Review

Present State of High Purity Metal Industry in Japan

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"High purity metal" is a word widely used, today. As to what is meant by this word, the author entertains an opinion that a metal may be defined as a "high purity metal" when it is refined so intensively as to be substantially changed in properties, or acquire quite new properties, thus being given new industrial uses on account of these new properties. It is quite natural, therefore, that the purity of a high purity metal varies according to the state of progress of the industries involved, and the kind of metal.

The refining of copper about 1927 may be referred to as to the first production of a high purity metal in Japan. At that time, the domestic copper had a low electrical conductivity, and thus could not be used in the production of conductors. However, upon discovering that this low conductivity was due to the presence of gold, silver and etc. in the copper, removal of these metals from the copper enabled its use as a conducting material. At present, there is no need to dwell upon the fact that high purity metals are playing important roles in the development of industries concerned with electronics, atomic power, high velocity flying objects and etc.

As mentioned above, many kinds of high purity metals are now being produced in Japan to fulfill the demands from various fields. At the same time, active studies are being made on the production, properties and utilization of more high purity metals.

In this paper, the present state of the production of several high purity metals in Japan is reviewed.

Production

1. Germanium

Being restricted by the available resources, high purity germanium is not being produced from its ores, but by reduction of germanium oxide either imported or recovered from germanium scrap, the production being quite peculiar as compared with the production of their metals.

The production of germanium and its oxide in the last several years¹⁾²⁾, and the productive capacity of other producers³⁾ are shown in Tables 1 and 2, respectively.

Although the production decreased sharply in 1963 because of the increasing stock of product, the producers have worked at their full capacity since the

Table 1 Annual Production of Germanium and its Oxide (kg)

Year	Germanium oxide	Germanium
1960	11,388	21,362
1961	14,724	24,373
1962	18,619	22,368
1963	14,385	14,593
1964	20,912	23,859

Table 2 Monthly Productive Capacity of Producers of Germanium and its Oxide (kg)

Company	Germanium oxide	Germanium
Tokyo Denshi Yakin Laboratory	300	250
Tokyo Electronic Metals Co., Ltd.	500	1,020
Mitsui Mining & Smelting Co., Ltd.	300	20
Japan Electric Metal Co., Ltd.	800	350

latter half of 1963 because of the brisk demand for germanium transistors. However, it being considered that silicon may gradually replace germanium in the near future, the merits of germanium and silicon are now under comparative examination from the industrial point of view.

2. Silicon

In contrast with germanium, the production of silicon is not restricted by the problem of resources.

Hydrogen reduction and thermal decomposition of trichlorosilane, thermal decomposition of silane, hydrogen reduction of silicon tetrachloride and etc. have been adopted for the production of silicon.

The production of silicon (polycrystalline)^{1),2)} and the productive capacities of its producers⁴⁾ are shown in Tables 3 and 4, respectively.

Table 3 Annual Production of Silicon (polycrystalline)

Year	Production (kg)
1959	24
1960	2,310
1961	3,921
1962	6,316
1963	10,743
1964	13,629

Table 4 Monthly Productive Capacity of Producers of Silicon (polycrystalline)

Company	Capacity (kg)
Shin-Etsu Chemical Industry Co., Ltd.	250
Komatsu Electronic Metals Co., Ltd.	300
Japan Electric Metal Co., Ltd.	150
Chisso Co., Ltd.	420
Osaka Titanium Co., Ltd.	250
Tokai Electrode Mfg. Co., Ltd.	100 (Stopped in 1965)

Notwithstanding the increase in production of silicon, most of the producers did not obtain a reasonable profit from this product. It is considered that this problem is mainly due to the small production units of the producers and the low market price of silicon, and that this is to be dissolved in the near future.

