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FeCrNiAl alloy intended for using as blade material fabricated by rapid solidification method

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We explored fabrication of ribbons of the FeCrNiAl alloy by rapid solidification method and succeeded in producing ribbons of 0.6 mm width and 30 μm thickness. The obtained ribbon-shape product is prospective for usage as blade material. The as-solidified product, however, is too hard and brittle for the succeeding punching operation to be applied for a final-product shape. This was found to be due to existence of extremely fine particles of NiAl based B2 phase uniformly dispersed in the matrix. We, therefore, searched for a heat-treatment for improving ductility and found an appropriate one. By this heat-treatment ductility was improved to such an extent that the punching operation was applicable. We then searched for a heat-treatment for producing Al_2O_3 on the surface which facilitates a hard and sharp blade-edge, and found an appropriate one. This heat-treatment simultaneously hardens the matrix once softened to an extent sufficient for supporting the Al_2O_3 layers. The final product is suitable for applications as blades, such as razors.

1. Introduction

When heated to a high temperature, FeCrNiAl alloys [1, 2] as well as FeCrAl alloys [3–7] form an oxidized surface layer consisting chiefly of Al_2O_3. The oxidized layer has good heat and corrosion resistance and high hardness. Possible applications of these alloys are as blade materials.

However, cold-rolling of FeCrNiAl alloys is extremely hard on account of their low ductility and high hardness resulting from high density of dispersed particles of NiAl based B2-structure intermetallic compound (hereinafter B2 phase particles) in the base material. Thus thickness of the rolled sheets cannot be reduced below 2 mm.

We explored fabrication of ribbons of a FeCrNiAl alloy by single-roll rapid solidification and application of the obtained ribbons as blade material. We obtained the ribbons successfully. However, the as-solidified ribbons were too brittle to be fabricated into a desired product-shape by punching method. Therefore, we searched for a heat-treatment suitable for improving ductility of the ribbon and also for a heat-treatment to form Al_2O_3 on the ribbon surface after the punching operation and simultaneously harden the matrix again. The present paper is to report on the properties of the ribbons and heat-treatments.

2. Experimental method

2.1. Sample preparation

Fe (99.9 mass%), Cr (99.8 mass%) and Ni (99.9 mass%), all electrolytic, and FeAl alloy (Fe : Al = 50 : 50 in mass%; purity: 99.7 mass%) were measured to specified amounts and melted in an Al_2O_3 crusible using a high-frequency vacuum furnace, and then cast into a Cu mold. The atmosphere during melting was Ar gas of 65 kPa. The resulting ingots were analyzed for chemical composition with an induction coupled plasma (ICP) quantometer. Alloys were then placed in a nozzle with a 0.4 mm diameter exit hole at its tip and melted in high frequency furnace under Ar atmosphere. The molten alloy was then expelled with a 70 kPa Ar gas onto a Cu wheel rotating at 2000 rpm to obtain ribbons. The molten metal temperature ranged from 1803 to 1833 K, and the distance between the nozzle tip and wheel surface was 5 mm.

2.2. Observation of the structure

Phases in the ribbon were identified using X-ray diffractometry. Microstructure of the ribbons after rapid solidification and heat-treatment was examined by a scanning electron microscope (SEM). The morphology of B2 phase particles were examined using a

transmission electron microscope (TEM, acceleration voltage: 200 kv).

2.3. Hardness measurement

Ribbons were embedded in resin and their selectioned surfaces polished, and hardness measured with an ultra-micro Vickers-hardness tester under a load of 5 g applied for 15 s.

3. Results

3.1. Shape and mechanical characteristics of as-solidified ribbon

Ribbons obtained by rapid solidification were 30 μm in thickness and 0.6 mm in width as shown in Fig. 1. The surface was smooth; the surface roughness was 1.5 μm. The as-solidified ribbons were so brittle that punching operation was not applicable. The Vickers-hardness was 6.6 GPa, which was larger than that obtained by the conventional cast-and-roll method (5.4 GPa). The elongation obtained by tensile test was 1.2%.

