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A new method of determining the orientation of the parent phase and the habit plane normals

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A new general method is proposed for determining the orientation of a parent crystal and the habit plane normals of a plate-shaped product phase without the aid of X-ray experiments. The determination starts with measuring angles between traces formed by the intersection of product phase variants with the surface of a specimen. The results reported are obtained by applying the method to three specific cases. As one of the applications, the unknown orientation of the habit plane of a 2H-stacking ordered martensite in a Ni_3Sn alloy is determined as $\{331\}$.

1. Introduction

The orientations of habit planes of plate-shaped transformation products are usually determined and indexed from the knowledge of the crystal orientation of their parent phases. The crystal orientation in most cases can easily be obtained from the parent phases retained at room temperature by means of X-ray or electron diffraction methods. In some cases, however, parent phases are not retainable at room temperature because their transformation temperature is high. For example, in Cu–Ga and Ni–Sn alloys [1–3] no parent phases can be obtained at room temperature irrespective of the applied quenching rate; when quenched slowly massive transformation occurs while martensitic transformation takes place when quenched quickly. In Fe–Ni, Fe–C and Fe–Ni–C alloys [4] massive martensitic transformation takes place and completes at temperatures above room temperature. Bainitic steels [5] also transform completely at temperatures above room temperature. In these cases, the crystal orientations of their parent phases would be difficult to discover unless high temperature X-ray experiments were employed.

There have been several methods proposed for determining the orientation of cubic crystals without the aid of X-ray experiments. In all the methods non-parallel traces of $\{111\}$ twins or $\{111\}$ slip planes appearing on a specimen surface are employed as references and the determination is made with graphical methods [6] or with the aid of charts or tables [7, 8]. Analytical methods were also proposed by Drazin and Otte [6] and Fong [9]. More recently Hoekstra *et al.* [10–12] simplified and further developed the analysis. They applied their method to the bainitic steel 35 NiCr 18 and determined the crystal orientation

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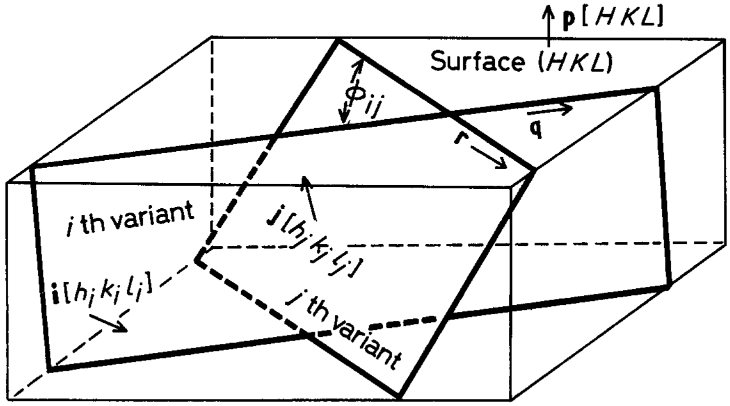

Figure 1 Geometrical relationship of two habit plane traces, ith and jth, appearing on a specimen surface (HKL) of a parent crystal.

of the parent phase using traces of $\{111\}$ twins and then determined the habit plane orientation of the bainite.

The purpose of the present research is to propose a general geometrical method to determine the orientations of parent phase crystals with cubic structure as well as to determine the habit plane orientations of their product phases without any X-ray experiments. The method utilizes the angles between variants of transformation products formed in one grain of their parent phase. As will be shown later, this method is applicable for determining orientations of crystal grains not only with $\{111\}$ traces but also with more general $\{hkl\}$ traces. As one of the applications, the habit plane orientation of a Ni_3Sn martensite (ordered 2H-type structure transformed from DO_3 structure [3]) will be determined with the present method.

2. A new method

Let us assume that the crystal structure of a parent plane is cubic and that the transformation products exhibit plate-like morphology. Usually such a transformation results in the formation of several crystallographically equivalent transformed variants with or without the retained parent phase. On the surface of the specimen, these variants appear as surface traces, the direction of which depends on the orientation of the habit plane and that of the specimen surface.

