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Determination of the parent grain orientation and habit plane normals for β_1' martensite in a Cu–Al–Ni–Mn shape memory alloy

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Martensite plates in β_1' shape memory alloys commonly form self-accommodating groups of four plate variants with habit plane normals clustered about a $\{1\,1\,0\}$ pole of the parent phase. In the present work, the crystallography of martensitic transformation in a Cu–Al–Ni–Mn shape memory alloy has been investigated with particular emphasis on accurate habit plane determination. The characteristically high martensite start (M_s) temperature of β_1' alloys makes it impossible to analyse samples containing isolated plates within a β_1 grain at ambient temperature. However, the parent β_1 grain orientation has been determined in this work by means of a new method of junction plane trace analysis, which is based on the knowledge that the junction planes are precisely $\{1\,1\,0\}_{\beta_1}$ planes. The mutual consistency of the experimental results indicates that this technique of junction plane traces analysis is a viable method for determining the parent β_1 grain orientation. Habit plane normals were determined by two surface trace measurements and referred to the parent crystal basis by using the β_1 grain orientation matrix. The habit plane normal was determined to be close to $\{1\,5\,5\}_{\beta_1}$ with the scatter in a series of mean value determinations being less than $\pm 1.2^\circ$.

1. Introduction

Shape memory phenomena are generally associated with thermoelastic martensitic transformation. In β' shape memory alloys martensite forms self-accommodating groups of four plate variants with habit plane normals clustered about a $\{1\,1\,0\}$ pole of the parent phase. Knowledge of the orientations of the habit planes is essential to an understanding of the detailed crystallography of the transformation and of the shape memory characteristics. Two surface trace analysis is commonly used to determine habit planes relative to the parent phase orientation that may be obtained from back-reflection Laue patterns or from twin vestige measurements. However, neither of these methods are appropriate for fully martensitic structures, and for alloys in this condition, an alternative method for determination of the parent phase orientation must be sought.

One such method, junction plane trace analysis, which is based on the experimental finding that the $(0\,1\,1)_{\beta_1}$ plane is parallel to the $(\bar{1}\,2\,8)_{\beta_1'}$ plane of the 18R structure and is therefore an unrotated plane in the transformation [1–4], has been used to determine the parent β_1 grain orientation. The method is based on the measurements of traces of martensite junction planes that, for β_1' martensites, have been shown [1, 2], to be parallel to a $\{1\,1\,0\}$ plane of the parent body centred cubic (b.c.c.) phase structure. For a cubic crystal, the angle between two $\{1\,1\,0\}$ normals is either 60 or 90°, and the orientation of the lattice of the parent phase is uniquely determined by the locations of two normals provided that the separation is 90°. Should the separation be 60°, there are two solutions to the orientation and additional information, such as the location of a third $\{1\,1\,0\}$ normal, must be used to determine the correct solution. In this case, three $\{1\,1\,0\}_{\beta_1}$ junction planes were measured to overdetermine the orientation and to obtain a check on the mutual consistency of the measured data.

This paper describes a procedure for determination of parent β_1 grain orientations in a Cu–Al–Ni–Mn shape memory alloy by junction plane analysis. The mutual consistency of the angular separations of the planes defined by the junction traces indicates that, at least for this transformation, the method is viable for determination of parent grain orientation. Verification of the method is also provided by the consistency of habit plane measurements and the result that the β_1' habit plane normals were close to a $\{1\,5\,5\}$ plane of the parent phase, consistent with previous measurements.

2. Experimental procedure

2.1. Materials

The material used for the study was a Cu–11.8 wt% Al–4.0 wt% Ni–4.0 wt% Mn shape memory alloy available as hot rolled strip. For this alloy, the disordered b.c.c. high temperature β phase undergoes

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DO_3 ordering to β_1 during quenching and then transforms to the 18R β'_1 martensite at lower temperatures [5–7]. The martensite start (M_s) and finish (M_f) temperatures for the alloy were found to be 46 and 7.5 °C, respectively.