3.2. Chemical composition and structure of as-solidified ribbon

Table I presents the obtained values of elements in the FeCrNiAl alloy before and after rapid solidification. Fig. 2 shows the X-ray diffraction pattern of an as-solidified ribbon. In addition to the peaks of the α solid solution phase with a body centered cubic (b.c.c.) structure, peaks of a B2 phase are present. Fig. 3 is a representative, bright-field TEM micrograph, which shows grains of uniform size of 2 to 3 μm. Fig. 4 shows further detail of the fine scale structure of the grains. Fig. 4a is a dark-field TEM micrograph of the B2 phase particles

TABLE I Alloy composition before and after rapid solidification (analyzed by induction coupled plasma method)

	Cr	Ni	Al	Zr	Fe
Before rapid solidification	34.5	21.4	6.38	0.31	Bal
After rapid solidification	34.3	21.5	6.45	0.30	Bal

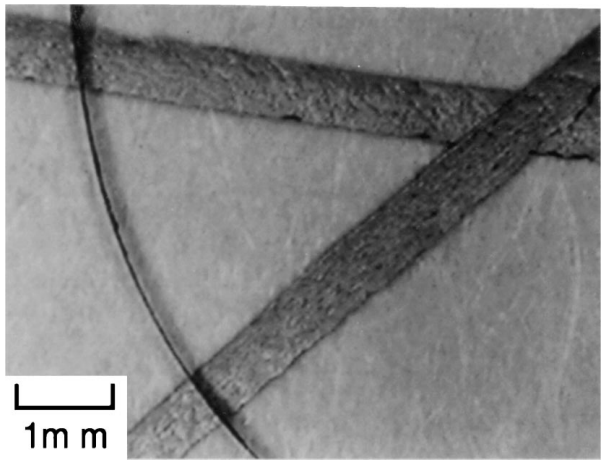

Figure 1 FeCrNiAl alloy ribbon produced by rapid solidification.

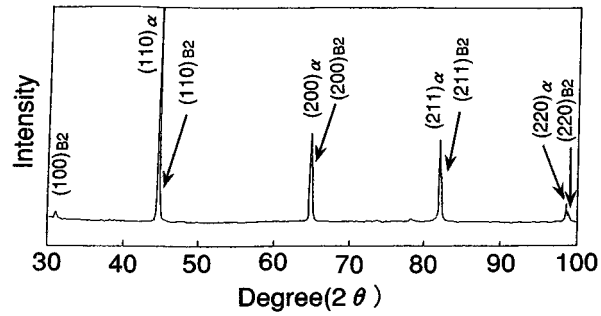

Figure 2 X-ray diffraction pattern of the rapidly solidified FeCrNiAl alloy.

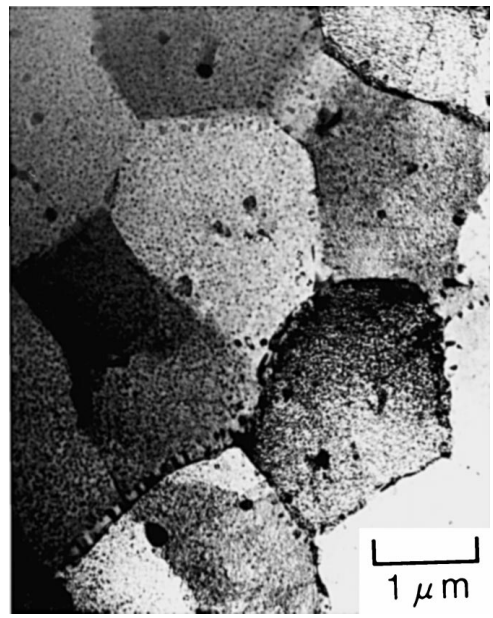

Figure 3 Electron micrograph of FeCrNiAl alloy ribbon made by rapid solidification (bright-field image).

obtained using the (100) super-lattice reflection from the selected area diffraction pattern shown in Fig. 4b. The image indicates that B2 phase particles of 30 nm are distributed uniformly.

