Softening by Coarsening of Ni-Al B2 Phase Particles in Fe-Cr-Ni-Al-Zr Alloy

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In Fe-Cr-Ni-Al stainless steels, surface alumina layer with good adhesion is formed by heat treatment at a high temperature in oxidizing atmosphere and can prevent burrs from being formed in grinding of cutting edges. On the other hand, as the precipitation velocity of Ni-Al B2 phase particles dispersed in the matrix of these steels is remarkably high, it is hard to obtain a full solid-solution condition without any fine precipitates by conventional solution treatment. And, the dispersed particles increase the hardness of the material and it is impossible to decrease the hardness sufficiently to form the cutting blade only by the annealing after cold rolling. According to the process developed in this study, most of the B2 phase particles are dissolved once, cooled slowly and, thus, combined with remaining particles to form coarse particles. As a result, precipitation of fine particles is prevented and softening necessary to form into given shapes is achieved.

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1. Introduction

The blades of electric shaver, especially the inner blades, are usually made of martensitic stainless steels.1–4) The manufacturing process for these blades is shown in Fig. 1(a). Namely, raw materials weighed in the ratio of given composition are melted, cast into molds, and hot and cold rolled into steel strips. The strips are press-formed into given shapes.5) Before press-forming, the strips are softened by annealing to prevent the fracture of the material or the wear of die in forming. The materials formed into given shapes are subjected to quenching to give sufficient properties as the materials for blades.6) In the case of blades for cutting something by snipping such as the blades of electric shaver, the press-formed products are formed into the shape of actual blade blocks by resin-molding and the blade edges are produced by grinding so as to insure smooth sliding surface. Thus, final products are completed.

During the grinding process for producing blade edges, usual martensitic stainless steels are deformed plastically and grinding burrs are produced. As a result, cutting quality is deteriorated. In order to solve the problem, by forming a hard layer with good adhesion on the surface different from the surface to be ground, the grinding burrs due to plastic deformation are cut off, resulting in good cutting edges.7–12) One of the means to achieve this is to add Al to Fe-Cr type stainless steel and to heat in an oxidizing atmosphere such as in the air to form a stable alumina layer on the surface.13–21) The surface alumina layer has a remarkable effect to control the formation of burrs at the blade edges. As this material is a usual ferrite type stainless steel, it has a very low hardness. Now, in the case of the materials to be applied to the blades of electric shaver, it is necessary to control the hardness of the materials as low as possible because the materials are subjected to plastic deformation such as punching. However, it is also necessary to keep a high hardness in the final products to control the wear of the materials due to sliding.

The methods to strengthen metallic materials include solid solution strengthening, utilization of transformation, work hardening, grain size refinement, dispersion strengthening, and precipitation strengthening.22,23) In the case of usual Fe-Cr type stainless steels, it is a common method for strengthening to add C, quench and, thus, strengthen the materials by martensitic transformation. However, as C prevents the formation of surface alumina layer in the alloy used in this study, it is not possible to use the above method.
In addition, because the heat treatment to form surface alumina requires the material to be held at a temperature higher than 1073 K for a given time, it may be impossible to keep the strength obtained by work hardening and to make the grains fine. It may also be impossible to utilize the effect of dispersion strengthening because the material will be subjected to melting for mass production.

On the other hand, it has been reported that when Ni is added to Fe-Cr-Al alloy in such an amount that ferritic state of the matrix can be kept by a suitable balance between the amount of Cr + Al and the amount of Ni, a hardness higher than that of Fe-Cr-Al can be obtained. It has also been reported that in the matrix structure of this alloy, Ni-Al phase particles are dispersed. This suggests a possibility that an increased hardness due to precipitation strengthening can be obtained in Fe-Cr-Ni-Al stainless steel.

Figure 1(a), as described before, shows a heat and working pattern before being subjected to the process for producing blade edges for usual martensitic blade steels. The steels are hot-worked and cold rolled into a given thickness. Then, the steels are subjected to annealing at 973 K as a softening treatment for press working to form blade shape and to quenching treatment by air cooling from 1323 K for ensuring the hardness necessary for blades. On the other hand, Fig. 1(b) shows a heat and working pattern for the steel of this study. This steel is hot-worked and cold rolled into a given thickness in the same way as usual blade steels. Then, this steel is subjected to a heat treatment for softening the Vickers hardness down to below 3.5 GPa and to a precipitation treatment for forming alumina layer on the surface and, at the same time, for ensuring the hardness higher than 5 GPa necessary for blades. The hardness of 3.5 GPa is the limiting and required hardness that makes press working possible, which is the target of this study.

Figure 2 shows the hardness change of this steel held for 1.8 ks at various temperatures and air-cooled. It is found from Fig. 2 that the as-cold rolled hardness of this steel is the hardness of 4.2 GPa and this steel can not be sufficiently softened by the holding for 1.8 ks at 623–1523 K and then air-cooling. Rather the hardness of this steel increased by holding in the range of 723–823 K and over 1173 K. The hardening caused by holding this steel in the range of 1173 K is due to the precipitation of the fine Ni-Al B$_2$ phase particles during the air-cooling. And the hardening caused by holding this steel in the range of 723–823 K is due to the formation of $\sigma$-phase as observed often in Fe-Cr family steels, which is the origin of $\sigma$-brittleness. In the softening heat treatment in Fig. 1(b), this steel is held at 1423 K, cooled down to 973 K at a controlled cooling rate and then air-cooled. The reason why the starting temperature of the air cooling was set at 973 K is to avoid $\sigma$-brittleness, that is the formation of $\sigma$-phase.

