Role of Annealing Twin in the Formation of Variant Structure of bct Martensite in Fe-Pd Alloy

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A variant structure of bct martensite in Fe-Pd alloys was examined by transmission electron microscopy. The martensite variants constructing a colony which were transformed in a single crystal of austenite consisted of four kinds of variants belonging to a same "plane-group". On the other hand, the variants which were transformed from recrystallized austenite with annealing twins were composed two kinds of them belonging to a same plane group and two of their mirror symmetrical ones about the annealing twin plane. The variants formed at the annealing twins had a common $[011]_b$ direction which was nearly normal to a $(111)_\gamma$ austenite annealing twin plane.

Shape deformation matrices of the variants and the coherency at the interfaces of neighboring variants were calculated by Wechsler, Lieberman and Read's phenomenological theory. The results indicated that control factors of the selection of variants of the colony were not only a magnitude of total shape deformation but also the coherency at variant interfaces. Annealing twins play a role of the nucleation sites of new martensite plates and reduces the number of kinds of variants from 24 to 6 by the formation of variant interfaces at the twin plane.

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I. Introduction

There have been a great deal of work⁽¹⁾⁻⁽⁵⁾ on the variant structures of thermoelastic martensite controlled by self accommodation which reduces the elastic strain associated with the transformation. It has been reported several kinds of martensite variants are formed simultaneously to adjoin to each other. These variant structures are mostly constructed of four kinds of variants whose habit-planes are near by the same $\{110\}$ austenite plane, called by a "plane-group". As the average of shape strain matrices is close to the unit matrix by two orders compared with the original matrix of each variant, it is concluded that this variant structure plays a reducer of elastic strain energy caused by the martensitic transformation.

It has been reported⁽⁶⁾⁽⁷⁾ that the self accommodation of non-thermoelastic martensitic transformation is achieved like the thermoelastic types, though the martensite plates of the non-thermoelastic type nucleate burstly and grow to the last size immediately differing from the thermoelastic one. The specimens used in these investigations were single or large grain size crystals. If the austenite has a small grain size comparable to the martensite plate size and includes annealing twins, as in a recrystallized specimen, it is predicted and actually observed that the variant structure is affected by the grain boundaries or the annealing twin boundaries. The frequently observed variant structures of the small grain specimens with the annealing twins were symmetrical

martensite variant formations about the annealing twins. This paper is aimed to make the comparison of the variant structure and the transformation process between austenite of single crystal and that with a small grain, and to clarify the role of austenite annealing twin in the formation of the variant structure of non-thermoelastic martensite.

II. Experimental Procedure

An alloy of iron-29.8 at% palladium was prepared. After homogenized at 1373 K for 216 ks, it was rolled down to a foil 50 μm thick. A single crystal was also made by a modified Bridgeman method. The crystal was cut down to foils parallel to (100) and they were mechanically thinned to 50 μm thick. Both foils were then solution treated at 1373 K for 36 ks followed by quenching into water. As the martensitic transformation temperature to bct of the alloy was lower than room temperature, it was further sub-zero cooled to obtain bct martensite. Specimens for transmission electron microscopy were made by electro-polishing in a mixed solution of acetic acid : perchloric acid : methanol = 9:1:1 at 275 K. They were examined in a JEM-200CX electron microscope. In order to prevent the specimen from bending by the magnetic field of the objective lens, an antimagnetic pole piece which reduces the magnetic field to 0.5 mT was equipped.

The crystallographic orientation of the austenite and the martensite was determined by both electron diffraction and extinction contour patterns. After tilted the specimen from the initial position so that the incident beam direction might be just parallel to a zone axis, in most cases $\langle 100 \rangle$, $\langle 110 \rangle$, or $\langle 111 \rangle$, the tilting angles from the initial beam direction were read off on the goniometer

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scales. Three- or four-zone axes with low order indices were measured for one variant. From the measured data, a stereographic triangle was drawn. However, because of data uncertainty occurring by the specimen tilt, the angles between measured zone axes were not necessarily consistent with each other. Then the data was corrected so that the mean square distance between the measured zone axis and the pole corresponding to it of the standard stereographic triangle becomes minimum. Other zone axis directions were calculated from these corrected data. Finally, they were plotted on a required stereographic projection, and the orientation relationships were determined. The accuracy of this method is inferior to the conventional X-ray Laue method, but has superiority regarding that the internal structure is examined at the same time and the kind of variant is uniquely determined.