2.2. Specimen preparation

Specimens, approximately $10 \times 4 \times 1$ mm, were cut from the strip, homogenized at 910 °C for 2.25 h then quenched in water to obtain β'_1 martensite. The specimens were mounted in cold setting resin, prepared metallographically and finally electropolished in a saturated solution of chromium trioxide and phosphoric acid for 10–15 s at 10 V to produce a strain free surface suitable for examination with polarized light.

Each specimen was searched for a grain containing three non-parallel junction plane traces and then sectioned to expose the traces in a second surface approximately normal to the original surface. The section surface was prepared metallographically, using the method described by McDougall and Kennon [8], to ensure that the sharpness of the interfacial edge was preserved, then electropolished to remove residual strain.

2.3. Angular measurements

Fig. 1. shows the $\{1\,1\,0\}$ β_1 junction plane (A) of a four plate group. Junction planes between nearly parallel pairs of plates in the four plate group are referred to as habit plane junctions (B and C in Fig. 1). For each specimen used in this study, the angles between the traces of three different $\{1\,1\,0\}$ junction planes in a grain, and the specimen reference edge were measured on the rotating stage of a Leitz MM6 metallograph with an estimated accuracy of $\pm 0.5°$.

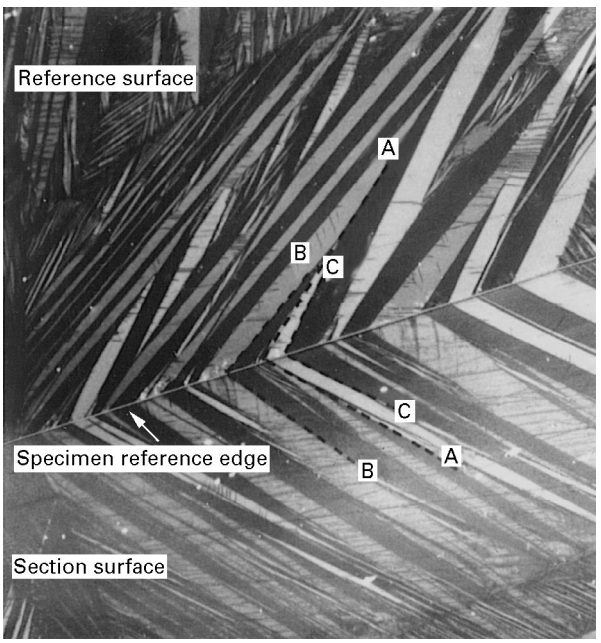

Figure 1 Matched up photomicrographs of two surfaces showing the traces of junction planes (A) and habit junction planes (B and C). Unetched, polarized light x100.

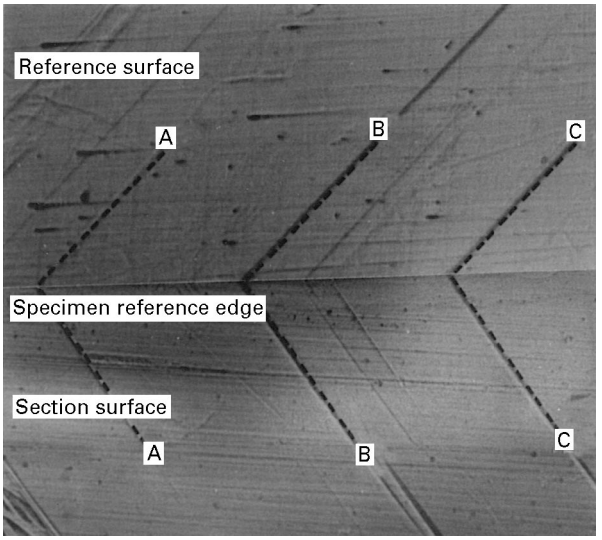

Figure 2 Matched up photomicrographs of two surfaces showing the traces of habit planes (A, B and C). Unetched, oblique illumination, x100.