3.3. Control of the matrix hardness by means of heat-treatment

For the rapidly solidified FeCrNiAl ribbon to be used as blade material it is necessary that the matrix is ductile enough for punching operation to be applicable for obtaining desired final blade shape. It is also necessary that after the punching, Al_2O_3 layer of a sufficient thickness is formed on the surface and the matrix is simultaneously hardened to an extent sufficient for supporting the Al_2O_3 layer. Therefore, we searched for suitable heat-treatments for these requirements.

In order to decide the heat-treatment condition that enables the punching operation, we first examined the relation between heat-treatment temperature and hardness. Fig. 5 shows Vickers-hardness of cross-section of a ribbon plotted against heat-treatment temperature. Specimens were held at the temperatures for 1.8 ks after rapid solidification. Vickers-hardness of as-solidified sample is 6.4 GPa and its hardness after heat-treatment at 873 K is 5.4 GPa. It is found that Vickers-hardness

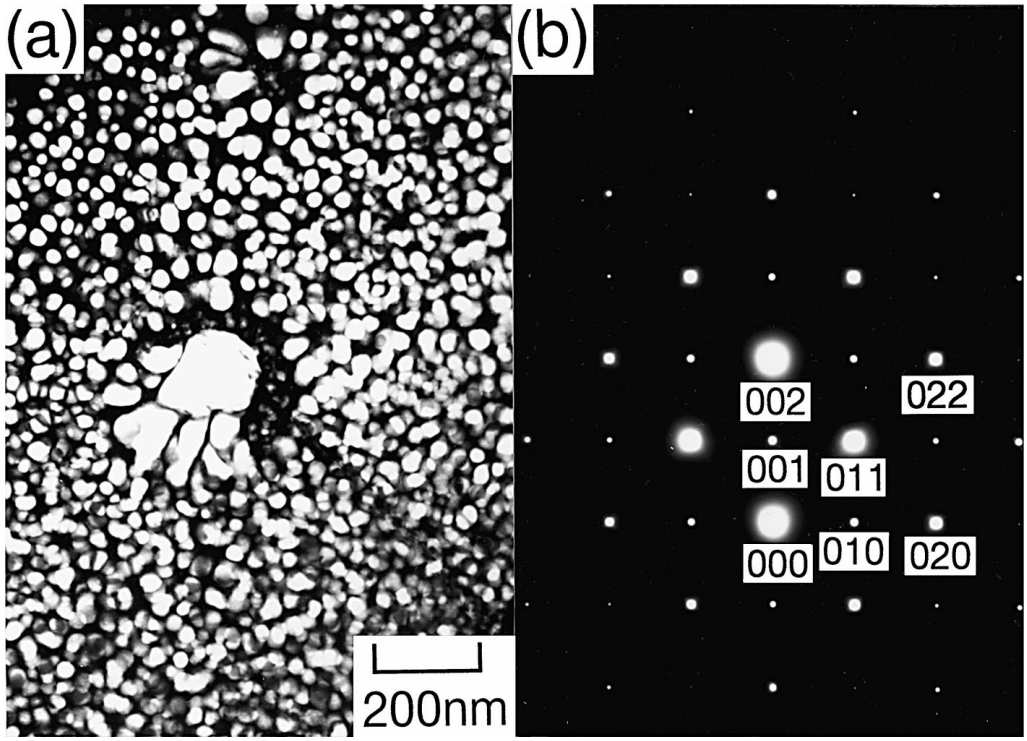

Figure 4 Electron micrograph and diffraction pattern of as-solidified FeCrNiAl alloy ribbon (a) Dark-field image by 001 reflection (b) Diffraction pattern.

