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# Texture memory effect in heavily cold-rolled Ni<sub>3</sub>Al single crystals

# Masahiko Demura<sup>a</sup>, Ya Xu<sup>b</sup> and Toshiyuki Hirano<sup>c</sup>

National Institute for Materials Science, 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047, *Japan* <sup>a</sup>DEMURA.Masahiko@nims.go.jp, <sup>b</sup>XU.Ya@nims.go.jp, <sup>c</sup>HIRANO.Toshiyuki@nims.go.jp

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**Abstract.** The paper presented the texture evolution during primary recrystallization and following grain growth in the heavily cold-rolled Ni<sub>3</sub>Al single crystals. It turned out that the texture evolution occurred in the two stages. First, primary recrystallization caused the drastic change of the as-rolled texture. Then, as grain growth proceeded, the texture returned to the same one as the as-rolled textures. This texture return can be designated as *Texture memory effect*. The mechanism of the texture memory effect was discussed based on the analysis of the orientation relationship between the as-rolled and the primary recrystallization textures.

#### Introduction

We have developed Ni<sub>3</sub>Al foils for high-temperature micro chemical devices [1-6]. For the application there are serious concerns about the brittleness in the foils, especially in the recrystallized state, since the polycrystalline Ni<sub>3</sub>Al is generally brittle due to the grain boundary (GB) fracture. However, our recent study [7] revealed that the ductility can be improved by heat treatment extending to grain growth. The ductility improvement is ascribed to the high fraction of the low angle boundaries (LABs) and  $\Sigma$  3 boundaries which are known crack-resistant [8].

In this heat treatment we found an interesting phenomenon on the texture evolution as follows [9]. The as-rolled foils have strong {110} textures. The {110} textures change into complicated ones consisting of several components by primary recrystallization. In the subsequent grain growth, the textures completely return to the same {110} textures as the as-rolled ones, which can be designated as *Texture memory effect*. Such texture return has rarely been observed in Ni<sub>3</sub>Al as well as in other common metals. In this paper, we present the texture evolution during the primary recrystallization and grain growth in the heavily cold-rolled Ni<sub>3</sub>Al single crystals.

# **Experimental procedures**

The specimens were prepared by the cold rolling of the single-crystalline sheets grown by the investment casting method. The cold rolling was performed without intermediated annealing. The

detail of the specimen preparation were given in our previous report [2]. The as-rolled texture, reduction, and thickness of the specimens used in this study were listed in Table 1. The specimens were heat-treated at temperatures of 873~1573K for 0.5h in an argon flow gas. The textures of the cold-rolled and the recrystallized specimens were measured by the X-ray Schultz back reflection method. The orientation maps of the recrystallized specimens were measured by the electron backscatter diffraction (EBSD) method.

**Table 1.** Specimens used in this study.

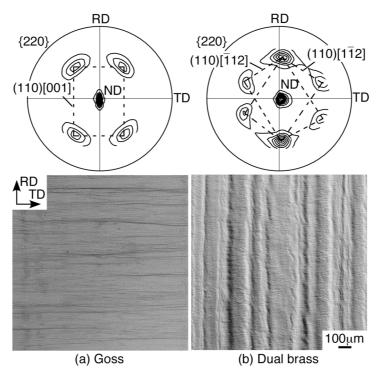
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As-rolled Texture	Reduction (%)	Thickness (µm)
Goss	84	366
Goss	95	84
Dual Brass	95	113

#### Results

As-rolled textures and microstructures. Figure 1 shows the as-rolled textures and microstructures. We here prepared the foils with two different as-rolled textures (Table 1). One is assigned as the Goss texture, where the normal direction (ND) of the foils are parallel to (110) and the rolling direction (RD) to [001], shown by the {220} pole figure in Fig. 1 (a). The Goss-textured specimens were prepared by the cold rolling of the Goss-oriented single crystals where no lattice rotation virtually occurred during the cold rolling. The specimens had a relatively homogeneous microstructure and no clear deformation bands were observed, which corresponds to the simple, Goss texture. These characteristics of the as-rolled textures and microstructures were essentially the same between the 84% and 95% cold-rolled foils.

The other as-rolled texture consists of the two symmetrical  $\{110\}$  textures, i.e.  $(110)[\overline{1}12]$  and  $(110)[\overline{1}12]$ , as shown by Fig. 1 (b). This can be called as the dual brass texture. The starting ingot was a single crystal with  $\sim$ (210)[001] and the cold rolling caused the lattice rotation with the texture splitting, resulting in the formation of the dual brass texture. The detailed mechanism of the dual brass texture has been presented in our previous paper [10]. The as-rolled microstructure consists of bands elongated along the RD, the width of which is  $\sim$ 100 µm (Fig. 1 (b)). Our previous TEM observation revealed that each band had a single brass texture [11]. Thus, two kinds of bands, (110)[-112] and (110)[1-12], which were alternately located in the transverse direction, formed the dual Brass textures in total.

