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CONDUCTOMETRIC DETERMINATION OF CARBON IN HIGH PURITY MOLYBDENUM, NIOBIUM, TANTALUM AND TUNGSTEN METALS

by

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MINERAL SCIENCES DIVISION

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R.G. Sabourin*, W.R. Inman** and R. Kobus***

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INTRODUCTION

The analytical laboratories of the Mineral Sciences Division of the Mines Branch, Department of Mines and Technical Surveys, have analysed samples of high purity molybdenum, niobium, tantalum and tungsten as part of a collaborative study by the Materials Panel of AGARD of NATO. Previous reports (IR 61-70 and 61-41) have described the methods used, and the results obtained for the determination of iron and nitrogen in these samples.

The subject of this report is the determination of carbon in the samples of high purity molybdenum, niobium, tantalum and tungsten prepared for the AGARD study.

This report is for submission to the Materials Panel of AGARD through Mr. H.V. Kinsey of the Physical Metallurgy Division, Mines Branch.

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PRINCIPLE

A sample is burned in an induction furnace. The resulting products of combustion are passed through a purifying train where the oxides of sulphur and water vapour released from the sample are removed. The remaining CO_2 and excess oxygen are then bubbled through a barium hydroxide solution in the measuring cell of a conductometric carbon determinator. A graph is established by burning several samples of known carbon content and determining the change in resistance caused by each.

APPARATUS

Fisher induction carbon furnace, Model No. 10-465, Serial No. 366*.

Leco conductometric carbon determinator, Model No. 515-000**.

REAGENTS

Barium Hydroxide Solution

Bubble CO_2 free air through 17.5 litres of distilled water for 45 minutes. Bring 1 litre of distilled water to a boil. Weigh 12 g of $Ba(OH)_2 \cdot 8H_2O$ and transfer to a 500 ml beaker. Dissolve the $Ba(OH)_2$ with enough of the boiled water to insure complete dissolution. Filter the $Ba(OH)_2$ solution into the 17.5 litres of CO_2 free water. Wash the beaker with boiled water and filter into the solution. Dilute to 18 litres with the distilled boiled water. Stopper and shake thoroughly. This $Ba(OH)_2$ solution should be prepared approximately one week prior to use.

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* Obtained from Fisher Scientific Co. Ltd., 8505 Devonshire Rd., Montreal 9, Que.

** Obtained from Laboratory Equipment Corporation, St. Joseph, Michigan, U.S.A.

Combustion Boats

Pre-ignite combustion boats designed for use with No. 10-465 Fisher induction carbon furnace for 3 minutes at 2500°F in a Globar type furnace in the presence of oxygen. Cool and place in a desiccator.

Combustion Sleeves

Pre-ignite combustion sleeves designed for use with No. 10-465 Fisher induction carbon furnace in a muffle furnace at 1000°C for several hours. Cool and place in a desiccator.

Accelerator

Tin metal - granular - 30 mesh.

Pure Iron

Pure iron granules - British Chemical Standard No. 149/1.

SAMPLE PREPARATION

Weigh 1.000 g of B.C.S. No. 149/1 and place in a previously ignited combustion boat. Add 0.5 g of tin. Weigh 1.000 g sample for material containing 0-0.035% C or less, or a fractional weight when carbon content is higher. Place in the pre-ignited combustion boat containing the pure iron and tin. Cover the sample with 0.5 g of tin.

PROCEDURE

Fill measuring and compensating cell with barium hydroxide solution. Balance the bridge on the cathode ray scope by adjusting the two vertical, parallel lines to make them coincide. Set the resistance dials at zero.

Place the sample in the combustion chamber of the Fisher induction furnace. Burn sample for 4 minutes with the oxygen flow adjusted at 350-400 ml of oxygen per minute. The reaction between the CO_2 and barium hydroxide in the measuring cell changes the resistance, unbalancing the bridge so that the parallel lines on the cathode ray scope separate. The lines on the scope continue to separate until all the CO_2 has bubbled through the measuring cell, this takes about 7 minutes. Balance the bridge by adjusting the variable resistance until the two lines on the scope coincide. Record the change in resistance. Empty the measuring cell. Remove the burned sample and proceed with the combustion of the next sample.

STANDARD REFERENCE CURVE

Prepare the graph using National Bureau of Standards samples such as 8h, 125A, 129A, 166, 170 and Leco 501-200 standard samples. Carbon values over the range of 0-0.035% plotted on co-ordinate paper result in a straight line. A correction is made for the carbon blank in the pure iron and accelerator. Before performing the determinations reagent blanks are run until constant values are obtained, ensuring that the apparatus has attained stability. Standard samples are analysed concurrently with the samples, to check the standard reference curve.

TABLE 1

Carbon Content of High Purity Molybdenum, Niobium, Tantalum and Tungsten Metals

MOLYBDENUM

Code No.	Source of Material	Per cent Carbon Replicate Values	Mean
Fr/Mo/3	France	0.0046, 0.0043, 0.0042	0.0044
US-Mo-P2F Lot # 200	Wah Chang Corp., Glen Cove, N.Y., U.S.A.	0.0061, 0.0059, 0.0065	0.0060
US-Mo-P2E Lot #Cr2966 RM	General Electric Co., Cleveland, Ohio, U.S.A.	0.0040, 0.0044, 0.0047	0.0044
HM-85	Murex Ltd., U.K.	0.022, 0.025, 0.022	0.023
Ge/Mo (pure metal)	Germany	0.016, 0.017, 0.017	0.017

NIOBIUM

Code No.	Source of Material	Per cent Carbon Replicate Values	Mean
Fr/Nb/1	France	0.089, 0.090, 0.082	0.087
US-Nb-P3B	Fansteel Metallurgical Corp., U.S.A.	0.164, 0.160, 0.156	0.160
HJ-57	Murex Ltd., U.K.	0.069, 0.071, 0.066	0.069
Be/Nb (pure metal powder)	Belgium	0.063, 0.069, 0.064	0.065
Ge/Nb (pure metal powder)	Germany	0.053, 0.041, 0.040	0.045
US/Nb/P5G	United States	0.0043, 0.0044, 0.0046	0.0044

(continued)

TABLE 1 (concluded)

TANTALUM

Code No.	Source of Material	Per cent Carbon Replicate Values	Mean
Fr/Ta/1	France	0.065, 0.072, 0.075	0.071
US/Ta/P3A	National Research Corp., Cambridge, Mass., U.S.A.	0.006, 0.008, 0.007	0.007
US/Ta/P3B	Fansteel Metallurgical Corp., U.S.A.	0.153, 0.162, 0.160	0.158
HM-84	Murex Ltd., U.K.	0.057, 0.055, 0.062	0.058
Ge/Ta (pure metal powder)	Germany	0.037, 0.036, 0.032	0.035
Be/Ta/I	Belgium	0.014, 0.016, 0.014	0.015

TUNGSTEN

Code No.	Source of Material	Per cent Carbon Replicate Values	Mean
Fr/W/3	France	0.0032, 0.0041, 0.0051	0.0041
US/W/P2D	United States	0.0039, 0.0038, 0.0042	0.0040
US/W/P2F	Wah Chang Corp., Glen Cove, N.Y., U.S.A.	0.0025, 0.0023, 0.0022	0.0023
HM-86	Murex Ltd., U.K.	0.015, 0.014, 0.015	0.015
Ge/W (pure metal powder)	Germany	0.0062, 0.0061, 0.0069	0.0064

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RGS:WRI:RK/DV