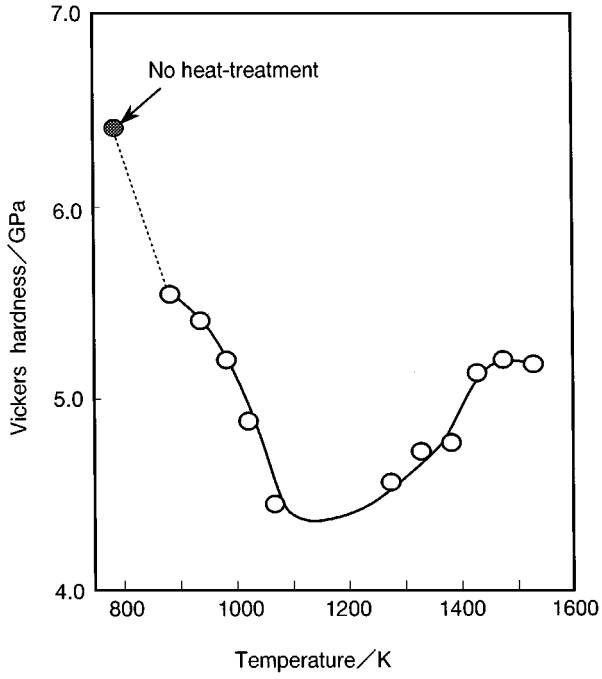

Figure 5 Vickers-hardness of the cross-section of FeCrNiAl alloy ribbon plotted against heat-treatment temperature. Specimens were held at each temperature for 1.8 ks after rapid solidification.

decreases to 4.4 GPa as the heat-treatment temperature is increased from 873 to 1073 K. However, for heat-treatment temperatures higher than this, hardness increases again. This phenomenon should be related to the dispersion state of B2 phase particles in the matrix. Therefore, we made TEM observation for examining the size and distribution state of the fine particles closer. Fig. 6a–d are TEM micrographs of the specimens heat-treated at temperatures between 873 and 1273 K. In the specimen heat-treated at 873 K (Fig. 6a), the particle size does not change from that of the as-solidified specimen (Fig. 4a). In the specimen heat-treated at 1073 K (Fig. 6b) particles of 60 nm size appear. This size is two times larger than that of the particles in the specimens heat-treated at 873 K. Such particle growth appears to cause the decrease in hardness with the increase of heat-treatment temperature from 873 to 1073 K as shown in Fig. 5. In the specimen heat-treated at 1373 K (Fig. 6c), the largest size of the particle is 400 nm in diameter. Other two average sizes are 20 and 100 nm. In the specimen heat-treated at 1473 K (Fig. 6d), the particles of the largest size seen in the specimen heat-treated at 1373 K (Fig. 6c) are not seen and only much smaller particles of 100 nm size exist. We believe that the particles were absorbed in the matrix when held at 1473 K and the particles of the 100 nm seen in Fig. 6d have separated again from the matrix during cooling. In the specimen heat-treated at 1073 K, the particles smaller than 60 nm do not exist in the matrix and it's Vickers-hardness is smaller than 4.5 GPa. The above observation indicates that, for reducing hardness, heat-treatment at a temperature at which particles grow fast but they do not dissolve in the matrix, for example at 1073 K, is effective. However, the hardness of 4.5 GPa is still not low enough for the punching operation to be applicable. Therefore we explored a heat-treatment for reducing hardness further and found an effective one.

