2. Thickness Evaluation of the "Trigon" particles of Gold Sol by the Electron Diffraction Method

Eiji SUITO and Natsu UYEDA

(Suito Laboratory)

The "Trigon" particles, the single micro-crystals of metallic gold, dispersed in the acidic gold sol, are so thin as to be almost transparent to electron beam. (This Bull. **26**, 79 (1951)). Recently, in a diffraction pattern, which was rather discontinuous Debye-Scherrer rings, obtained by a three-stage electron microscope (JEM-4) from such flaky crystals, many diffracted spots were found accompanied by series of subsidiary spots ranking in a line through the main spots as shown in Fig. 1. These

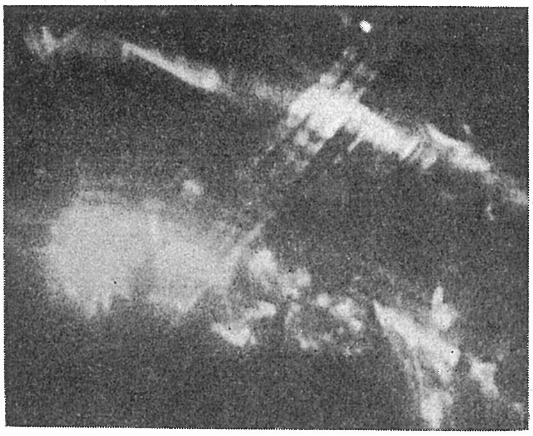

Fig. 1. Diffracted spots accompanied by subsidiary spots of intensity maxima found in the Debye-Scherrer rings of (111) and (200) planes obtained from the "Trigon" particles (camera length : 800 cm).

sets of subsidiary spots are given by the intersection of the Ewald sphere and the set of concentric circles drawn as the loci of the positions of the subsidiary intensity maxima, which appear on both sides of the main maxima ranking along the line parallel to the normal of the flaky surface, because a rotation of the reciprocal lattice takes place owing to the warping of the flaky crystal. (*Proc. Japan Acad.* **21**, 324 (1953)). Each position of these maxima is determined by each of discrete measures, h_n, which have a close relation to the thickness D of the flakes. The relation between the angular width, $\Delta\theta_n$, of the subsidiary diffracted spots to the main one and the value h_n is given by Eq. (1), when d and d_n are interplanar distances of the plane, whose reciplocal lattice point is now under consideration, and the planes along the flaky surfaces respectively,

$$h_n = d_N\left\{\sqrt{\frac{\sin^2\theta_1}{d^2} + \frac{2\Delta\theta_n}{\lambda d} + \frac{(\Delta\theta_n)^2}{\lambda^2}} - \frac{\sin\theta_1}{d}\right\}, \qquad \cdots\cdots\cdots\cdots (1)$$

where θ_1 is the angle between the direction of the incident beam and normal to the flaky surface. If the number of the unit cells along the depth of the flake is given by M, D is equal to $_N$. On the other hand, M is evaluated by the next equation, which can be derived by applying the dynamical theory of electron diffraction (for example, H. Bethe: Ann. d. Phys. 87, (1928) 55);

$$M = 2/\sqrt{h_5^2 - h_3^2}, \qquad \cdots\cdots\cdots\cdots (2)$$

where h_3 and h_5 are evaluated from Eq. (1) with observed values of $\Delta\theta_3$ and $\Delta\theta_5$, which correspond to the lst and 2nd subsidiary diffracted spots respectively. The flaky surface corresponds to (111) planes of the face-centered cubic lattice (E. Suito and N. Uyeda; J. Electronmicroscopy, **1** (1953) 33), and then d_N = 2.35 Å. In the Debye-Scherrer rings obtained, about ten or more spots were elected, and $\Delta\theta_n$s were calculated. The values of M and D were calculated through the agency of Eqs. (1) and (2), and the results are shown in Table 1.

Table 1.

(*hkl*)	$\Delta\theta_n \times 10^4$	h_n	M	D(Å)	h_n'	M'	D'(Å)
(111)	2.03 5.12	0.026 .063	35	82.3	0.027 .068	32	75.2
	3.78 5.73	.047 .069	39	91.7	.049 .076	35	82.3
	4.72 8.88	.058 .103	23	54.1	.062 .117	20	47.0
	2.33 4.15	.030 .051	49	115.2	.031 .055	45	103.4
	2.79 5.07	.035 .062	39	91.7	.037 .067	36	84.6
	2.80 4.85	.035 .055	47	110.5	.037 .064	42	98.7
	4.66 8.31	.057 .096	26	61.1	.061 .109	22	51.7
(200)	6.53 10.71	.049 .079	32	75.2	.050 .082	31	72.9
	3.73 7.00	.028 .052	45	105.8	.029 .053	44	103.4
	5.07 9.33	.038 .069	35	82.3	.039 .071	34	79.9
(311)	2.77 5.05	.078 .130	19	44.7	.087 .016	15	35.3
		M. V.	35	83.1	M. V.	32	75.4

When a simple approximation is adopted to Eq. (1), it becomes as follows :

$$h_n' = d_N \cdot \Delta\theta_n / \lambda \sin\theta_1 \quad \cdots\cdots (3)$$

The D' and M' evaluated by this equation, are also shown in Table 1. They are somewhat smaller than those obtained by the Eq. (1). Whichever method may be adopted, the results show that the thickness of the flaky single microcystals of metallic gold is very thin, and that its distribution extends over a range of about 30–120 Å, The average value of the thickness is about 80 Å in D and about 30 in M values. (see *Proc. Japan Acadenry*, **29**, 331 (1953)).

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3. Formation and Aging of Precipitates. (IV)

Observation of the Gel and the Gelatinous Preciqitate by Electron Microscope

Eiji SUITO and Kazuyoshi TAKIYAMA

(Suito Laboratory)

I. Sol and gel having fiber structure. Some inorganic sols or gels have the fiber structure and a few authors have reported on these sols or gels. The authors have also observed vanadium pentoxide sol and mercury sulfosalicylic acid gel by electron microscope.

Vanadium pentoxide sol : 2 g of NH_4VO_3 was mixed with 6 ml. of 3 N HCl and precipitated V_2O_5 was washed, then it was dispersed in 200 ml. of water. After one night the sol was composed of short fibrils a few mμ wide and about 1 μ long. After one month the fibers became long and its length was a few μ, but the width was only 10 mμ as shown in Photo 1. The fibers have probably grown along a particular axis on standing. (Electron diffraction pattern of the fiber crystal was obtained.)

Mercury sulfosalicylic acid gel : 2 g of sulfosalicylic acid was dissolved in 40 ml. of water and some yellow mercury oxide was added to the solution at 80°C. The solution was heated to boil and stored at room temperature. The solution became gelatinous after one night. The electron microscopic observation showed that the gel had the net work or fiber structure (Photo 2). This structure did not change during a few months.

II. Gelatinous precipitates. $Fe(OH)_3$ and $Al(OH)_3$ precipitates are gelatinous and hard to filter and wash. The authors observed these hydroxides in order to know the general shape of the gelatinous precipitate.