To distinguish the habit plane traces clearly, a specimen was heated on the microscope stage to above the austenite or parent phase start temperature (A_s) until the specific plate group being measured reverted to the parent phase. The associated relief effects were then photographed in the reference and section surfaces, Fig. 2, and the angles between the habit plane traces and the reference edge were measured from the photographs with an estimated accuracy of $\pm 1°$ using a calibrated protractor.

For each specimen, the angle β_0 between the reference and section surfaces was measured with an estimated accuracy of $\pm 0.05°$ using a Unicam S25 single crystal goniometer.

2.4. Grain orientation determination

For each specimen, the parent β_1 grain orientation was determined directly from the experimental data by numerical analysis. The correlations used in this work were originally provided by Greninger and Troiano [9] in the stereographic projection determination of the orientation of austenite grains from traces of $\{1\,1\,1\}$ twin plates.

Stereographic analysis was used for an initial appraisal of experimental data and for presentation of results, but was insufficiently accurate to ensure that the method was valid. For this latter purpose, the data were processed numerically to obtain the grain orientations.

Essentially, the orientation was specified by a rotation matrix, $\boldsymbol{R}$, which relates the basis, C, defined by the $\langle 1\,0\,0 \rangle$ axes of the β_1 grain to the specimen geometry basis, I, defined by the following: i_1 is the unit vector parallel to the reference edge, i_3 is the unit vector parallel to the normal to the reference surface, and $i_2 = i_3 \times i_1$.

The successive columns of the three by three rotation $\boldsymbol{R}$ (or transformation, $\boldsymbol{T}$) matrix $\boldsymbol{R} = \boldsymbol{ITC}$ are the base vectors $[1\,0\,0]_C$, $[0\,1\,0]_C$ and $[0\,0\,1]_C$ referred to the I basis.

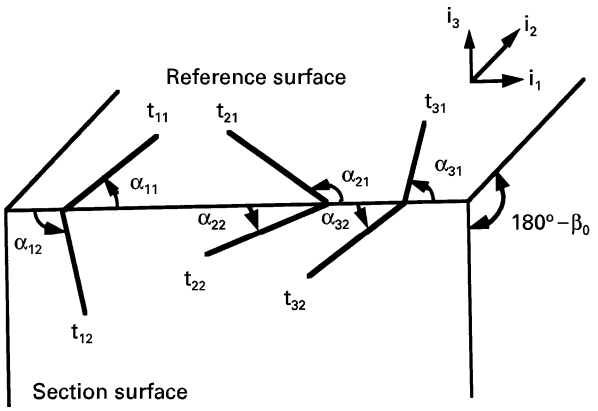

Figure 3 Diagram showing three different pairs or junction plane traces in the two surfaces of a single β_1 grain at an angle of $(180–\beta_0)^\circ$. The angle measurements defining the normal of the junction planes are made relative to the orthonormal basis, *I*.

Initially, the traces, *t*, of the junction planes were referred to the common basis, *I*, by the relationship

$$t = [\cos\alpha;\ \sin\alpha\cos\phi;\ \sin\alpha\sin\phi]_I$$

where α is the angle from i_1 to the trace, measured anti-clockwise, and ϕ is the angle between i_3 and the normal to the plane within which the trace lies (see Fig. 3).

For traces in the reference surface, $\phi = 0^\circ$, while for traces in the section surface, $\phi = \beta_0$.

Thus, the pole of the plane, p_x, defined by traces $t_{x,1}$ in the reference surface and $t_{x,2}$ in the section surface is parallel with $p_x = (t_{x_1} \times t_{x_2})_I$. This pole should be closely parallel with a $\{1\,1\,0\}_C$ pole of the b.c.c parent structure and so consistency of the trace measurements was assessed from the calculated angles between the three poles (p_1, p_2 and p_3) obtained from the dot products, $p_1 \cdot p_2$; $p_1 \cdot p_3$ and $p_2 \cdot p_3$. These products should be 0.5 or zero corresponding to angles of 60 or 90°.