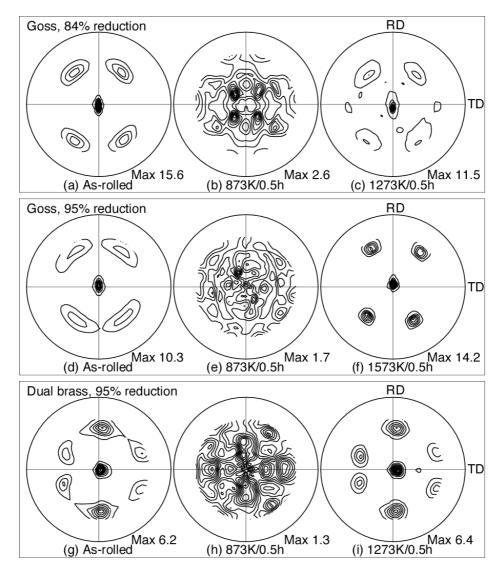


**Figure 1.** The textures and microstructures in the cold-rolled Ni<sub>3</sub>Al single crystals: (a) Goss specimen with 84% reduction and (b) dual Brass specimen with 95% reduction.

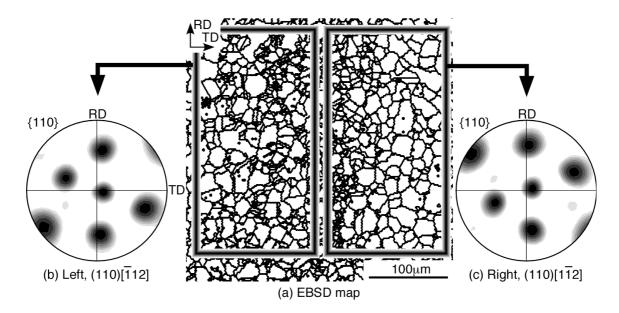
**Recrystallization microstructures.** In all the specimens, the primary recrystallization completed at 873 K/0.5 h, which was confirmed by the SEM observation [7,9]. The recrystallization microstructures consisted of equiaxed grains and a large number of the annealing twins. The grain growth occurred by the heat treatment at higher temperatures. For example, in the Goss specimens with the 84% reduction, the average grain size increased from 0.5  $\mu$ m at 873/0.5 h to 70  $\mu$ m at 1573 K/0.5 h. There was no significant difference in the recrystallization microstructures between the Goss and the dual Brass foils.

**Texture evolution during the recrystallization and grain growth.** Figure 2 (a-c) shows the texture evolution by the recrystallization and grain growth in the Goss specimen with 84% reduction. The texture changed drastically by the primary recrystallization, as shown by Fig. 2 (b). The primary recrystallization texture consisted of several components which are different from the as-rolled, Goss texture. The texture became weak, as shown by the decrease in the maximum intensity of the {220} poles. However, as the grain growth proceeded, the primary recrystallization texture disappeared gradually and the Goss texture appeared instead (Fig. 2 (c)). This is the texture memory effect in the Goss specimen as we previously reported [9]. Similar texture evolution was obtained in the 95% cold-rolled foils (Fig. 2 (d-f)), showing no significant effect of the cold reduction level in this range.

Fig. 2 (g-i) shows the texture evolution in the dual brass specimen with 95% reduction. Also in this case, the primary recrystallization caused the texture changes into complicated one. Note that it returned to the dual Brass texture by the subsequent grain growth. This result means that the texture memory effect occurs in the dual Brass texture as well as the Goss texture.



**Figure 2.** {220} X-ray pole figures showing the texture evolution during the recrystallization and grain growth in the heavily cold-rolled Ni<sub>3</sub>Al single crystals: (a-c) the Goss specimen with 84% reduction, (d-f) the Goss specimen with 95% reduction, and (g-f) the dual Brass specimen with 95% reduction.



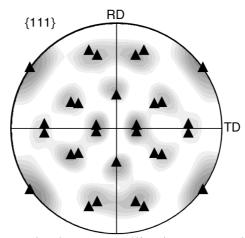
**Figure 3.** Orientation map for the dual Brass specimen heat-treated at 1273K/0.5h. (b) and (c) show the {110} pole figure obtained in the left-hand and right-hand rectangular regions surrounded by the thick lines, respectively.

Figure 3 shows the results of the EBSD measurements in the dual Brass specimen heat-treated at 1273K/0.5h, corresponding to the state in Fig. 3 (i) where the dual Brass texture had returned. It turned out that the specimen consisted of two bands, each of which had a single brass texture. For example, the left region had (110)[112] texture (Fig. 3 (b)), while the right one had (110)[112] texture (Fig. 3 (c)). This banded structure and the band width is very similar to that observed in the as-rolled microstructures. The band width is also in agreement with that of the as-rolled banded structure. This agreement means that the texture memory effect occurred individually in each band. In other words, it can be concluded that the band boundary, or region between the adjacent two bands caused no significant effect on the texture evolution during the primary recrystallization and the grain growth in each band.