A numerical analysis of bct martensite was carried out, based on the Wechsler, Lieberman and Read phenomenological theory⁽⁸⁾. The required lattice parameters of the austenite and the martensite for the calculation were determined at room temperature by X-ray diffractometry, using Co-K-alpha radiation.

III. Experimental Results

A measured crystallographic orientation relationship between the austenite and the martensite is shown in Fig. 1. The open circles indicate the directions of $\langle 100 \rangle_b$, $\langle 110 \rangle_b$ and $\langle 111 \rangle_b$ zone axes. Two relationships were obtained; they were between the martensite matrix and the austenite, and between the martensite internal twins and the austenite, respectively. This result shows that the crystallographic orientation relationship of the martensitic transformation is the Nishiyama-Wassermann's relationship.

Figure 2 shows optical micrographs of surface relief of martensite transformed in an austenite single crystal. The black and white contrasts show the martensite variants. Taking into account of the appearance of contrast reversal of each variant, it is thought that there are two or four kinds of variants in the colony. In the low

Fig. 1 The plot of the directions of $\langle 100 \rangle_b$, $\langle 110 \rangle_b$ and $\langle 111 \rangle_b$ zone axes on $(010)_f$ stereographic projections showing two types of crystallographic orientation relationships between fcc austenite and bct martensite matrix (a), or internal twin (b) in an Fe-29.8 at%Pd alloy. The subscript b and f means bct martensite and fcc austenite, respectively.

Fig. 2 Optical micrographs showing various surface relief of bct martensite transformed in a single crystal.

martensite density area, the zigzag variant structure is observed. On further cooling, this zigzag structure was developed by the growth of newly induced martensite variants. Thus, in an austenite single crystal, the sites of transformation of new martensite are at the edge of martensite plates which were previously transformed. The reason is that if martensite plates exist, a large stress is accumulated at the edge of plates to induce favorable variants.

Figure 3(a) shows an electron micrograph of the internal structure of such a variant colony. The parallel straight line contrasts in each variant are the traces of internal twins. The type of each variant was determined uniquely by the crystallographic relationship and the trace analysis of internal twins. Their habit planes determined were shown in Fig. 3(b). This figure shows that the variants in the same colony belong to the same plane group and the zigzag variant structure was constructed by the type A and the type C variants. From the directions of the traces of variants, it is found that the variant structures observed as parallel traces were composed of the types A and B, and the kink traces were constructed of the types A and D, or the crystallographically equivalent variant combinations. This results coincide to the result for bct martensite in Fe-Ni-C alloys⁽⁷⁾ and show that the same self accommodation was achieved in the present case.

Figure 4 shows an optical micrograph of the surface relief of martensite transformed in a recrystallized specimen. The average grain sizes of the austenite is about $100 \mu\text{m}$ that is comparable to the sizes of the martensite plates. Thus most of martensite plates reach the grain boundaries, and in this figure are seen symmetrical variant structures about straight lines. The

Fig. 3 (a) An electron micrograph showing a variant structure of bct martensite transformed in a single crystal. (b) Habit plane normals of the variant in (a) on (111) standard projection.

Fig. 5 (a) An electron micrograph showing a variant structure of bct martensite transformed in a recrystallized specimen. (b) Habit plane normals of the variant in (a) on the (111) standard projection.

Fig. 4 An optical micrograph showing surface relief of bct martensite transformed in a recrystallized specimen.

partially transformed specimen showed that the straight lines corresponded to $\{111\}$ annealing twin boundaries of the austenite and most variants were grown from these straight lines. This fact indicates that the sites of transformation of new martensite were the intersection points of the annealing twins and the martensite plates which were previously transformed.

The internal structure of this type colony is shown in

Fig. 5(a), in which the same internal twin traces as Fig. 3(a) are seen. From the contrasts of variants and the internal twin trace directions, it was found that two types of variants were transformed alternately in each side of the austenite annealing twin. The determined variant structures and their habit planes are shown in Fig. 5(b). As mentioned above, these variant structures include the austenite annealing twins. Thus, the habit planes of two types of variants were plotted at the exact index positions, but those of other two types were plotted at the (111) symmetry index positions. In contrast to the single crystal case, the variant combination of type A with type C was not observed, but always observed the combination of the types A and B.