Experimental error resulted in deviations of the dot products from 0.5 or zero so that a correction procedure was applied to ensure that the rotation matrices, ***R***, were exactly orthogonal. Correction was made by selecting the two poles, p_x and p_y, for which the error in the dot product was least, then selecting that pole, say p_x, for which the experimental data appeared to be most reliable. It was then assumed that the location of p_x was correct and that p_y was located in the plane defined by $p_x \times p_y$. Thus, the location of p_y was adjusted to obtain the required precise value of the angle between the two poles. Finally, the location of the third pole was adjusted to obtain the correct values of the other two dot products. After these adjustments the vectors p_1, p_2 and p_3 representing the three $\{1\,1\,0\}_C$ poles were mutually consistent.

The final part of the analysis involved conversion of the poles (p_x; *I*) to the appropriate $\{1\,1\,0\}_C$ plane normal. This step was facilitated by introducing the basis *U* defined by the following where u_1 is the unit vector parallel with p_1, u_2 is the unit vector parallel with $p_1 \times p_3$, and u_3 is $u_1 \times u_2$.

These vectors defined the rotation matrix $\boldsymbol{R}_1 = \boldsymbol{ITU}$ that related the bases *I* and *U*. Specific indices for the vectors (p_1, p_2, p_3; *U*) were identified by reference to the stereographic projection, and relevant cross products then identified the base vectors of the *C* basis. The matrix $\boldsymbol{R}_2 = \boldsymbol{UTC}$ relating the *U* and *C* bases was found from these base vectors. Finally, the required matrix, ***R***, was obtained as

$$\boldsymbol{R} = \boldsymbol{R}_1\boldsymbol{R}_2 = (\boldsymbol{ITU})(\boldsymbol{UTC}) = \boldsymbol{ITC}$$

in which the successive columns of the ***ITC*** are the vectors $[1\,0\,0]_C$, $[0\,1\,0]_C$ and $[0\,0\,1]_C$ referred to the *I* basis.

3. Results and discussion

3.1. The grain orientation

Measurements of the traces of three non-parallel junction planes within a fully martensitic grain in a Cu–Al–Ni–Mn shape memory alloy have been used to determine the orientation of the parent β_1 grain. As the angles between the planes closely approximated to 60 or 90°, each junction plane was confirmed to be a $\{1\,1\,0\}_C$ plane of the b.c.c. parent grain. The average error of determination of the angles between the normals to the planes was 0.6°.

TABLE I β_1 grain orientation matrices for four specimens of Cu–Al–Ni–Mn alloy

Specimen	Matrix (***ITC***)			The cosine values of the three junction plane normals relative to the *C* basis[a]		
1	−0.054 581;	0.854 815;	−0.516 055	0.000 000;	−0.707 110;	0.707 103
	−0.924 518;	0.151 979;	0.349 527	−0.707 103;	0.000 000;	0.707 110
	0.377 210;	0.496 810;	0.781 996	0.707 148;	0.707 125;	−0.000 021
2	0.194 110;	−0.973 910;	0.117 558	0.000 000;	−0.707 145;	0.707 068
	0.969 554;	0.172 219;	−0.174 143	0.707 145;	0.000 000;	0.707 068
	0.149 354;	0.147 780;	0.977 678	0.000 000;	0.707 067;	0.707 145
3	−0.912 226;	−0.341 652;	0.225 948	−0.707 145;	0.707 067;	0.000 072
	0.403 502;	−0.844 780;	0.351 534	0.707 145;	0.000 037;	0.707 178
	0.070 916;	0.411 850;	0.908 497	−0.000 001;	0.707 104;	0.707 107
4	0.846 397;	0.492 663;	−0.202 227	0.707 129;	0.707 146;	0.000 001
	−0.532 477;	0.789 286;	−0.305 770	−0.707 101;	0.000 015;	0.707 113
	0.008 973;	0.366 484;	0.930 381	0.000 000;	−0.707 095;	0.707 117

[a] The *C* basis is the orthonormal set of axes defining the parent crystal structure.

