IIB 2010

# Martensite/austenite interfaces in ultrafine grained Fe-Ni-C alloy

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Received: 16 August 2010/Accepted: 19 October 2010/Published online: 2 November 2010 © Springer Science+Business Media, LLC 2010

**Abstract** In this article, orientation relationships (ORs) at martensite/austenite interface (M/A interface) transformed from coarse-grained and ultrafine-grained austenites were investigated. The results showed that the OR at the M/A interfaces of lenticular martensite transformed from coarsegrained austenite was close to Kurdjumov-Sachs (K-S). The OR of butterfly martensite was close to K-S in the outer side of M/A interface and it deviated to Nishiyama-Wasserman (N–W) at the inner side of M/A interface. In contrast, the OR of lenticular martensite transformed from ultrafine-grained austenite was close to Greninger-Troiano (G-T), and the OR of butterfly martensite was close to K-S at outer side of M/A interface and it deviated to G-T at the inner side of M/A interface. The significantly small size of martensite plate transformed from ultrafine-grained austenite resulted in the different ORs from coarse-grained austenite.

### Introduction

Recently, ultrafine-grained (UFG) materials with mean grain sizes smaller than 1 µm fabricated by severe plastic deformation have been extensively studied, because UFG materials exhibit superior mechanical properties [1]. The significantly large area of grain boundaries per unit volume in UFG materials would affect martensitic transformation behavior as martensitic transformation usually begins from grain boundaries. So far, however, there is no deep understanding about

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martensitic transformation from UFG materials. The orientation relationship (OR) of martensite/austenite interface (M/A interface) is especially important to get better understanding of martensitic transformation from a view point of crystallographic aspect [2-6]. The structure of M/A interface represents important information concerning nucleation and growth mechanisms in martensitic transformation [7, 8]. It is well known that martensitic transformation leads to a reproducible OR between the parent and product lattices [9–12]. Concerning the martensitic transformation in ironbased alloys, there are three possible ORs between the austenite and martensite phases, i.e., Greninger-Troiano (G-T) OR  $(\{111\}_A //1^\circ \text{ from } \{011\}_M, \langle \overline{112} \rangle_A //2.5^\circ \text{ from } \langle 0\overline{11} \rangle_M)$ Nishiyama–Wasserman (N–W) OR  $({111}_A//{011}_M,$  $\langle \overline{1}\overline{1}2 \rangle_{A} // \langle 0\overline{1}1 \rangle_{M}$ ) and Kurdjumov–Sachs (K–S) OR ({111}<sub>A</sub>//  $\{011\}_{M}, \langle \overline{1}01 \rangle_{A} // \langle \overline{1}\overline{1}1 \rangle_{M} \rangle$ , where A and M represent austenite (f.c.c. mother phase) and martensite, respectively. The G-T OR is the intermediate OR between K-S and N-W ORs. Different OR is observed depending on martensite morphology, such as lath, butterfly, lenticular, and thin plate [13]. According to recent studies, the OR of martensite can also vary within a given martensite plate [14, 15].

To clarify the characteristics of martensitic transformation from UFG austenite, OR at M/A interface should be assessed in detail, which has, however, not yet to be accomplished. In this study, we investigated the OR at the M/A interface of martensite in a Fe–24Ni–0.3C alloy transformed from coarse-grained austenite and UFG austenite fabricated by the accumulative roll-bonding (ARB) process.

#### **Experimental**

In this study, Fe–24Ni–0.3C wt% alloy sheets were used as starting material. The chemical composition of the sheets is

Table 1	Chemical	composition	of the	alloy	studied	(wt%)

С	Si	Mn	Р	S	Ni	0	Ν	Fe
0.29	0.01	0.07	< 0.005	< 0.0005	24.09	0.0008	0.0006	Bal.



Fig. 1 Schematic illustration showing the principle of the ARB process

represented in Table 1. The starting material had equiaxed austenite grains with mean grain size of 35  $\mu$ m. The sheets were provided to ARB process to obtain UFG structures.

The ARB is a severe plastic deformation (SPD) process using rolling deformation. Figure 1 is a schematic illustration showing the principle of the ARB process. In the ARB process, 50% rolled sheet is cut into two, stacked to be the initial dimension after degreasing and wire brushing the surface, and then rolled again. The rolling in the ARB process is not only a deformation process but also a bonding process (roll-bonding). The ARB process can apply significant amount of plastic strain into the materials, because the procedures can be repeated limitlessly.