3. Uranium

Tokai Refinery of Atomic Fuel Corp. has put a test plant in operation to produce uranium from imported uranium concentrate (yellow cake), the product being not only offered to Japan Atomic Energy Research Institute but also to others for various experiments and studies. Mitsubishi Metal Mining Co., Ltd., Furukawa Electric Co., Ltd. and Sumitomo Metal Mining Co., Ltd., investigated the produc-

tion of uranium in their own test plants, and the attempts of most of these companies were successful.

The production of producers of uranium is shown in Table 5.^{5),6)}

Table 5 Annual Production of Producers of Uranium (kg)

Year Company	1961	1962	1963	1964
Atomic Fuel Corp.	14,789	17,435	17,836	2,683
Mitsubishi Metal Mining Co., Ltd.	10	30	6	4
Total	14,799	17,465	17,842	2,687

On the other hand, Atomic Fuel Corp. which has tested a pilot plant for the production of yellow cake from the domestic ore has recently developed a process in which the production of yellow cake is directly connected with the production of uranium. This process comprises the preparation of yellow cake by a sulfuric acid leaching of the domestic ore, the preparation of uranium tetrafluoride from the cake by a wet method called "Excer Process" and the preparation of uranium by magnesium reduction of the tetrafluoride.

4. Tantalum

Tantalite containing more than 60 per cent tantalum pentoxide is being used as a raw material, for the demand of niobium which is contained in tantalite is very small in Japan at present. At the present time, potassium fluotantalite imported is being used with tantalite by some producers, for the production of tantalum may thus be increased without the increase of the facilities necessary for the processing of ore. Tantalum scrap offered by manu-

factures of electronic instruments is also being re-used as a raw material.

The production of producers of capacitor grade and metallurgical grade tantalum^{5),7)} is shown in Table 6.

Table 6 Annual Production of Producers of Tantalum (kg)

Year Company	1961	1962	1963
Komatsu Manufacturing Co., Ltd.	542	630	540
Shin-Etsu Chemical Industry Co., Ltd.	379	816	876
Nippon Soda Co., Ltd.	304	545	1,644
Tokyo Denkai Co., Ltd.	-	325	1,052
Total	1,225	2,316	4,112

For the production of tantalum, sodium reduction of potassium fluotantalite has been adopted by Nippon Soda Co., Ltd., while the electrolytic method had been adopted by the other producers. However, the sodium reduction method has recently been adopted by producers other than Nippon Soda Co., Ltd. too, because of reasons such as that this method is rather preferable for obtaining capacitor grade tantalum powder.

As shown in Table 6, the production in 1963 was larger than that in 1962, for the increase in the consumption of the domestic product in place of imported tantalum.

At present, the productive capacity of each producer is about 200 ~ 300 kilograms per month and remains as yet far from the scale of a commercial production.

To assure the steady development of the production of tantalum, it is necessary to facilitate the obtaining of the raw material, to improve the ties between the producers and the electronic industry, which is a large consumer, and to exploit new potential uses of tantalum. Further,

Table 9 Annual Production of Producers of Magnesium from Anhydrous Magnesium Chloride (metric tons)

Year Company	1959	1960	1961	1962	1963	1964
Osaka Titanium Co., Ltd.	-	-	15	355	713	1,003
Toho Titanium Co., Ltd.	1,634	1,444	1,485	909	699	1,244
Asahi Chemical Industry Co., Ltd.	678	719	645	395	-	-
Sumitomo Chemical Co., Ltd.	604	741	631	274	-	-
Nippon Soda Co., Ltd.	14	112	-	-	-	-
Total	2,930	3,016	2,776	1,933	1,412	2,247

tons in 1964.

The production of magnesium in the last several years ^{1),11)} is shown in Table 8.

On the other hand, the production of magnesium by the method of electrolysis of anhydrous magnesium chloride recovered from magnesium reduction of titanium tetrachloride is shown in Table 9.

7. Aluminum

In Japan, aluminum of a purity higher than 99.2 per cent is called high purity

aluminum, because this is superior in electrical conductivity, light reflection, corrosion resistivity and etc. to the regular grade aluminum.