The method described in Section 2.4. was used to determine the prior β_1 grain orientation for four specimens, as set out in Table I. In this table, column two gives the orthogonal matrix ITC, and column three gives the direction cosines for the three junction plane normals, relative to the C basis, used in the analysis for each specimen. Slight deviations from the exact $\{1\,1\,0\}_C$ orientations are due to rounding errors in calculating the matrix ITC. However, it is obvious that the direction cosines closely approximate to $\{\sqrt{(2)}/2,\ \sqrt{(2)}/2, 0\}_C$ and consequently, the angles between each junction plane normal and the $[1\,0\,0]_C$; $[0\,1\,0]_C$; $[0\,0\,1]_C$ axes are very close to either 45 or 90°.

The calculated grain orientation results were used to express measurements of the crystallographic orientation of the habit plane normals in terms of the parent phase crystallography.

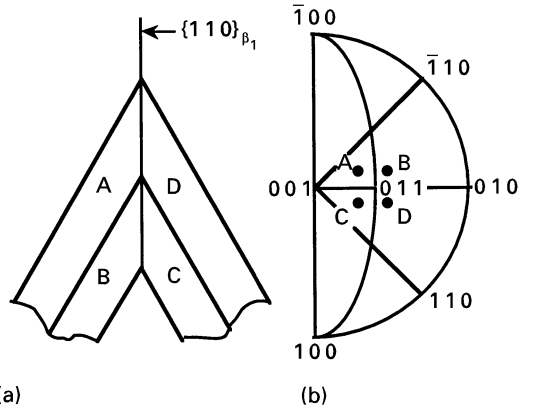

Figure 4 Crystallographic relations between the four martensite various (designated A, B, C and D) in the $(0\,1\,1)_C$ plate group for β'_1 martensite, and (b) habit plane normals of the four variants in (a) in standard projection.

3.2. The habit plane

The martensite habit plane is one of the most important crystallographic features of any martensitic transformation. In a four plate group, as occurs in the Cu–Al–Ni–Mn alloy used in this work, the four habit plane normals cluster about one of the $\{1\,1\,0\}$ poles of the parent phase as shown schematically in Fig. 4. To determine these habit planes for each of the four specimens for which the β_1 grain orientation was determined, traces of the relevant four habit planes, clustered around each of the three junction planes, were measured in the reference and section surfaces.

For each habit plane, the trace data specified the orientation of the plane relative to the I basis, and the rotation matrix ITC was then used to refer the plane to the crystallographic basis C. For each specimen, the 12 measured habit planes were normalized to the standard stereographic triangle $[0\,0\,1] - [0\,1\,1] - [1\,1\,1]$ and the average values for the four specimens are as follows:

Specimen 1: $(-0.153\ 111, 0.684\ 776, 0.712\ 488)_C$
Specimen 2: $(-0.156\ 401, 0.673\ 683, 0.722\ 281)_C$
Specimen 3: $(-0.149\ 300, 0.683\ 549, 0.714\ 472)_C$
Specimen 4: $(-0.155\ 024, 0.684\ 973, 0.711\ 884)_C$

These average habit plane normals are plotted in Fig. 5.

For each specimen, the measured habit planes were highly consistent, with the largest difference between two normals being 2.37°, and the smallest being 0.04°, with an average angular separation of 0.94°. The average of the four mean habit plane measurements was $(-0.153\ 463, 0.681\ 761, 0.715\ 298)_C$, which is 1.57° from $(\bar{1}\,5\,5)_C$.

Differences in the habit plane determinations may be ascribed to systematic error of measurement, together with errors consequent upon steps in the fine structure of the interface and upon inevitable curvature of the surfaces near the reference edge. These small errors in measurement of the junction plane traces necessarily led to errors in determination of the orientation of the junction planes by cross product calculations and subsequently, to determination of $\langle 1\,0\,0\rangle_C$ axes. It was estimated that the cumulative error in the matrix ITC, from all sources, could not exceed 2°.