Fig. 1 represents the geometrical relationship between two traces, non-parallel with each other. Let the one variant be taken as the ith variant and the orientations of its habit plane be denoted as (h_i, k_i, l_i) by using the indices of the parent cubic phase. When a vector $\mathbf{i}$ is chosen normal to the habit plane, the unit vector $\mathbf{q}$ parallel to the direction of the trace then becomes

$$\mathbf{q} = \frac{\mathbf{P} \times \mathbf{i}}{|\mathbf{P} \times \mathbf{i}|}$$

where $\mathbf{P}$ is the vector normal to the specimen surface (HKL). For the other variant, the jth, the unit vector of its trace direction $\mathbf{r}$ is similarily given as

$$\mathbf{r} = \frac{\mathbf{P} \times \mathbf{j}}{|\mathbf{P} \times \mathbf{j}|}$$

where $\mathbf{j}$ is the vector normal to the habit plane of the jth variant, (h_j, k_j, l_j). Here, the surface normal $\mathbf{P}$ is unknown and thus the vectors $\mathbf{q}$ and $\mathbf{r}$ should be used in a combined fashion for the determination of $\mathbf{P}$. The angle made by the two traces thus can be given as

$$\phi_{ij} = \arccos(\mathbf{q} \cdot \mathbf{r}).$$

The angle ϕ_{ij} is a quantity which can be measured experimentally with an optical micrograph.

Of the nine unknown variables $(h_i, k_i, l_i, h_j, k_j, l_j, H, K, L)$ used to describe the angle ϕ_{ij}, only *four* are independent. This is due to the following reasons: (i) $\mathbf{i}$ and $\mathbf{j}$ are considered to be crystallographically equivalent and, thus, if the three components h_i, k_i, and l_i, are found, then the other three components h_j, k_j, l_j can be expressed by the appropriate permutation and assignment of sign of the former three components. This reduces the number of independent variables from nine to six. (ii) Since the magnitude of each vector is not important, we can normalize all the vectors. This further reduces the number from six to four, as for example h, k, H and K. (Hereafter the subscripts i and j will be removed from the vector components of the habit plane.) Thus, the knowledge of *four* independent angles between the various traces of

TABLE I Four types of habit planes.

Type	Habit plane	Variants, n	Independent variables	Least number of traces necessary, tentative
1	$\{hhh\}$	4	2	3
2	$\{hh0\}$	6	3	4
3	$\{hhk\}$	12	3	4
4	$\{hkl\}$	24	4	5

variants will supply the information necessary to obtain the orientations of habit plane $\{hkl\}$ and the specimen surface (HKL). To obtain the four independent angles, we in general need at least *five* independent traces. For more specific cases, however, even a lesser number of traces would be sufficient. Following the above consideration, the minimum number of traces necessary for the determination of such independent variables is classified and tabulated in Table I*, which also includes the number of independent variables and the total number of variants crystallographically expected, n.

For the determination of the habit plane and surface orientation of a parent crystal it is convenient to use a computer. This is because any measured angle ϕ_{ij} is given by a combination of two variants out of their family. The combination is unknown and a suitable combination has to be sought from numerous possibilities, which can be easily done in a computer.

Let the number of variants observed be g out of n, the total number crystallographically expected (Table I). Number all the observed traces from 1 to g and take any one of them as a reference trace (say the ith trace; $1 \leqslant i \leqslant g$), and then measure angles ϕ_{ij} where $j = 1, 2, \ldots g$, and $i \neq j$. The value of ϕ_{ij} can be taken so that it falls in the range of $0 < \phi_{ij} \leqslant 90°$. Next, choose another trace as a reference and measure angles in a similar way. This process is repeated until all angles ϕ_{ij} are measured where $i, j = 1, 2, \ldots g$ and $i \neq j$. Angles thus obtained are compared with angles computed as follows (see Fig. 2):

A plausible plane is assumed to be a habit plane. Then, the orientation of a specimen surface (HKL) is chosen in the 001–011–$\bar{1}11$ standard stereotriangle. For the assumed habit plane