# **Discussions**

The present results demonstrate that the texture memory effect occurs on both of the Goss and dual Brass specimens. That is, this effect seems not to be a phenomenon which appears in some special texture. This fact suggests that the primary recrystallization texture may have a relationship to the as-rolled texture. We thus examined the relationship between the as-rolled texture and the primary recrystallization texture in the case of the Goss specimens with 84% reduction. As a result, we found that the primary recrystallization texture had the rotation relationship about <111> with the rotation angle of 40° to the as-rolled texture. Figure 4 shows the {111} pole figure in the primary recrystallization texture together with the {111} poles of the 40°-rotated Goss about <111> shown by the triangles. It is clear that the peak positions in the primary recrystallization texture are in well agreement with those of the 40°<111>-rotated Goss orientations. Thus, it turned out that the orientation information of the as-rolled texture was literally memorized in the primary recrystallization texture by means of the 40°<111> rotation relationship. Similar relationship has been reported in the cold-rolled Ni<sub>3</sub>Al single crystal by Escher et al. [12].

Based on this analysis, we discuss the mechanism of the texture memory effect. In fcc metals, it is known that the GBs between the grains with  $40^{\circ} < 111 >$  rotation relationship exhibit high mobility [13]. Assuming the high mobility of the  $40^{\circ} < 111 >$  GBs in Ni<sub>3</sub>Al which has an fcc-based ordered structure, the texture memory effect can be explained by the preferential growth mechanism as follows. First, in the primary recrystallization stage, the grains with  $40^{\circ} < 111 >$  rotation relationship



**Figure 4.**  $\{111\}$  pole figures in the recrystallization texture in the Goss specimen. The triangles show the  $\{111\}$  poles of the  $40^{\circ}$ <111>-rotated Goss orientations.

to the as-rolled texture can grow faster than the others, resulting in the formation of the  $40^{\circ}<111>$ -rotated texture as the primary recrystallization texture. Then, in the subsequent grain growth stage, the similar preferential growth occurs on the grains with the same orientation as the as-rolled texture since these grains can form the  $40^{\circ}<111>$  GBs with the grains in the primary recrystallization texture. The details of the mechanism will be presented in elsewhere [14].

So far, there are no reports on the texture memory effect in Ni<sub>3</sub>Al though the recrystallization texture in heavily cold-rolled Ni<sub>3</sub>Al has been studied [12,15-17]. In the cold-rolled single crystals, Escher et al. [12] reported that the as-rolled texture, which is almost a single {110} texture, changes into a weak and complicated one by recrystallization, which probably corresponds to the first stage in the texture memory effect. However, they did not find the texture return by the grain growth. This is probably because their heat-treatment conditions, 1073K/1-10s, were not sufficient for grain growth to cause the texture return. We confirmed that the texture return appeared evidently after 10h at 1073K [14]. That is, the enough grain growth is necessary for the texture memory effect.

In the case of the boron-doped polycrystals [15-17], it was reported that the as-rolled textures consisted of several components including the {110} textures and they change into more complicated textures which are different from the as-rolled ones. Regarding the texture evolution by the subsequent grain growth, observed was the tendency that the {110} texture components appeared during the grain growth [17]. However, the occurrence of the texture memory effect was not evident. According to the present result on the dual Brass specimen, the texture memory effect can occur even if a specimen consists of several regions with different orientations, indicating that the texture memory effect may appear in the polycrystalline form. In the cold-rolled polycrystalline Ni<sub>3</sub>Al, however, shear bands or deformation bands developed heavily, extending across several grains [18]. Probably, such deformation heterogeneities prevent the occurrence of the texture memory effect in the polycrystalline form. On the other hands, the cold-rolled single crystals exhibited rather homogeneous deformation microstructure (Fig. 1), and showed the texture memory effect. We can thus conclude that the homogeneity in the microstructure is one of the crucial conditions for the texture memory effect.

One may have the question whether the texture memory effect occurs only on Ni<sub>3</sub>Al or not. In the above discussions, we did not base on the characteristics that are unique to Ni<sub>3</sub>Al, e.g. its ordered structure. According to the preferential growth mechanism based on the assumption of the high mobility of the 40°<111> GBs, the texture memory effect can occur in the fcc metals similarly. In fact, Gottstein [19] mentioned a similar texture evolution in Cu-30%Zn alloys very briefly, but no detailed information was given there. To my knowledge, there are no other reports on the texture memory effect in the literature. The further studies are necessary to determine the more exact conditions for the texture memory effect.

### **Conclusion**

The texture evolution during the recrystallization and grain growth was examined in the cold-rolled  $Ni_3Al$  single crystals with the different as-rolled textures, the Goss and the dual Brass textures. The texture memory effect occurred in both the textures. The orientation analysis revealed that the recrystallization texture had a  $40^{\circ} < 111 >$  rotation relationship to the as-rolled texture. Based on this fact, we proposed the preferential growth mechanism to explain the texture change by the primary recrystallization and the texture return by the subsequent grain growth, assuming that the  $40^{\circ} < 111 >$  GBs have high mobility.

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