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An Examination of the Imperfection of Aluminium Crystals

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Abstract

The imperfection of thin aluminium crystal was examined by means of convergent X-rays. The writer could never observe the formation of a perfect single crystal by the stress-annealing method. The axis of small rotation of the crystal fragments was always [2II], and the amount of rotation ranged from 20' to 30'.

Some years ago T. Fujiwara¹ devised a method for examining a crystal with convergent X-rays, and used this method to detect the imperfection of a tungsten crystal.² After him K. Tanaka³ detected the manner of imperfection of a selenium crystal by the same method. This method is especially suitable for finding small inclinations between small crystal fragments when a crystal is composed of such small fragments. In the present experiment the writer applied this method in examining the perfections of thin aluminium crystals prepared by the stress-annealing method which uses K radiation of molybdenum. In this test the specimen was illuminated by the X-rays starting from various points on the surface of the target. Therefore characteristic X-rays having a suitable inclination for a given atomic plane of the crystal are reflected as their spectral lines on the photographic film. When the portion of the specimen which is illuminated by the Xrays is an entirely perfect single crystal, the spectral lines thus obtained are, of course, two doublets of K_{α} and K_{β} rays; but if the crystal is imperfect, and is composed of a few crystal fragments which have nearly the same but a little different orientation, then the spectral lines separate into several components. This method is very sensitive in such an examination, and even a slight imperfection of the crystal which can not be detected by the ordinary Laue-spots, can be brought to light very clearly, as is shown by Figs. 1 and 2, in Plate I. Fig. 1 shows the Laue-spots of a portion of an aluminium crystal and Fig.

^{1.} T. Fujiwara: These Memoirs 11, 283 (1928).

^{2.} T. Fujiwara: These Memoirs 13, 149 (1930).

^{- 3.} K. Tanaka: These Memoirs 17, 59 (1934).

 $_2$ is the photograph taken by illuminating the same portion of the crystal with convergent X-rays. In order to obtain sharp components of the spectral lines, the writer used a conical slit with an opening of 0.1 mm, and the specimen of an aluminium plate 0.1 mm in thickness, which was composed of a single crystal. In taking a photograph, the specimen was first illuminated by convergent X-rays, the central



portion of the photographic film being protected from the direct X-rays with a small lead patch placed just in front of it; and then the image of the direct Xrays normal to the photographic film was impressed on this film by inserting a pin hole slit (S_2) with an opening of 0.3 mm at a predeterminate position in

front of the conical slit (S_1) , and by taking off the small patch in front of the film. Thus, by knowing the position of the direct X-rays which strike the film normally, the determination of the indices of the atomic planes which caused the spectral lines and the diffused Laue-spots is very much facilitated. As is seen in Fig. 2, Plate I, the same spectral lines appearing in a diffused Laue-spot consists of many sharp com-The aspect of such components is enlarged and reproduced ponents. in Fig. 3, Plate I. The distance between the components and the central spot, and the mutual distances between the components were accurately measured by Hilger's travelling micrometer. The indices of the atomic planes which caused the spectral lines were easily determined by the help of the crystallographic globe¹, from the positions of the direct X-rays, Laue-spots and the spectral lines.

Now let the distance between the central spot and a component of the spectral lines be D, and the distance between the sample and the photographic film be D', then we can find the angle φ between the X-rays which is due to the said component, and the direct Xrays which strike the film normally by $\tan \varphi = \frac{D}{D'}$. Thus by know-

^{1.} U. Yoshida: Jap. Journ. Physics, 4, 133 (1927); and S. Takeyama: These Mem., 12, 257 (1929).

ing the values of φ for every component, $\Delta \varphi$ the difference in the values of φ can be found immediately for any pair of the components. Every one of these components must belong to any one of the components which belong to the K_{α} and K_{β} doublets. The performance of this designation is of first importance.

From Bragg's equation $2d_{kkl}\sin\theta = \lambda$, the value of d_{kkl} being known as an atomic plane of known indices, the values of θ_{α_2} , θ_{α_1} and θ_{β_1} are obtained by giving the known values of the wave length of K_{α_2} , K_{α_1} and K_{β_1} lines of molybdenum respectively. The values of $\theta_{\alpha_2} - \theta_{\alpha_1} - \theta_{\beta_1}$ thus calculated are shown in Table I for various atomic

planes. As the intensity of the K_{β_2} component was so weak that this component did not appear on the film in the present case, it was omitted in the table. By comparing the values of $\theta_{\alpha_2} - \theta_{\alpha_1}$ and $\theta_{\alpha_1} - \theta_{\beta_1}$ with those $\Delta \varphi$ obtained for various pairs of the component lines appearing on the film, we can choose a set of the

Table I								
Indices	$\theta_{\alpha_2} - \theta_{\alpha_1}$	$\theta_{\alpha_1} - \theta_{\beta_1}$						
120	5' 20''	1° 21′ 53″						
311	7 22	I 52 54						
33 I	IO 2	2 32 59						
511	. I2 3 6	3827						

component lines as the spectrum of K_{α_2} , K_{α_1} and K_{β_1} lines which are caused by the same crystal fragment of aluminium. Thus when the position of the aluminium crystal which is illuminated by the X-rays consists of two or more crystal fragments oriented a little differently, we obtain a corresponding number of the sets of the spectral lines of K_{α_2} , K_{α_1} and K_{β_1} . When this classification is made, the value of $\Delta \varphi$ between two component lines which are caused by two crystal fragments oriented a little differently and both belonging to one of any of the spectral lines K_{α_2} , K_{α_1} and K_{β_1} , immediately gives the inclination between the corresponding atomic planes of the two crystal When more than two sets of the spectral components fragments. which are caused by different atomic planes of the crystal are impressed on the same photographic film with the same portion of the specimen, we can obtain the amount of rotation of two crystal fragments as a whole, from the inclinations between the corresponding atomic planes belonging to different crystal fragments. This was very easily done with the aid of the crystallographic globe. The results obtained with four different specimens are tabulated in the tables from Table II to Table V.