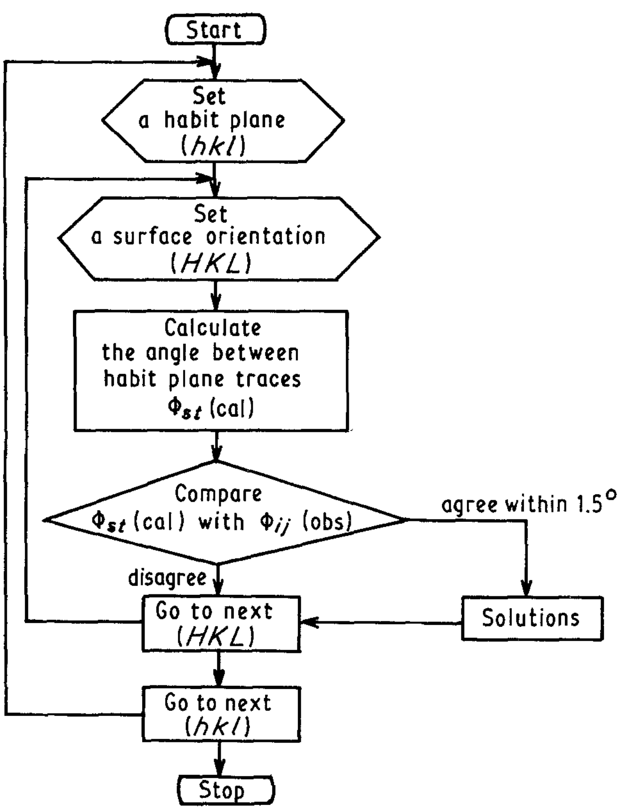

Figure 2 Flow chart of a process for determining habit planes and surface orientations.

*The minimum number does not necessarily mean that it leads to a unique solution. Therefore, the numbers shown in Table I constitute the necessary conditions rather than the sufficient conditions. This point will be discussed later.

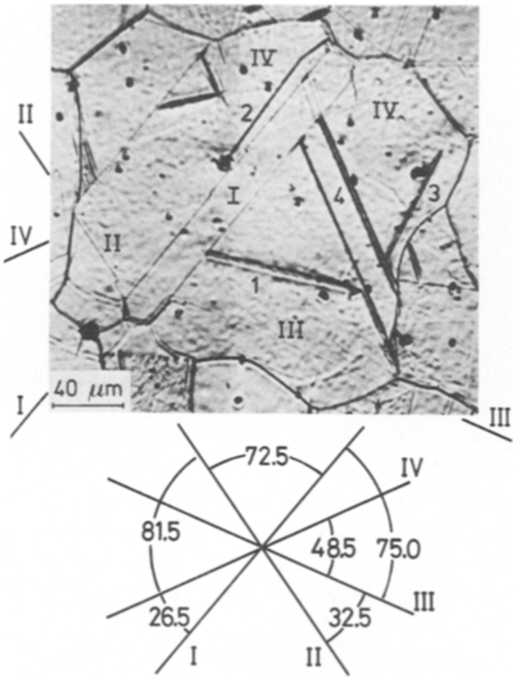

Figure 3 (a) Optical micrograph of a bainitic steel taken at 365° C after partial transformation. Non-parallel annealing twin traces I to IV are seen. (Traces 1 to 4 are bainite variants.) From Hoekstra *et al.* [11], by courtesy of *Acta Metall.* (b) Mutual angles between those twin traces.

orientation and the chosen specimen surface, all possible angles ϕ_{st} are computed for a given reference, say the sth ($1 \leqslant s \leqslant n$) varying t from 1 to n ($t \neq s$). Angles thus computed are compared with the angles ϕ_{ij}. If the measured angles with a fixed i are all in good agreement within a certain allowance with those computed, the assumed habit plane and the selected surface orientation are the ones desired and the results are printed out: that is, the ith trace chosen as a reference is identified and indexed as the sth habit plane. If the desired set of solutions cannot be found after the above procedure, calculation is repeated by changing the surface orientation (HKL) systematically in the stereotriangle and also by changing s up to n so that all the possibilities are investigated.

