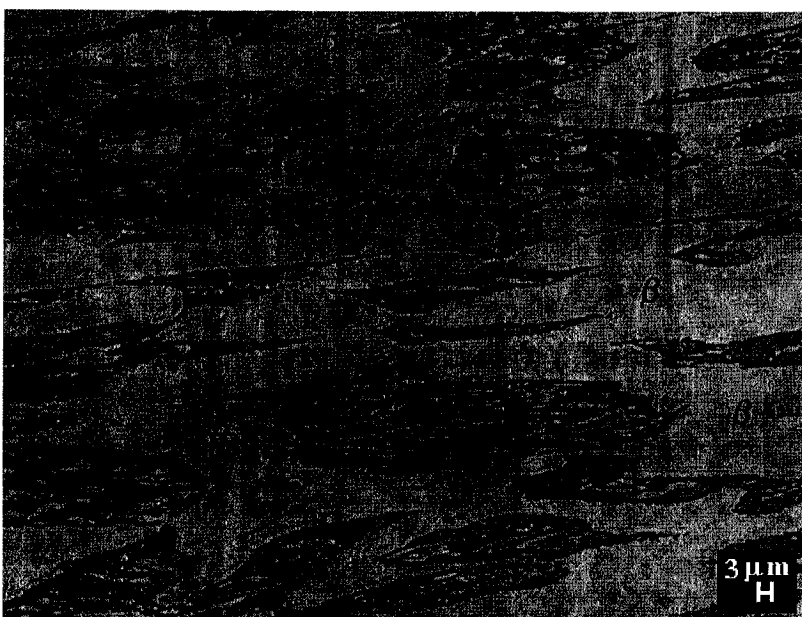


Figure 5. A microstructure of hot rolled specimens which shows partially fragmented  $\beta$ -phase in  $\gamma$ -phase.

**Conclusions**

- 1.- The tensile ductility at room temperature for as-cast and hot-rolled  $Ni_{57}Fe_{18}Al_{25}$  alloys was improved. This was higher for the hot-rolled specimens, which showed a microstructure of elongated  $\beta$ -grains with  $\gamma$ -phase at grain boundaries.
- 2.- The improvement in ductility was attributed mainly to the formation of the  $\gamma$ -phase that

decorates the grain boundaries of the  $\beta$ -phase.

### Acknowledgments

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### Grain Refin

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**Keywords:** Superplasticity,

**Abstract.** In the present st  
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### Introduction

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