The explored heat-treatment is as follows: An as-solidified ribbon is heat-treated at 1373 K for 0.5 ks, cooled slowly to 973 K with a period of 3.6 ks and air-cooled from 973 to 300 K at a rate of 100 K/s. The Vickers-hardness after this heat-treatment was 3.4 GPa, which is less than that of the specimen heat-treated at 1073 K for 1.8 ks (4.3 GPa as shown in Fig. 5). By

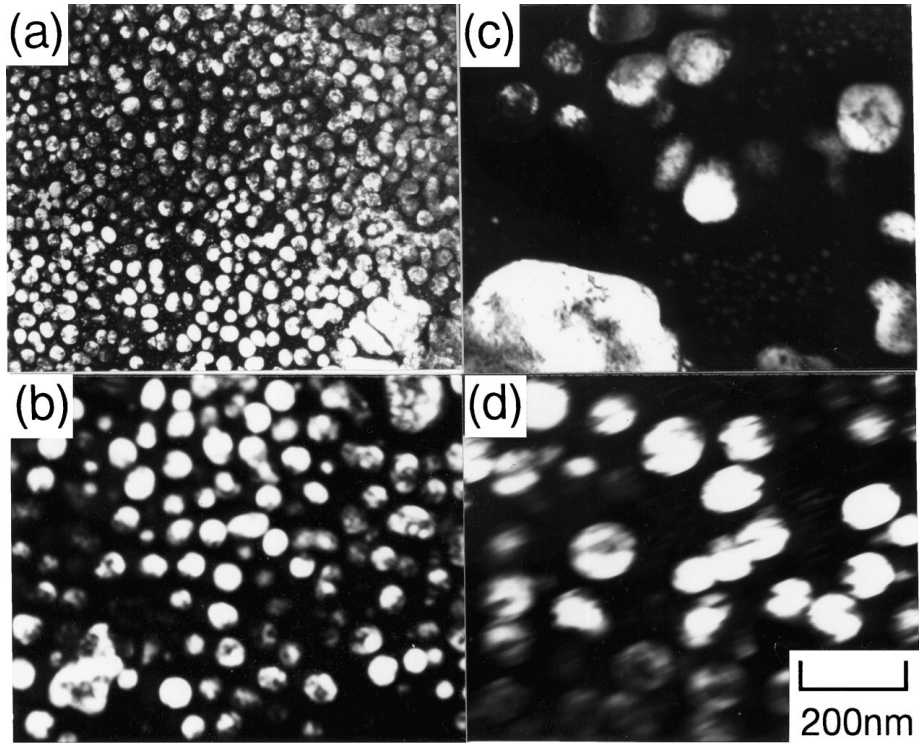

Figure 6 Change of microstructure due to heat-treatment (Dark field TEM images by order-reflections). Specimens were held for 1.8 ks at each temperature after rapid-solidification. (a) 873 K, (b) 1073 K, (c) 1373 K, (d) 1473 K.

tensile testing the softened ribbon, its elongation was found to be 6%, which is very much improved compared to 1.2% of the as-solidified state. The softened ribbon was successfully fabricated into desired shapes by punching operation.

In order to see the microstructure of the softened state, TEM observation was done and the obtained micrograph is shown in Fig. 7. It is seen that the average particle size is 500 nm and no smaller particles exist in the matrix. The particle size, 500 nm, is about ten times larger than that of particles formed by heat-treatment at 1073 K for 1.8 ks (seen in Fig. 6b). The softening appears to be due to this large particle size and the absence of the fine particles in the matrix.

After punching operation, Al_2O_3 layer for blade-edge have to be formed on the surfaces. In order to find a suitable heat-treatment condition for this purpose, we examined the heat-treatment dependence of the Al_2O_3 thickness. We believe that the relation between the thickness of the Al_2O_3 layer and heat-treatment period follows the parabola-law that has been confirmed for a FeCrAl [8]. Thus, we examined the relation between Al_2O_3 thickness and heat-treatment temperature. Fig. 8 shows SEM micrographs of ribbons after heat-treatment at several temperatures between 1073 and 1473 K for 11.8 ks. Black layers of the both surfaces of each ribbon are Al_2O_3 layers. They do not form at 1073 K. Al_2O_3 layers are 1.9 μm thick form at 1273 K, 3.0 μm thick with 1373 K and 3.7 μm thick with 1473 K. The higher the heat-treatment temperature, the thicker is the Al_2O_3 layer. The root-like Al_2O_3 growing from the Al_2O_3 layer into the matrix appear to be the reason for the good adhesion of the Al_2O_3 layers [9].