3. Results

The present method was applied to the three types of habit plane orientation, i.e. types 1, 2 and 3 in Table I. For a specimen surface, about 10 000 different poles were systematically chosen from the stereotriangle and the allowance of the angles used was 1.5° as an estimate of an error in measuring angles from an optical micrograph.

TABLE II Angles ϕ_{ij} between annealing twin traces.

ϕ_{ij}	Traces (j)			
	1	2	3	4
ϕ_{1j}		72.5	75.0	26.5
ϕ_{2j}	72.5		32.5	81.5
ϕ_{3j}	75.0	32.5		48.5
ϕ_{4j}	26.5	81.5	48.5	

3.1. $\{hhh\}$ plane

The habit plane of this type is nothing but the $\{111\}$ plane. Thus, our purpose reduces only to determine the orientation of a specimen surface.

Fig. 3a is an optical micrograph of a partially transformed bainitic steel taken at 365° C by Hoekstra *et al.* [11]. They used non-parallel annealing twin traces I, II, III, and IV and determined the surface orientation to be $(\bar{1}\,5.5\,11)$ applying their graphical method (which is only applicable to the case in which the habit plane is $\{111\}$. We first employed all of the four traces although only three traces are necessary according to Table I. (The case in which three out of four traces are employed will be discussed later.) Fig. 3b shows mutual angles ϕ_{ij} measured between paired traces, which are tabulated in Table II. Angles ϕ_{st} were then computed for a specimen surface selected systematically as in the previous section and a comparison of angles was made. Table III shows one of the results obtained when the surface orientation was chosen as $(\bar{1}\,7\,14)$. Table III indicates that trace III chosen as a reference is the (111) plane, trace I is the $(1\bar{1}1)$, trace II is the $(11\bar{1})$ and trace IV is the $(\bar{1}11)$ plane. In order to demonstrate that indexing was done correctly, a $(\bar{1}\,7\,14)$ stereoprojection is drawn in Fig. 4 showing four $\{111\}$ poles and their trace directions; it can be seen that each of them is parallel to the respective measured trace. (Traces I to IV in Fig. 4 are actually the inversions of those in Fig. 3b. This is because we have assigned the surface normal inside the chosen standard triangle.)

TABLE III Angles calculated between the (111) reference plane and the other $\{111\}$ planes.

Reference (111)	$(1\bar{1}1)$	$(11\bar{1})$	$(\bar{1}11)$
Tr. 1	–	–	–
Tr. 2	–	–	–
Tr. 3	75.01	31.72	48.54
Tr. 4	–	–	–

Specimen orientation $(\bar{1}\,7\,14)$ present; $(\bar{1}\,5.5\,11)$ Hoekstra *et al.* [11].

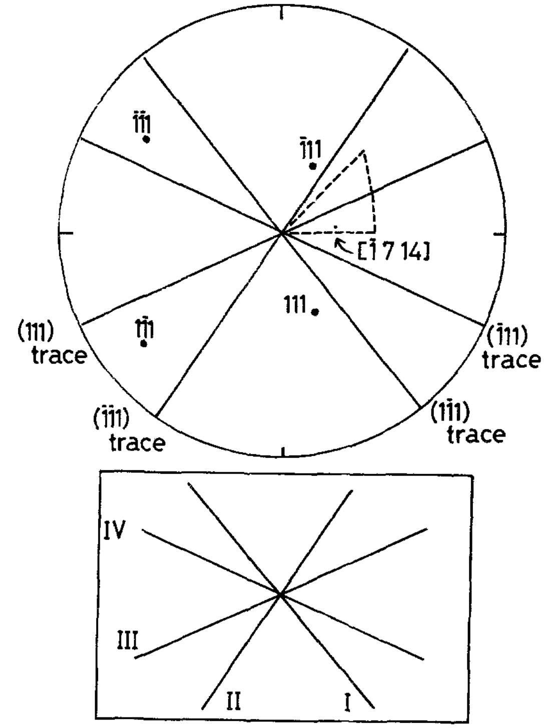

Figure 4 Stereoprojection of the $(\bar{1}\ 7\ 14)$ pole (above) and annealing twin traces (which are actually the reversions of those in Fig. 3b because the surface normal is assigned inside the chosen standard triangle).