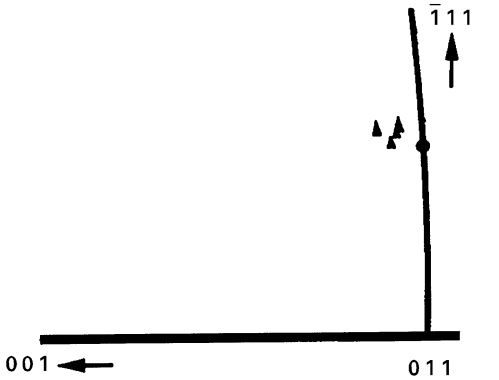

Figure 5 Partial standard stereographic triangle showing the orientations of the average habit planes for the four specimens 1, 2, 3 and 4. (▲) Habit plane normals, (●) $(\bar{1}\,5\,5)_C$.

For copper–based shape memory alloys, the habit plane of β'_1 martensite has been reported to be close to $\{1\,3\,3\}_C$ [10], $\{1\,4\,4\}_C$ [11], $\{1\,5\,5\}_C$ [12], $\{1\,6\,6\}_C$ [13] and $\{2\,11\,12\}_C$ [14]. The present result for the habit plane normal is 1.57° from $\{1\,5\,5\}_C$.

4. Conclusions

For a Cu–11.8 wt% Al–4.0 w% Ni–4.0 wt% Mn shape memory alloy, the β_1 grain orientation and habit plane normals, have been determined from measurements of $\{1\,1\,0\}_{\beta_1}$ junction plane and habit plane traces with the following conclusions.

1. The $\{1\,1\,0\}_{\beta_1}$ junction plane trace analysis method gave self-consistent results for the prior β_1 grain orientation. Although the junction plane measurements and the derived transformation matrix, ITC, were subject to systematic error, the scatter in the four mean experimental results for the habit plane normal was less than $\pm$ 1.2°.

2. The average value of four mean habit plane measurements for the β_1 to β'_1 martensite transformation was found to be: $(-0.153\,463, 0.681\,761, 0.715\,298)_C$, which is about 1.57° from $(\bar{1}\,5\,5)_C$.

References

1. S. KAJIWARA and Z. NISHIYMAM , *Jpn J. Appl. Phys.* **3** (1964) 749.
2. K. OTSUKA and K. SHIMIZU, *Phil. Mag.* **24** (1974) 481.
3. K. SHIMIZU, H. SAKUMOTO and K. OTSUKA , *Trans JIM* **16** (1975) 582.
4. S. A. SALTYKOV, "Stereometric metallography", 3rd Edn (Metallurgizdat, Moscow, 1970).
5. I. ELO, *Ann. Uni. Turkuensis A* **129** (1969) 3.
6. C. M. WAYMAN, "Introduction to the crystallography of martensitic transformation" (Macmillan, London, 1964).
7. K. SHIMIZU and T. TADAKI, "Shape Memory Alloys" (edit. H. Funakubo) (Gordon and Breach, New York, 1987).
8. P. G. McDOUGALL and N. F. KENNON, *J. Aust. Inst. Metals* **5** (1960) 200.
9. A. B. GRENINGER and A. R. TROIANO, *Trans AIME* **140** (1940) 307.
10. P. R. SWANN and H. WARLIMONT, *Acta Metall.* **11** (1963) 511.
11. S. SATO, A. MURAYAMA and Z. NISHIYAMA, *Mem. ISIR Osaka Univ.* **23** (1966) 59.
12. K. OTSUKA, T. NAKAMURA and K. SHIMIZU, *Trans JIM* **15** (1974) 200.
13. K. OTSUKA, K. NAKAI and K. SHIMIZU, *Scripta Metall.* **8** (1974) 913.
14. K. TAKEZAWA, T. SHINDO and S. SATO, *ibid.* **10** (1976) 13.

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