In this study, it was investigated to control the morphology of Ni-Al B$_2$ phase particles in cooling process to 973 K after holding at 1423 K by decreasing the cooling rate and, as a result, to soften Fe-Cr-Ni-Al stainless steel.

2. Method of Experiment

2.1 Preparation of specimens

Given amounts of electrolytic iron, electrolytic chromium, electrolytic nickel, Fe-Al base alloy, and Fe-Zr base alloy were weighed, melted in an alumina crucible in a high-frequency vacuum melting furnace and cast into a copper mould to produce an ingot. The compounding ratios were Cr: 33.5 mass%, Ni: 21.0 mass%, Al: 6.7 mass%, Zr: 0.3 mass%, and Fe: balance.

The ingot was hot rolled and cold rolled to a thickness of 0.12 mm. The cold rolled sheet was cleaned in acetone and used for specimens.

2.2 Heat treatment

A box furnace was used for heat treatment. Specimens were put in alumina boats placed in the furnace which was kept at a given temperature in the air and taken out together with alumina boats and left on an alumina board (air cooling, cooling rate $\approx$ 100 K/s) or thrown into water (water cooling, cooling rate $\geq$ 150 K/s). Cooling rate was changed by programming the operation of the temperature regulator of the box furnace.

2.3 Measurement and microstructural observation

The specimens were embedded in resin, polished and burnished, and subjected to hardness measurement with a micro-Vickers hardness tester (Mitsutoyo HM-124) under a load of 300 g. Microscopic observation with an optical microscope was made for specimens prepared in the same way as the above. Scanning electron microscope observation was made for specimens that were embedded in resin, polished and burnished, and etched with nitrohydrochloric acid. Transmission electron microscope observation was made for specimens that were polished from the surface and etched.

3. Results and Discussion

Figure 3 and Figure 4 show a typical microstructure and the change in hardness, respectively, of the specimens that were held at 1423 K and cooled down to 973 K with various cooling rate. At the holding temperature, 1423 K, alumina layer is possible to be formed on the surface. Below 973 K, the specimens were air-cooled to avoid $\sigma$-brittleness observed in Fig. 2. From the optical microstructures in the cross sections in rolling direction in Fig. 3, it can be seen that Ni-
Al B$_2$ phase particles become coarser and more spheroidized and the ratio of the coarse particles is increased with the decrease in the cooling rate. This is presumably because the low cooling rate at high temperature range has an effect similar to the effect of high temperature holding to spheroidize coarse particles and, at the same time, Ni-Al B$_2$ phase particles that have resolved once at the holding temperature of 1423 K can precipitate finely on the spot only by a very small fraction because of the slow cooling rate but precipitate mainly at the coarse particles selected as precipitation sites. From Fig. 4, it is found that the hardness is decreased with the decrease in the cooling rate. This is presumably because, when the cooling rate is slow, Ni-Al B$_2$ phase particles that have resolved once at the holding temperature of 1423 K select existing particles as precipitation sites or precipitate newly only at limited precipitation sites and, as a result, grow into coarse particles.

To verify this, the specimens were held at 1423 K for 5.4 ks, cooled down to 973 K at a cooling rate of 0.022 K/s and then air-cooled. The microstructures during the softening treatment were observed in detail. Figure 5(a) shows the scanning electron microscopy image for the initial stage (as cold rolled), (b) for the stage as water cooled after holding at 1423 K for 5.4 ks, and (c) for the stage as air-cooled from 973 K. Figure 6(a) shows the dark field image of transmission electron microscopy for the stage as water cooled after holding at 1423 K for 5.4 ks and (b) for the stage as air-cooled from 973 K. From Fig. 6, the decrease in the aspect ratio of B$_2$ phase particles larger than 1 μm is recognized. From Fig. 5, the decrease in the number of particles smaller than 1 μm existing between coarse particles is recognized. From Fig. 6, it is found that the fine particles recognized in Fig. 5 has grown up by slow cooling and the number of fine particles smaller than 50 nm has been decreased.

As a result, it can be said that the decrease in the number of fine particles smaller than 50 nm has decreased the contribution of precipitation hardening and has lowered the hardness.

4. Conclusion

It was investigated to softening of Fe-Cr-Ni-Al stainless steel through the control the morphology of Ni-Al B$_2$ phase particles by cooling rate to 973 K from 1423 K and the following results were obtained:

As the material is subjected to press-forming at a stage in the course of manufacturing blades, it is desirable that the hardness of this material at this stage is decreased as low as
The temperature range below 973 K does not contribute to the cooling rate equal to or higher than air-cooling below 973 K. This is necessary to avoid brittleness by cooling the material at a temperature higher than 973 K. If the material is subjected to an annealing at a temperature between 873 and 1073 K that is usually used for cold worked ferrous materials, the hardness can not be decreased sufficiently because only the increase in the hardness due to work hardening is removed by this type of annealing. A heat treatment is required by which fine Ni-Al B$_2$ phase particles to form coarser particles. In this regard, it is necessary to avoid $\sigma$ brittleness by cooling the material at a cooling rate equal to or higher than air-cooling below 973 K. The temperature range below 973 K does not contribute to the coarsening of B$_2$ phase particles. A hardness of Hv $\leq$ 3.5 GPa was obtained by this heat treatment.

Figure 7(a) shows the cross section of the material that has been subjected to the optimum softening heat treatment developed by this study and to the press forming to shape sharply-angled cutting blade of the inner blade of electric shaver. No cracks are observed. Figure 7(b) shows the cross section of the material that has been subjected to the usual annealing treatment and to the press forming. Many cracks are observed in this case. The actual softening heat treatment is practiced in vacuum so that alumina is not formed on the surface.

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