In Fig. 4 a standard stereotriangle was also drawn, enclosing one small spot. The spot area indicates all the surface orientations obtained, in which the measured angles ϕ_{3j} in Table II agree with the calculated angles within the angle allowance of 1.5°. The spot area is approximately circular with a radius corresponding to ~0.5° and the $(\bar{1}7\ 14)$ given in Table III is a pole near the centre of the circle. It should be noted that the surface orientation obtained here is in very good agreement (within 1.0°) with that obtained by Hoekstra *et al.* [11], $(\bar{1}\,5.5\,11)$. However, the present method is certainly much easier to use compared with their method.

3.2. $\{h\,h\,0\}$ habit plane: $\{1\ 1\ 0\}$

Let the present method be applied to another type in which the number of variants is greater than in the previous type. Fig. 5 depicts traces of intervariant boundaries of an 18R martensite in a Cu–Zn–Ga crystal, which were taken from Fig. 6 in a paper by Saburi *et al.* [13]. Saburi *et al.* [13], employed back-reflection Laue photography and determined the surface orientation to be

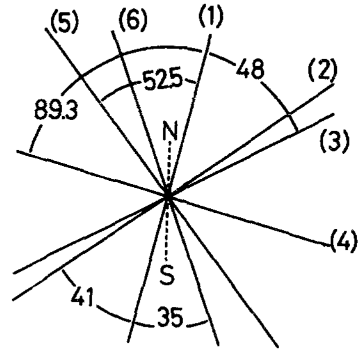

Figure 5 $\{1\,1\,0\}$ intervariant boundaries of 18R martensite in a Cu–Zn–Ga alloy. From Saburi *et al.* [13].

$(\bar{1}\,2.4\,16.1)$ and the habit plane of the intervariant boundary to be $\{1\ 1\ 0\}$.

From the six traces seen in Fig. 5, both five and six traces were employed for the orientation determination. These numbers are both greater than the minimum number given in Table I. Table IV shows one of the results of similar calculations which is obtained when the $\{1\,1\,0\}$ plane was assumed to be the habit plane orientation of the intervariant boundary and also when trace 1 was chosen as a reference line. From Table IV, one can see that trace 1 is the $(1\,1\,0)$, trace 2 the $(0\,1\,1)$, trace 3 the $(0\,1\,\bar{1})$, trace 4 the $(\bar{1}\,1\,0)$, trace 5 the $(1\,0\,1)$ and trace 6 the $(1\,0\,\bar{1})$. The surface orientation here obtained was $(\bar{1}\ 3\ 17)$, which is again very close (within 2.5°) of the $(\bar{1}\ 2.14\ 16.1)$ reported by Saburi *et al.* [13].

Fig. 6 shows the ranges of surface orientations possible within the angle allowance calculated by using five and six traces. The area of the open circle corresponds to the results of the five-trace analysis and is larger than that of the closed circle obtained through the six-trace analysis. Angle deviations of the surface orientation are about 3° in the case of the five traces while it is less than 0.5° in the case of the six traces.

TABLE IV Angles measured between trace 1 and the other traces in Fig. 5 and the angles calculated between the $(1\,1\,0)$ reference plane and the other $\{1\,1\,0\}$ planes for the $(\bar{1}\ 3\ 17)$ specimen surface.

$i = 1, j =$	ϕ_{ij}	$(1\,1\,0)$	ϕ_{cal}
2	41	$(1\,0\,\bar{1})$	33.9
3	48	$(0\,1\,1)$	41.0
4	89.3	$(0\,1\,\bar{1})$	48.0
5	52.5	$(1\,0\,1)$	53.7
6	35	$(1\,\bar{1}\,0)$	89.2

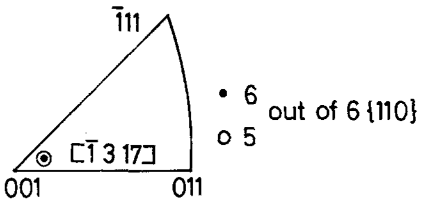

Figure 6 Surface orientation of Fig. 5, determined by using both five and six of the available traces.