The lattice parameters were measured by X-ray diffractometry. These parameters were obtained at room temperature as follows; $a=0.3756$ nm for fcc austenite and $a=0.2964$ nm and $c=0.3015$ nm for bct martensite, respectively. Atomic volumes of the fcc austenite and the bct martensite are given as 1.325×10^{-2} nm³ and 1.324×10^{-2} nm³. Since the volume change by the transformation is very small, it is suggested that no effective shape change associated with the martensitic transformation takes place, if a suitable variants combination is attained.

IV. Discussion

The expected crystallographic relationships by the observed lattice parameters were calculated by the phenomenological theory. In Table 1 are compared some crystallographic parameters with the observed ones. Each parameter coincides very well, indicating that this martensitic transformation is well described by the phenomenological theory. Then, the calculated crystallographic parameters are adopted for the later discussion.

Generally, a reduction of shape deformation by self accommodation is described by the average of matrices of variants which construct a variant colony, and compared with the original shape deformation matrix. The study of self accommodation of thermoelastic martensite indicated that it is advantageous to take the combination of variants belonging to the same plane group. Table 2 shows the shape change matrices and the maximum strains calculated for observed and supposed variant structures which are indicated in Fig. 6. Every matrix is close to the unit matrix compared with that of the original shape deformation. This indicates that every variants combination in Fig. 6 acts a self accommodation structure. Especially, for four variants combination (Fig. 6(6)(7)(8)), the shape deformation matrices are very close to the unit matrix. But the variants combination of No. 8 in Fig. 6 was never observed, which is not explained only from this total shape deformation matrix analysis.

The calculation of taking the average of matrices is based on that these variants are transformed together to keep contact with each other at a stress-free interface. In non-thermoelastic martensitic transformations, however, each variant is transformed independently and generally some stress energy is accumulated at their interface. In order to estimate coherency at their interface, the calculation was done as follows.

Let consider a unit vector u in austenite. This vector was deformed to u_1 or u_2 by the transformation of variant type 1 or type 2, respectively. Then the difference vector between u_1 and u_2 becomes the strain at the inter-

Table 1 Comparison of experimental and calculated results for some crystallographic parameters of bct martensitic transformation of an Fe-29.8 at%Pd.

	Experimental	Calculated
Habit plane	$\begin{bmatrix} 0.1415 \\ 0.7160 \\ 0.6836 \end{bmatrix}$	$\begin{bmatrix} 0.151548 \\ 0.744435 \\ 0.650269 \end{bmatrix}$
Orientation relationship	$(011)_b$ 0.7 deg. from $(111)_f$ $[01\bar{1}]_b$ 1.0 deg. from $[11\bar{2}]_f$	$(011)_b$ 0.78 deg. from $(111)_f$ $[01\bar{1}]_b$ 0.78 deg. from $[11\bar{2}]_f$
Shape change		$\begin{bmatrix} -0.037237 \\ 0.146830 \\ -0.159780 \end{bmatrix}$

Table 2 Calculated shape deformation matrices and maximum strains for single variant and various variant combinations shown in Fig. 6.

				$\Delta u_{\max}$	
	Original	0.99436	-0.02772	-0.02421	0.1556
		0.02225	1.10931	0.09548	
		-0.02421	-0.11895	0.89610	
(1)	A + B	0.99436	-0.02597	-0.02597	0.0208
		-0.00098	1.00270	-0.01173	
		-0.00098	-0.01173	1.00270	
(2)	A + C	0.99436	-0.00175	0.00175	0.0285
		0.02323	1.00270	-0.01173	
		-0.02323	-0.01173	1.00270	
(3)	A + D	0.99436	0.00000	0.00000	0.1536
		0.00000	1.10931	0.09548	
		0.00000	-0.11895	0.89610	
(4)	A + A'	0.99884	-0.01331	-0.1139	0.0774
		-0.06814	1.02884	0.01343	
		0.04343	-0.04138	0.97209	
(5)	C + C'	0.99884	0.01139	0.01332	0.0774
		-0.04343	0.97209	-0.04138	
		0.06814	0.01343	1.02884	
(6)	A + B + C + D	0.99436	0.00000	0.00000	0.0144
		0.00000	1.00270	-0.01173	
		0.00000	-0.01173	1.00270	
(7)	A + B + A' + B'	0.99884	-0.01235	-0.01235	0.0259
		-0.01235	1.00046	-0.01397	
		-0.01235	-0.01397	1.00046	
(8)	A + C + A' + C'	0.99884	-0.00097	0.00097	0.0610
		-0.05578	1.00046	-0.1397	
		0.05578	-0.01397	1.00046