In order to prevent deformation-induced martensitic transformation during the ARB process, stacked sheets were held at 873 K (at which austenite phase is stable) for 600 s and subsequently roll-bonded. The ARB was repeated up to six cycles, so that the total equivalent strain accumulated was 4.8. The rolling was carried out using a two high mill with a roll diameter of 310 mm with lubrication. After six cycles ARB process, we observed elongated UFG austenite with the mean grain size (thickness) of 300–400 nm.

Two kinds of method were used to obtain martensitic microstructure: thermally induced martensitic transformation and deformation-induced martensitic transformation. For thermally induced martensitic transformation process, the samples were quenched in liquid nitrogen.

Table 2
Angle-axis
values
for
different
orientation
relationships

between martensite and austenite [16]
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ORs between alpha and gamma	Angle-axis
K–S	42.85 (17.8 17.8 96.8)
G–T	44.26 (12.2 18.4 97.5)
N–W	45.99 (8.3 20.1 97.6)

Deformation-induced martensitic transformation was induced by tensile test of 10–27% strain at room temperature. The field-emission type scanning electron microscope (SEM) equipped with electron back-scattering diffraction (EBSD) and orientation imaging microscopy (OIM) software was used for analyzing the OR between M/A interface. The accuracy of measurement by EBSD was  $\pm 1^{\circ}$ . All the microstructures were observed from the transverse direction (TD) of the sheets.

OR between M/A interface can be expressed by a lattice rotation about a certain axis for a certain angle that leads to the superposition of the coordinate system of martensite and austenite. Table 2 shows the angle-axis values calculated for the ideal K–S, N–W, and G–T ORs [16]. By comparing the experimentally obtained angle-axis pairs with the calculated ones, ORs were determined.

## **Results and discussion**

# Martensite/austenite interface in coarse-grained austenite

To observe the interface and recognize the OR between martensite and parent phase, there are many methods using transmission electron microscopy (TEM) or X-ray diffraction (XRD). However, it is difficult to measure the local OR by XRD because it can measure only the average orientation of a relatively large area. TEM is unable to measure the orientation in a wide area precisely. In contrast, EBSD can measure the orientation in a wide area of bulk-sized specimen easily and precisely.

Figure 2a and b is the EBSD orientation images of thermally induced martensite and deformation-induced martensite (27% strain), respectively, from coarse-grained austenite. The yellow background shows the austenite phase as matrix. Characterized by the smoothly curved M/A interface, the morphology of thermally induced martensite in Fig. 2a is lenticular type. On the other hand, the martensite plates composed of two plates with specific variants are observed in Fig. 2b, indicating that the morphology of the deformation-induced martensite is butterfly type. The boundary maps of thermally induced martensite and





deformation-induced martensite obtained by EBSD measurement are shown in Fig. 3a and b. In the boundary maps, pink and green areas represent martensite and austenite phases, respectively. K–S, G–T, and N–W ORs are shown as blue, purple, and red lines as interfaces. Based on this method, we find that both sides of M/A interface of lenticular martensite thermally induced from coarse-grained austenite is close to K–S OR, as shown in Fig. 3a. Figure 3c shows the local misorientation angle profile of lenticular martensite along x-x' line. We find that the orientation gradually changes from the center of martensite plate (midrib) to the M/A interface, and the regions at both sides of midrib have the same orientation. This indicates that the plate of lenticular martensite nucleates at midrib region and it propagates to both sides, as previously reported by Shibata et al. [14, 17]. In contrast to the lenticular martensite, inner and outer sides of M/A interface in the butterfly martensite have different OR, i.e., outer side is close to K–S and inner side is close to N–W as shown in Fig. 3b. The local misorientation profile from outer to inner side of butterfly martensite is shown in Fig. 3d. This profile indicates that the orientation inside the butterfly martensite changes monotonously. Thus, it is confirmed that butterfly martensite nucleated from one side, and it grew from one side to the other side. Zhang et al. [18] reported that the butterfly martensite nucleated holding K–S OR, and the OR deviated to N–W during the growth. Sato et al. [15] also reported that the inner side of M/A interface of the butterfly

Fig. 3 a, b Distribution of ORs along the M/A interface thermally induced martensitic transformation and deformation-induced martensitic transformed from coarse-grained austenite. c, d misorientation profile along x-x' in a and b



martensite mainly exhibited a G–T OR, while the outer side of M/A interface showed both K–S and G–T ORs. Our results are in good agreement with the previous results.

