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## TEM STUDY OF PRECIPITATION IN A NiAl-3Ti-0.5Hf SINGLE-CRYSTAL ALLOY

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Three directionally solidified NiAl single-crystal alloys, NiAl-3Ti, NiAl-0.5Hf and NiAl-3Ti-0.5Hf (at.%), were grown by a Bridgeman technique using high purity alumina crucibles. The ingots were homogenized for 32h at 1644 K followed by aging for 6h at 1255 K and finally furnace cooled under an argon atmosphere. This heat treatment was found to be very effective in dissolving Hf-rich interdendritic particles that were present in the as-cast structure, and at the same time it produced fine second-phase precipitates in the alloy.

Samples for transmission electron microscopy (TEM) were prepared from 3 mm diameter cylinders electro-discharge machined from the heat-treated ingots. Slices sectioned from the cylinders were mechanically ground and electrochemically thinned in a twin-jet Tenupol-3 polisher. Microstructural and energy-dispersive X-ray spectroscopy (EDXS) studies were conducted in a Philips 400T TEM equipped with a double tilt goniometer and a KEVEX Si/Li X-ray detector.

TEM microstructures of the NiAl-3Ti and NiAl-0.5Hf samples were featureless except for the presence of some random dislocations. No homogeneous precipitation was observed in either of the two alloys suggesting that 3 at.% Ti in the first alloy and 0.5 at.% Hf in the second alloy were in solid solution in the NiAl matrix. The microstructure of the NiAl-3Ti-0.5Hf alloy was, however, very different and showed a high density of precipitates. A TEM bright-field image of the alloy close to a  $\langle 001 \rangle_{\text{NiAl}}$  zone-axis is presented in Fig. 1(a). It shows a high density of fine precipitates (size ~ 5-50nm) distributed uniformly throughout the foil, and some coarse precipitates (size ~ 100-700nm) nucleated preferentially on dislocations on  $\{100\}_{\text{NiAl}}$  planes. The coarse precipitates were always associated with precipitate free zones. The fine precipitates, shown at a higher magnification in Fig. 1(b), clearly exhibited a plate-shape morphology with associated coherency strain. Using EDXS, electron diffraction (Fig. 1(c)) and trace analysis these fine precipitates were identified as Ni<sub>2</sub>AlTi Heusler phase ( $L2_1$ ,  $a = 0.587\text{nm}$ ) formed on the  $\{100\}_{\text{NiAl}}$  planes with a cube-on-cube orientation relationship. Two types of coarse precipitates were found to nucleate on dislocations; edge-on plates (marked T, size ~ 100-250nm) and almost equiaxed precipitates (marked H, size ~ 200-700nm). These precipitates were also found to be Heusler phases; T: Ni<sub>2</sub>AlTi, H: Ni<sub>2</sub>AlHf ( $L2_1$ ,  $a = 0.608\text{nm}$ ). Both of these phases are formed by further ordering of the B2 structure of NiAl (ordered BCC,  $a = 0.289\text{nm}$ ) during cooling<sup>1,2</sup>. The lattice misfits of the Ni<sub>2</sub>AlTi and Ni<sub>2</sub>AlHf phases with the NiAl matrix are 1.5 % and 5.2 %, respectively. Due to large misfit of the Ni<sub>2</sub>AlHf phase, a square network of misfit dislocations of spacing ~ 5.6nm was often observed at the Ni<sub>2</sub>AlHf/NiAl interface, which made its presence unmistakable and unique.

Dislocation segments lying along the  $\langle 110 \rangle_{\text{NiAl}}$  directions were often unstable and found to sharply bend along the  $\langle 100 \rangle_{\text{NiAl}}$  directions, giving them a zigzag appearance (arrows in Fig 1(a)). Preferred nucleation of the Ni<sub>2</sub>AlTi precipitates on such segments produced a saw-tooth appearance. In several regions of the foil, a dense precipitation of large Ni<sub>2</sub>AlTi plates and some Ni<sub>2</sub>AlHf

precipitates were observed along the  $\langle 110 \rangle$  directions in a  $\langle 001 \rangle_{\text{NiAl}}$  zone-axis (Fig 2). This is believed to be due to preferential nucleation of the  $\text{Ni}_2\text{AlTi}$  precipitates possibly on prismatic dislocation loops.