High purity aluminum is mainly used for making the electrolytic condensers for television and radio sets, and reflecting mirror.

At present, high purity aluminum is being produced by Nippon Light Metal Co., Ltd. and Sumitomo Chemical Co., Ltd. using the triple layer electrolytic refining method.

The production of high purity aluminum ^{1),12)} and that of various purity-grades ¹⁾ are shown in Tables 10 and 11, respectively.

Altogether, 6 electrolytic cells of 45,000 amperes and 18 electrolytic cells of 32,500 amperes are installed in Japan. It was reported that these cells were operated at 82.5 per cent of capacity in 1962.

8. Other high purity metals

Besides the high purity metals mentioned above, high purity tellurium, indium, gallium, bismuth, cadmium, selenium, antimony, arsenic, zinc, lead, copper, tin, gold, silver and etc. are now being produced. Their grades and productive capacities ¹³⁾ are summarized in Table 12.

The production of reactor grade and

Table 10 Annual Production of High Purity Aluminum (metric tons)

Year	Production
1961	1,177
1962	1,929
1963	1,869
1964	1,938

Table 11 Annual Production of High Purity Aluminum of Various Purity-grades (metric tons)

Year Purity	1961	1962
>99.99 per cent	1,056	1,861
>99.95 per cent	106	58
>99.92 per cent	15	10
Total	1,177	1,929

it is important to encourage the demand for the niobium coexisting in the ore.

5. Titanium

The production of titanium was begun in 1953, the product being exported to U.S.A., and has rapidly grown up. However, being affected by the decrease in the manufacturing of military jet planes in U.S.A. after 1957, the export to U.S.A. had remarkably decreased, and the production of titanium in Japan had to be curtailed. On account of this change in the situation, efforts were steadily made to exploit the domestic demand for titanium as a corrosion resisting material for chemical industry, as well as to rationalize the process of production.

As the export to U.S.A. for military uses, the reference from U.S.A. for civilian demand and the distribution for civilian demand in Japan have increased since 1964, the producers are now operating at their full capacity

The production and the distribution of titanium sponge ^{8),9)} are shown in Table 7.

Table 7 Annual Production and Distribution of Titanium Sponge (metric tons)

Year	Production	Distribution		
		Export	Domestic consumption	Total
1959	2,477	1,798	259	2,057
1960	2,307	1,933	394	2,327
1961	2,283	1,567	545	2,112
1962	1,543	1,239	585	1,824
1963	1,759	1,282	863	2,145
1964	2,993	1,606	1,500	3,106

At present, titanium sponge is being produced by Osaka Titanium Co., Ltd. and Toho Titanium Co., Ltd.

A mixture of domestic titanium slag and imported rutile (or ilmenite) is used as the raw material, and the Kroll process was adopted.

In order to fulfill the recent vigorous demand, the productive capacities per month of Osaka Titanium Co., Ltd. and Toho Titanium Co., Ltd. were increased in November, 1964, up to 200 metric tons with an addition of 50 metric tons and up to 170 metric tons with an addition of 30 metric tons, respectively. Both companies are planning to increase the productive capacity ¹⁰⁾

6. Magnesium

The production of magnesium was begun as early as 1929 and had steadily expanded with the development of the aircraft industry. However, since aircraft production stopped at the end of World War II, the production of magnesium was also halted. When the production of titanium, which requires high purity magnesium as a reducing agent, was born, the production of magnesium again started in 1954.