In these tables the first column gives the indices of the atomic planes, the second column the distances between the central spot and

Indices	D mm	φ	Kind of lin	spectral res	Inclination between the same atomic planes
331	34.30 34.10 34.09 33.90 31.19 31.00	44° 25′ 20″ 44 15 20 44 15 0 44 4 50 41 42 20 41 31 50	α ₂ α ₁ β ₁	$a_2 a_1 \beta_1$	10′20″
511	48.72 48.42 48.34 48.05	54° 18' 10" 54 8 10 54 5 40 53 55 40	α ₂ α ₁	α ₂ α ₁	10′ 0″
210	39.86 39.75 39.48 39.37	48° 42′ 50″ 48 38 0 48 26 40 48 21 50	α ₂ α ₁	α ₂ α ₁	16' 10"

Table II

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10	blo	
ı a	LHC	

Indices	D mm	ę	Kind of sp lines	oectral	Inclination between the same atomic planes
331	29.55 29.38 29.32 29.15 26.87 26.66	44° 34′ 0″ 44 24 0 44 20 30 44 10 20 41 51 0 41 37 20	α ₂ α ₁ β ₁	α ₂ α ₁ β ₁	13'40"
511	42.19 41.86 41.83 41.54	54° 33′ 50″ 54 22 20 54 21 20 54 9 40	α ₂ α ₁	α ₂ α ₁	11' 40''
210	34-55 34-45 34.19 34.09	49° 1′ 40″ 48 56 50 48 44 10 48 39 20	a ₂ a ₁	a ₂ a ₁	17' 30"

Table	TV

Indices	D mm	φ	Kind of spectral lines		Inclination between the same atomic planes	
115	56.90 56.53 56.36 55.99	62° 12' 0" 62 I 40 61 59 30 51 49 0	α ₂ α ₁	α ₂ α ₁	-	10' 30"
331	27.43 27.27 27.14 26.98 24.92 24.66	$\begin{array}{ccccccc} 42^{\circ} & 26' & 0'' \\ 42 & 16 & 0 \\ 42 & 8 & 20 \\ 41 & 58 & 10 \\ 39 & 43 & 0 \\ 39 & 25 & 10 \end{array}$	$a_2 \\ a_1 \\ \beta_1$	α ₂ α ₁ β ₁		17' 40"

Indices	D mm	φ	Kind of spect	al lines	Inclination between the same atomic planes
151	38.03 37.83 37.75 37.55 37.54 37.26 33.77 33.58	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	$\left[\begin{array}{c} \alpha_2 \\ \alpha_1 \\ \alpha_2 \\ \alpha_1 \\ \alpha_2 \\ \alpha_1 \end{array} \right]$ β_1	α ₂ α ₁ β ₁	8' 40'' 13' 10'' 8' 50''

Table IV (Continued)

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Indices	D mm	ę	Kind of spectral lines			Inclination between the same atomic planes
115	45.62 45.21 45.11 44.70	63° 14' 40" 63 2 0 62 59 0 62 46 20	α ₂ α ₁	a ₂ a ₁		15′40″
331	21.19 21.07 20.94 20.82 19.26 19.02	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	α ₂ α ₁ β ₁	α ₂ α ₁ β ₁		20' 40'' 20' 50'' 20' 50''
15ī	29.33 29.27 29.11 29.07 28.86 26.18 26.04	$\begin{array}{c} 51^{\circ} 54' & 0''\\ 51 & 50 & 0\\ 51 & 41 & 20\\ 51 & 39 & 10\\ 51 & 26 & 40\\ 48 & 41 & 40\\ 48 & 33 & 0 \end{array}$	α ₁ β ₁	α ₂ α ₁	α ₂ α ₁ β ₁	8' 40'' 14' 40'' 8' 40''

the spectral lines, the third column the values of φ , the fourth column the designation of the spectral lines, the fifth column the angle be-

tween two corresponding atomic planes which belong to different crystal fragments. Here it must be noted that the spectral lines belonging to a given crystal fragment are tabulated in the same subcolumn in the fourth column. The axis of small rotation of the crystal fragments is obtained in the manner stated before, and the results obtained with four different specimens are represented in Fig. 2 as a stereographic projection. From this figure, it is seen clearly that the axis of small rotation of



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the crystal fragments of aluminium is always [211] as far as the present experiment is concerned.

This axis seems to have a special significance in a crystal composed of the face centred cubic lattice, because this axis has also been found to be the axis of the fibrous arrangement in the case of rolled platinum foil¹ and of a compressed aluminium plate.² The angle of rotation of the crystal fragments around this is tabulated in Table VI.

Lable VI								
No. of the specimen	No. I	No. 2	No. 3	No. 4				
Angle of rotation	24'	28'	20'	.24'				

The angle of such rotation is not always the same and ranges from about 20' to about 30'. In all the writer's experiments with thin specimens of aluminium

about 0.1 mm thick, an entirely perfect single crystal has never been observed.

In conclusion the writer wishes to express his sincere thanks to Prof. U. Yoshida and Dr. K. Tanaka for their kind guidance and suggestions.

1. S. Tanaka: These Memoirs 8, 319 (1925).

2. Y. Fukami: These Memoirs 15, 23 (1932).

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Fig. 2





X 25