The two types (Sections 3.1 and 3.2) used above prove that the present method is simple and useful for the determination of the orientation of a habit plane and a parent phase.

3.3. $\{h\,h\,k\}$ habit plane

Finally, martensite plates in a Ni_3Sn crystal were chosen for the determination of their habit plane. The martensite was produced by quenching a Ni–24.6 at% Sn alloy from 1373 K into iced water. With this heat treatment, the DO_3-type ordered parent phase is known to transform into the 2H-type ordered martensite [3]. The parent phase is unretainable at room temperature regardless of the quenching rate, and therefore the orientation of the habit plane of the martensite has not yet been reported.

Fig. 7 shows an optical micrograph of the martensite. The traces of the six variants were depicted below the micrograph with their mutural angles measured. These angles ϕ_{ij} are tabulated in Table Va. Since six non-parallel traces were obtained, the $\{1\,1\,1\}$ type plane should not be the present habit plane. Among the other possibilities such as $\{1\,1\,0\}$, $\{h\,h\,k\}$ and $\{h\,k\,l\}$, $\{1\,1\,0\}$ was first assumed as the habit plane and a similar process to that aforementioned was followed. However, no suitable surface orientation was obtained. Thus, $\{h\,h\,k\}$ was chosen as a next step. In this case there are many planes which are conceived as $\{h\,h\,k\}$-type planes such as $\{2\,2\,1\}$, $\{3\,3\,1\}$ and $\{4\,4\,1\}$. These

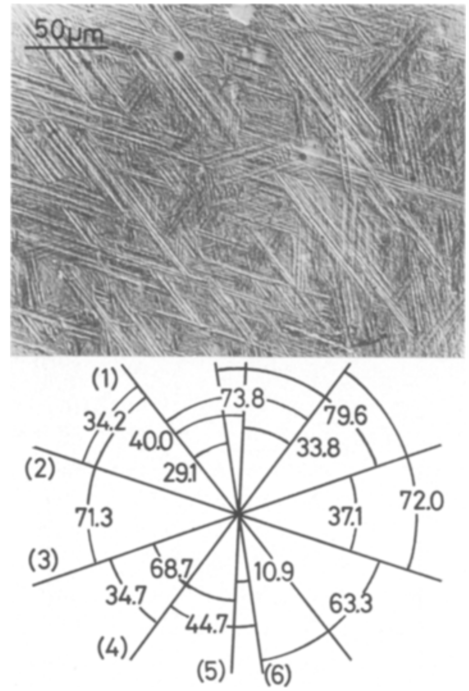

Figure 7 Optical micrograph of 2H martensite variants in a Ni_3Sn alloy, formed by quenching rapidly from 1100° C.

three planes were actually employed and assumed as the habit plane because they are close to a reported habit plane (being between $\{2\,2\,1\}$ and $\{3\,3\,1\}$) of the 2H-type martensite in a Cu–Ni–Al alloy transformed similarly from the DO_3-ordered structure [14]. The calculation revealed that neither $\{2\,2\,1\}$ nor $\{4\,4\,1\}$ was the present habit plane.

The calculated results agreed with the measured angles only when the $\{3\,3\,1\}$-type planes were assumed for the habit plane orientation. As shown in Table Va and b, we can reasonably conclude that trace 1 is $(1\,3\,\bar{3})$, trace 2 is $(\bar{1}\,3\,3)$, trace 3 is $(3\,\bar{1}\,3)$, trace 4 is $(\bar{3}\,1\,3)$, trace 5 is $(3\,1\,\bar{3})$ and trace

TABLE Va Angles ϕ_{ij} measured between the ith and jth traces in Fig. 7.