At present, the production of magnesium is being carried out by Furukawa Magnesium Co., Ltd. who uses the domestic dolomite ore as the raw material, and utilizes the ferrosilicon reduction process. This company has remarkably increased her productivity by improving the technique of production. Thus, the monthly productive capacity per one reduction furnace was only 13 metric tons in 1960, but increased to 17 metric tons in 1961 and to 25 metric tons in 1964, the productivity in 1964 being about twice that in 1960. The company's annual productive capacity amounted to 5,700 metric

Table 8 Annual Production of Magnesium

Year	Production (metric tons)
1959	1,563
1960	2,144
1961	2,247
1962	2,087
1963	2,434
1964	2,736

Table 12 Monthly Productive Capacity of Producers of Various High Purity Metals (kg)

Metal	Purity (per cent)	Company	Capacity
Tellurium	>99.999, >99.9999	Mitsubishi Metal Mining Co., Ltd.	500
	>99.999	Mitsui Mining & Smelting Co., Ltd.	100
	>99.999	Nippon Mining Co., Ltd.	100
Indium	>99.999, >99.9999	Mitsubishi Metal Mining Co., Ltd.	10
	>99.999	Mitsui Mining & Smelting Co., Ltd.	100
	>99.999	Tokyo Electronic Metals Co., Ltd.	200
Gallium	>99.999, >99.9999	Mitsubishi Metal Mining Co., Ltd.	5
Bismuth	>99.999, >99.0000	Mitsubishi Metal Mining Co., Ltd.	500
	>99.999	Nippon Mining Co., Ltd.	100
	>99.9999	Nippon Soda Co., Ltd.	100
	< 99.999	Mitsui Mining & Smelting Co., Ltd.	200
Cadmium	< 99.9999	Nippon Soda Co., Ltd.	50 18,000
	>99.999	Mitsui Mining & Smelting Co., Ltd.	
	>99.998	Nippon Mining Co., Ltd.	
	>99.999	Furukawa Electric Co., Ltd.	
Selenium	>99.995	Nippon Mining Co., Ltd.	1,900
	>99.999, >99.9999	Mitsubishi Metal Mining Co., Ltd.	1,000
	>99.999	Mitsui Mining & Smelting Co., Ltd.	100
	>99.99	Sumitomo Metal Mining Co., Ltd.	-
Antimony	>99.999, >99.9999	Mitsubishi Metal Mining Co., Ltd.	50
	>99.999	Mitsui Mining & Smelting Co., Ltd.	50
Arsenic	>99.999, >99.9999	Mitsubishi Metal Mining Co., Ltd.	5
Zinc	>99.999, >99.9999	Mitsui Mining & Smelting Co., Ltd.	1,000
	>99.9999	Nippon Soda Co., Ltd.	50
Lead	>99.999	Mitsui Mining & Smelting Co., Ltd.	100
	>99.999	Mitsubishi Metal Mining Co., Ltd.	50
Copper	>99.999	Mitsubishi Metal Mining Co., Ltd.	2,000
Tin	>99.999	Mitsubishi Metal Mining Co., Ltd.	70
Gold	>99.999	Nippon Mining Co., Ltd.	1.5
Silver	>99.999	Mitsubishi Metal Mining Co., Ltd.	10

commercial grade zirconium had been attempted by Toyo Zirconium Co., Ltd., but was stopped in 1962 because of the stagnation of export and so on. The facilities and technique for the production have been supervised by Central Research Laboratory of Nippon Mining Co., Ltd. who is maintaining the facilities and technique for possible future developments.

Conclusion

During World War II, many high purity metals which had previously never been beyond the object of basic scientific research were taken up as industrial materials and their excellent properties have contributed to a remarkable progress in the fields of science and engineering. Since then their production made a rapid development till it secured the present unique position in the metal industry in Japan.

As to the present position occupied by the high purity metals as industrial materials, it is possible to say that besides the discovery of their excellent properties the newly exploited techniques of refining and working which enabled the high purity metals to be prepared on the industrial scales, such as those used in obtaining germanium for transistors, which vigorously calls for a purity as high as 99.9999999 per cent, or zirconium for atomic reactors, the allowable content of hafnium of which is of the order of one millionth, contribute to the developments of the science and engineering.

Various kinds of high purity metals prepared by highly advanced techniques will make important materials that play an active part in the industry of the so-called Space Age.

In this paper, beginning with proposing an industrial definition of high purity metals, the present state of the production and consumption of the high purity metals in this country are described.

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