We then explored a heat-treatment for increasing the hardness that was once reduced for the punching operation to be applicable, to an extent that can hold the surface Al_2O_3 formed. For increasing the hardness of the alloy, it is necessary to dissolve the B2 phase particles in the matrix by heating above 1473 K (see Fig. 6d) and to have fine particles be dispersed during cooling. We tried the following heat-treatment on the once softened specimen. A softened specimen was held at 1523 K for 3.6 ks and the cooled to room temperature at a rate of

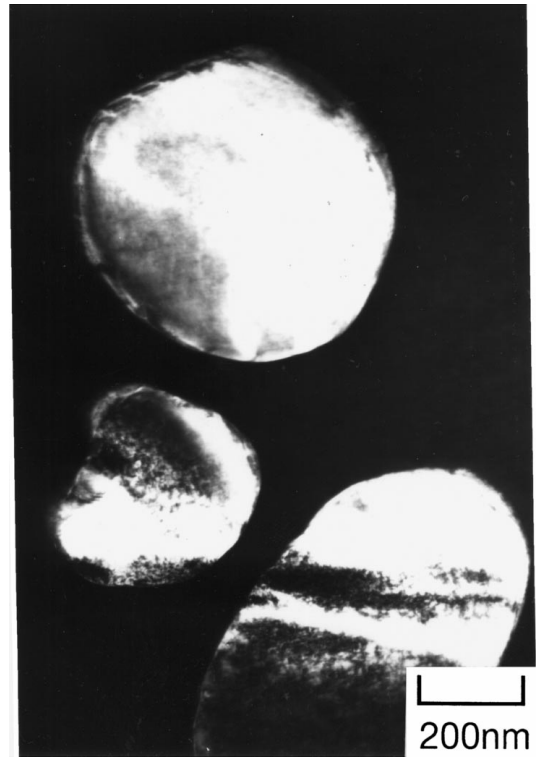

Figure 7 Micro-structure of the ribbon held at 1373 K for 0.5 ks, followed by cooling to 973 K, in 3.6 ks and air-cooling from 973 to 300 K with a rate of 100 K/s (Dark-field electron micrograph).

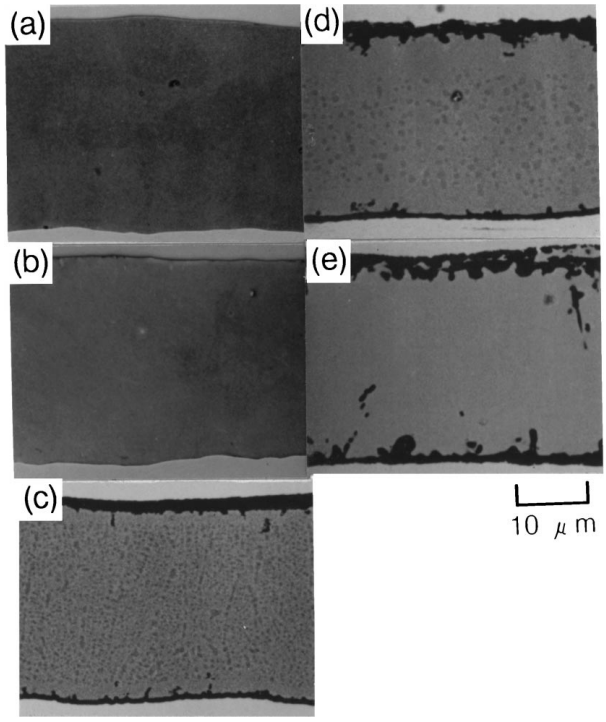

Figure 8 Change of microstructure of the FeCrNiAl alloy ribbon due to heat-treatment (SEM image of the ribbon). Specimens were held for 11.8 ks at each temperature after rapid-solidification. (a) Before heat-treatment, (b) 1073 K, (c) 1273 K, (d) 1373 K, (e) 1473 K.

