Purification of Graphite. I. Removal of Vanadium by Chlorine Gas Treatment

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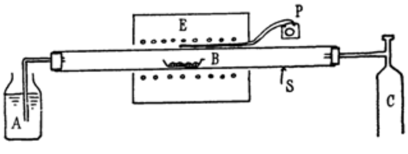
Many kinds of industrial uses require high purity graphite. Particularly anode graphite electrode for the chlorinecaustic electrolysis must be especially freed from vanadium[1] among other metal impurities. In the following study the writers tried to remove a small amount of vanadium from carbon materials using chlorine gas, as Y. Kato and R. Yazima[2] did in removing silicon, aluminum and iron from coke samples.

1) Removal of Vanadium from Powdered Samples

The powder of a graphite or coke sample (0.5 g.) which can pass through a 100 mesh sieve is taken in a porcelain boat (50×8 ×8 mm.) and placed in an electric furnace as shown in Fig 1. It is heated for one to two hours in the current of chlorine gas, the current velocity of which is adjusted at about 20 cc. per minute. Then the sample with the addition of sodium carbonate powder is ashed and the vanadium is determined according



A. 5N–NaOH sol. E. Electric furnace.
B. Porcelain boat. P. Pyrometer.
C. Cylinder chlorine. S. Silica tube.

Fig. 1. Apparatus for Cl_2 treatment.

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[1] G. Angel, T. Lundén, S. Dahlerus and R. Brännland, *J. Electrochem. Soc.*, **102**, 124, 246 (1955).

[2] Y. Kato and R. Yazima, *J. Electrochem. Soc. Japan*, **6**, 168 (1938).

to the colorimetric method proposed by the present writer[3].

The results given in Table I show that vanadium removal is nearly complete at 400°C for the graphite sample, while for coke, amorphous carbon, it is incomplete even at 600°C. It is highly probable that the different behavior between graphite and coke is due to their difference in crystal structure.

TABLE I

REMOVAL OF VANADIUM FROM POWDERED SAMPLE

No.	Sample	Temp. (°C)	Time treated (hr.)	Vanadium content (γ/0.5 g.) original	after Cl_2 treatment
1	Anode graphite	400	1	136	9
2	Anode graphite	400	2	136	7
3	Coke	400	1	230	190
4	Coke	500	1	230	190
5	Coke	600	1	230	200

TABLE II

REMOVAL OF VANADIUM FROM GRAPHITE RODS

No.	Temp. (°C)	Time (hr.)	Vanadium content* after Cl_2 treatment		
			Central part (γ/0.5 g.)	Outer part (γ/0.5 g.)	Removal (%)
1	100	1	100	105	33
2	220	1	92	95	39
3	340	1	21	20	86
4	400	1	11	12	93
5	520	1	9	8	94
6	620	1	6	5	96

* original vanadium content: 140–170 γ/0.5 g., mean 150 γ/0.5 g.

2) Removal of Vanadium from Graphite Rods

A number of rods (10×10×35 mm.) were cut from a sample of anode graphite for mercury cell process, a product of graphitization at a temperature above 2200°C. The rods were treated with chlorine gas in a similar way as in experiment (1). After this treatment, the vanadium content of two separate portions of the rods (outer and central) was determind. The results given in Table II show that the vanadium content does not differ much between these two portions. This fact makes it probable that vanadium escapes as some volatile chloride such as VCl_4, b.p. 154°C or $VOCl_3$, b.p. 127.2°C.

Further, the state of vanadium in the original sample is beyond our present knowledge and the only thing that can definitely be said is that the element can not occur as carbide because vanadium carbide is attacked by chlorine gas only when it is heated red[4]

Studies concerning other impurities will be published shortly.

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[3] K. Sugawara, M. Tanaka and A. Kozawa, This Bulletin, 28, 492 (1955).

[4] H.J Emeléus and J.S. Anderson, "Modern Aspects of Inorganic Chemistry", (1952), p. 475.