A high density of fine precipitation as seen in Fig. 1(a) is expected to enhance the creep properties of NiAl single crystals significantly and is under investigation.

#### References

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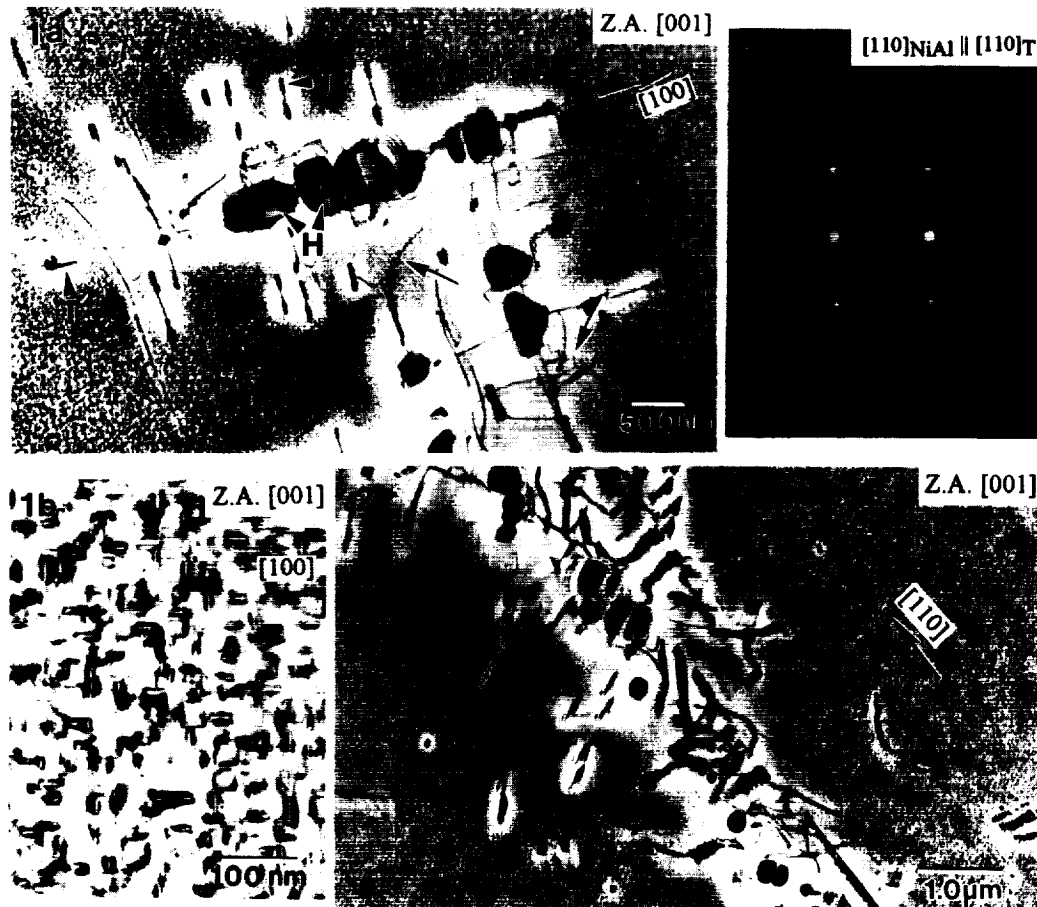


Fig. 1 (a) General microstructure of the NiAl-3Ti-0.5Hf single-crystal alloy in the as-heat treated condition. A higher magnification view of region F and its SADP are shown in (b,c), respectively.

Fig. 2 Preferential precipitation of large  $\text{Ni}_2\text{AlTi}$  plates and equiaxed  $\text{Ni}_2\text{AlHf}$  phase along a  $\langle 110 \rangle$  direction in a  $\langle 001 \rangle_{\text{NiAl}}$  zone-axis.