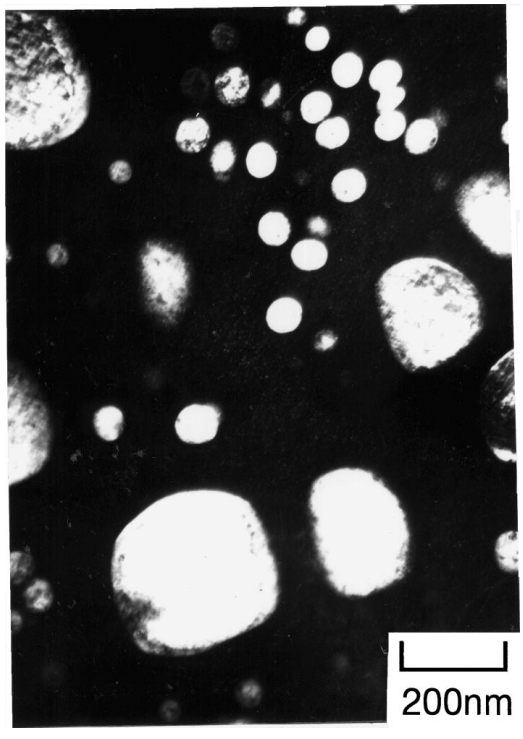

Figure 9 Micro-structure of a specimen held at 1523 K, for 3.6 ks and cooled to room-temperature with the rate of 100 K/s.

100 K/s. The obtained hardness was 5.1 GPa. In order to make clear the reason for this hardness increase, we did an TEM observation. The obtained micrograph is shown in Fig. 9. It is seen that particles of 250 nm size are dispersed and also that the matrix is dispersed with many smaller particles of 50 nm size. We believe that these 50 nm size particles are responsible for the increased hardness.

For using the ribbons as blade material, it is required that both the Al_2O_3 formation on the surface and the improvement of the matrix hardness for supporting the Al_2O_3 are achieved simultaneously by a heat-treatment. For formation of Al_2O_3 layer thicker than 2 μm, holding at a temperature above 1473 K for longer than 1.8 ks is needed. For improving the matrix hardness, holding at a temperature above 1473 K is needed to dissolve once the B2 phase particles in the matrix. Thus, as an heat-treatment that satisfies these two conditions, we tried 1523 K, 3.6 ks holding followed by 100 K/s cooling of a softened specimen. The obtained thickness of the surface Al_2O_3 was 2 μm and the matrix hardness was 5 GPa, both of which satisfy the requirement and proves this heat-treatment is appropriate.

4. Conclusion

1. Using single-roller rapid solidification method, we succeeded in producing thin Fe-34.3Cr-21.5Ni-6.5Al alloy ribbon (width 0.6 mm thickness 30 μm). In the α phase grains of the as-solidified ribbon, fine NiAl based B2 phase particles of diameters 30 nm are dispersed.

2. The as-solidified ribbons are extremely hard and brittle and, therefore, punching operation for fabrication of the product-shape is impossible.

3. By performing an appropriate heat-treatment, the hardness can be reduced and the ductility can be improved to such an extent that punching operation is applicable.

4. By an appropriate heat-treatment after punching operation for a product-shape, Al_2O_3 layer of a sufficient thickness can be formed on the surface and simultaneously the matrix can be hardened to an extent sufficient for supporting the Al_2O_3 layers.

5. Mechanical properties, such as hardness and ductility, can be controlled by heat-treatment.

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