ϕ_{ij}	Traces (j)					
	1	2	3	4	5	6
ϕ_{1j}		34.2	71.3	73.8	40.0	29.1
ϕ_{2j}	34.2		37.1	72.0	74.2	63.3
ϕ_{3j}	71.3	37.1		34.7	68.7	79.6
ϕ_{4j}	73.8	72.0	34.7		33.8	44.7
ϕ_{5j}	40.0	74.2	68.7	33.8		10.9
ϕ_{6j}	29.1	63.3	79.6	44.7	10.9	

TABLE Vb Angles calculated between the $(3\,1\,\bar{3})$ reference plane and the other $\{1\,3\,3\}$ planes for the $(\bar{1}\;4\;10)$ specimen surface.

Reference $(3\,1\,\bar{3})$	$(1\,3\,\bar{3})$	$(\bar{1}\,3\,3)$	$(3\,\bar{1}\,3)$	$(\bar{3}\,1\,3)$	$(3\,3\,\bar{1})$
Tr. 1	–	–	–	–	–
Tr. 2	–	–	–	–	–
Tr. 3	–	–	–	–	–
Tr. 4	–	–	–	–	–
Tr. 5	41.36	74.35	68.93	32.52	9.95
Tr. 6	–	–	–	–	–

Specimen orientation determined $(\bar{1}\;4\;10)$.

6 is $(3 3 \bar{1})$. In this case, the surface orientation was identified as $(\bar{1}\ 4\ 10)$. Again, it was more accurately obtained by using all six traces (within 0.5°) than by using only five traces (within 3°).

4. Discussion

The new method of habit plane and crystal orientation determination described in the previous sections is not only general but also more convenient to use compared with several other methods [4, 6–11]. The previous methods can only be applied to crystals containing the $\{1 1 1\}$ twin or slip planes for the crystal orientation determination and then stereoprojection analyses are made for the habit plane determination. In the present method, however, any cubic crystal can be used provided it contains the necessary number of variants for the analysis. Besides, this method allows one to determine both habit plane and crystal orientation at the same time.

Recently, Hoekstra *et al.* [10, 11] discussed the least number of $\{1 1 1\}$ plane traces necessary for the determination of crystal orientation. They derived three linear trigonometric equations and concluded that four non-parallel traces are needed to obtain a unique solution. They also concluded that if only three out of four traces are available, there are two, three or four possibilities for the crystal orientation. The results obtained here are consistent with their conclusion: in the case where three out of four $\{1 1 1\}$ traces, labelled I, II and III in Fig. 3, were considered, the orientation obtained was localized around two poles. One was $(\bar{1}\ 7\ 14)$, which is explained in the previous sections and the other was $(\bar{3}\ 5\ 13)$. Angle deviations were large at about 5° around each pole.

Similar results were also obtained in the case of the $\{1 1 0\}$ traces. As shown in Fig. 6, the unique solution of the crystal orientation was obtained when either five or six out of the six traces were available. In the case of only four out of the six traces being considered, angle deviations were very large and it was virtually impossible to identify the unique surface orientation. Unfortunately, the rigorous discussion of necessary and sufficient conditions to give a unique set of solutions for any general orientations of $\{h k l\}$ and $(H K L)$ has not been carried out until now. However, even if more than two sets of solutions are obtained, we believe that we can still make a judgement to select the most reasonable one. For example, other independent information such as surface relief measurement or the applications of the phenomenological crystallographic theory [15–18] will certainly reduce the ambiguity.

In fact, the phenomenological crystallographic theory applied to the present 2H martensite in the Ni–Sn alloy gave the predicted orientation of the habit plane very close to $\{3 3 1\}$ [19], in excellent agreement with the analysed results based on the present study.

5. Conclusions

1. The orientation determination of an unretained parent phase can be made using only an optical micrograph if a great enough number of product phase variants are formed in a planar shape.

2. The present method allows one to determine not only the orientation of parent phase crystals but also that of the habit planes at the same time without the aid of X-ray experiments.

3. The habit plane of a 2H-type martensite in a Ni_3Sn alloy was determined as $\{3 3 1\}$, which is very close to the habit plane of the same type of martensite in a Cu–Al–Ni alloy [14].

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