Martensite/austenite interface in ultrafine-grained austenite

From UFG austenite fabricated by the ARB process (six cycles), two kinds of martensite morphology appeared through a thermally induced martensitic transformation, as shown in the boundary map of Fig. 4a and b. Note that the martensite in Fig. 4a is plate type, and local orientation change in Fig. 4c is quite similar to that of the lenticular martensite in Fig. 3c. We suppose that the morphology of the martensite in Fig. 4a is lenticular type. On the other hand, the martensite in Fig. 4b is butterfly type, because the martensite is composed of two variants and the misorientation inside the martensite plate (Fig. 4d) increases monotonically from the outer to inner side of M/A interface.

As shown in Fig. 4a, both sides of M/A interface of the lenticular martensite transformed from UFG austenite satisfies G–T OR, which is different from that transformed

from coarse-grained austenite (Fig. 3a). Misorientation profile inside the lenticular martensite plate transformed from UFG austenite in Fig. 4c indicates that the amount of misorientation angle from the center of martensite plate reached up to 4° at the M/A interface which is more than that transformed from coarse-grained austenite (Fig. 3c), even though the width of the martensite plate transformed from UFG austenite is three times narrower than that transformed from coarse-grained austenite. Since ARB process is a severe plastic deformation, it introduces high density of dislocations and other defects. We deduced that the inheritance of these dislocations and defects by martensite resulted in large misorientations inside the martensite plate. According to the previous study [14], lenticular martensite holds G-T OR at the earlier stage of transformation, but the OR gradually deviates towards K-S during growth. Due to the significantly small width of martensite plate transformed from UFG austenite, we suppose that the OR remains G-T even after growth of the martensite plate.

As shown in Fig. 4b, the outer side of M/A interface of the butterfly martesnite transformed from UFG austenite holds K–S OR, which is the same as that transformed from



coarse-grained austenite. The OR at inner side of M/A interface, however, is G-T OR and different from that transformed from coarse-grained austenite. As well as the case of the lenticular martensite in Fig. 4a, the difference in OR of the butterfly martensite between that transformed from coarse-grained austenite and that from UFG austenite can be explained in terms of the martensite plate size. According to Fig. 3b, the OR of the butterfly martensite changes from K-S to N-W during growth. Significantly fine austenite grain size may complete growth of the butterfly with a transition state during changing the OR from K-S to N-W, resulting in G-T OR at the inner side of M/A interface in Fig. 4b. There is also a possibility that the high density of dislocations introduced by the ARB process changed the OR, because dislocations could affect the coherency of M/A interface.

## Conclusion

In this research, we studied OR at the M/A interface of martensite transformed from coarse-grained austenite and UFG austenite in a Fe–24Ni–0.3C alloy.

- Morphology of the martensite thermally induced from coarse-grained austenite was lenticular type. The both sides of M/A interface of the lenticular martensite had K–S OR. The misorientation from the middle part of the martensite plate (midrib) reached about 3° at the M/A interface, but the orientation was almost the same on both sides of midrib.
- 2- Butterfly martensite was formed by tensile deformation of coarse-grained austenite. The outer side of M/A interface had OR close to K–S, while the inner side of M/A interface satisfied N–W OR. Because the orientation inside the martensite plate continuously changed from one M/A interface to the other, the OR changed from the K–S to the N–W during the growth.
- 3- From UFG austenite, two kinds of morphologies, i.e., butterfly and lenticular martensites were thermally

transformed. In contrast to the martensite transformed from coarse-grained austenite, M/A interface of both sides of lenticular martensite had OR close to G–T. The outer side of M/A interface of butterfly martensite was close to K–S and it deviated towards G–T at the inner side. The significantly small size of the martensite plate transformed from UFG austenite resulted in different OR from that transformed from coarsegrained austenite.

Acknowledgement This research was financially supported by the Grant-in-Aid for scientific research on innovative area, "Bulk Nanostructured Metals", through Ministry of Education, Culture, Sports, Science and Technology (MEXT), Japan, (contract No. 22102002) and the support is gratefully acknowledged.

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