Fig. 6 Combinations of variants for the calculations of the strain at their interfaces and total shape deformations.

face between two variants. For the various combination of variants illustrated in Fig. 6, the strains at their interface $u_1 - u_2$ were calculated for all directions u . The results were plotted on a (100) standard projection as shown in Fig. 7. For the combination without annealing twins (Fig. 7(1)(2)(3)), the calculation indicates that the

Fig. 7 Equi-strain contours at the interfaces of the variant combinations in Fig. 6(1)–(5) on the (100) standard projection. The figures mean the percent.

combination of type A and B variants and of type A and C variants have an interface which has the strain less than 1%. This interface is expected to be stable for the cases. But the combination of type A and D variants does not have such a small strain interface. This corresponds to the fact that their interfaces were observed to be the extended planer surface for the combination A and B or A and C, and the observed reduced curvature surface for the combination A and D. For the combination with annealing twin (Fig. 7(4)(5)), they must have a (111) interface which corresponds to an annealing twin plane. The combination of A and A' has a strain less than 3% on the (111) interface, indicating the reduction of the strain energy at the interface. On the other hand, the combination of C and C' has a large strain on the (111) interface. Their planes with small interface strains of the combinations of the various variants except for those of types C and C' coincided with the actually observed interfaces.

As mentioned above, when annealing twins exist, the sites of transformation of new martensite were changed from the edge of previously transformed martensite plates to the intersection of an annealing twin and a martensite plate. Then the transforming martensite plates are forced to have an interface at the {111} annealing twin boundary, and another variant is transformed in the other side of annealing twin to compensate the strain energy. But this reduction cannot be performed by all kinds of variants. Figure 8 shows the variant groups for four kinds of annealing twins. When the type I annealing twin exists, the variants which can be transformed at the annealing twin are I_A , I_B , $I_{A'}$ and so on, because other kinds of variants cause large strain energies at the twin

Fig. 8 (001) stereographic projection showing the calculated habit plane normals of the 4 martensite variants groups including 6 variants in it. Each variant belonging the group (I) transforms at the annealing twin (I), and so on.

boundary as shown in the above calculations. These variants have a common [011] direction which is normal to the annealing twin plane. Then the variant structure is restricted only to the combination A, A', B and B' type by the annealing twin.

V. Conclusion

The role of annealing twins of austenite in the bct

martensitic transformation was studied by detailed examinations of the variant structures of Fe-Pd alloys. It was found that their structures were constructed of the restricted number of kinds of variants. The control factors of the selection of variants were as follows.

(1) The reduction of total shape change. This is a common factor for discussion of the self accommodation.

(2) The degree of strains at the interface of variants.

(3) The sites of martensite nucleation.

Taking into consideration of these factors, the number of kinds of variants which can be transformed at the annealing twin is reduced from 24 to 6 kinds of variants.

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REFERENCES

- (1) M. Umemoto and C. M. Wayman: *Acta Met.*, **26** (1970), 1529.
- (2) H. Tas, L. Delaey and A. Deruyttere: *Met. Trans.*, **4** (1973), 2833.
- (3) K. Takezawa, T. Shindo and S. Sato: *Scripta Met.*, **10** (1976), 13.
- (4) T. Saburi and C. M. Wayman: *Acta Met.*, **27** (1979), 979.
- (5) K. Adachi, J. Perkins and C. M. Wayman: *Acta Met.*, **34** (1986), 2471.
- (6) H. Okamoto, M. Oka and I. Tamura: *Trans. JIM*, **19** (1978), 674.
- (7) H. Okamoto and M. Oka: *Acta Met.*, **27** (1979), 1789.
- (8) M. S. Wechsler, D. S. Lieberman and T. A. Read: *Trans. AIME*, **197** (1953), 1503.