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Microhardness Tester for Metals at Elevated Temperatures

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An apparatus has been devised for measuring the hardness of electrodeposited coatings at temperatures up to 900° C in an inert atmosphere. Coatings thicker than about 0.07 millimeter (0.003 inch) may be tested. The main parts of the apparatus are: (1) The indenting mechanism, consisting of a Vicker's diamond mounted on a shaft of fused silica; (2) a mechanical device for raising and lowering the indenter; (3) a micrometer device for orienting the specimen under the indenter; (4) the heating unit. The force on the indenter is varied either by dead-weight loading or by changing the gas pressure inside of the apparatus. Some typical measurements made with the apparatus are given.

I. Introduction

The hot-hardness of a metal may be an important indication of its performance in certain applications at elevated temperatures. As a relation usually exists between the hardness of a metal and some of its other mechanical properties, such as tensile strength, a measurement of hardness may also be useful for obtaining an estimate of those mechanical properties that are more difficult to measure. The development of a microhardness tester that could be used for testing electrodeposits at elevated temperatures was undertaken in connection with studies of electrodeposited metals and alloys.

The hot-hardness of bulk metals has been measured by the same methods as those used at room temperature. These methods utilized a variety of different types of indenters to produce indentations in metals by static or dynamic loading. Other methods were based on mutual indentation procedures, which involved pressing together two cylinders of metal. None of these methods will be discussed in detail because excellent reviews may be found in the literature [1, 2, 3].[1] Of various types of indenters used, the only ones suitable for metal coatings are pointed diamonds, by means of which a micro-indentation may be produced with a small load. Hot-hardness testers with diamond indenters have been designed by P. Bens [4] and E. C. Bishop and M. Cohen [5] for use on bulk metals. However, with these instruments the high loads customary for bulk materials were used in producing the indentations. The procedure consisted in placing the heating unit with the specimen on the platform of a commercial hardness tester and then applying the load on the indenter with that instrument.

The loads ordinarily used in measuring the hardness of bulk metals are too large for use on electrodeposits because they result in too deep an indentation, and in testing electrodeposits at room temperature so-called microhardness testers are used. An ordinary hardness tester and a microhardness tester differ mainly in the magnitude of the load applied to the indenter. The same type of indenter is used for both. Whereas in a Vickers machine the load is usually of the order of 10 kg, in a microtester, the load will range from 25 to 1,000 g. Low loads are particularly necessary in testing hardness at elevated temperatures because metals are considerably softer than at room temperature.

The following example will illustrate the requirement of a low load in the measurement of electrodeposits. A metal that is quite hard at room temperature may have a hardness of only 50 Vickers at an elevated temperature. The impression made with the usual 10 kg-load would have a depth of about 0.087 mm (0.0035 in.). As it is usually considered that the thickness of a coating should be at least 10 times the depth of the indentation to obviate any effect of the basis metal, the coating would have to be about 0.87 mm (0.035 in.) thick for a satisfactory measurement. If a load of 200 g were used, the depth of the impression would be only 0.012 mm. (0.0005 in.). Thus the coating need be only 0.12 mm (0.005 in.) thick. An indentation made with a 10 kg-load would have almost penetrated a coating of this thickness.

II. Design of the Microhardness Tester

The apparatus of Bens and that of Bishop and Cohen were not suitable for measuring the hot-hardness of electrodeposits, because of the high loads used. Some measurements were made by Bens on electro deposits produced in this Bureau; but these deposits were fairly thick, and it is not certain that the effect of the basis metal was obviated. The hot-hardness tester described in this report followed to some extent the designs of Bens and of Bishop and Cohen. Two difficulties are involved in adapting microhardness testing to elevated temperatures. (1) A closed system with an inert atmosphere must be used to prevent oxidation of the specimens. If the specimen were allowed to oxidize, the thickness of the oxide film might be comparable to the depth of the indentation produced with a light load, and the hardness of the film might be quite different from that of the metal. Also, oxidation occurring after a microindentation had been made would obliterate the impression. (2) In order that a light load may be applied accurately, the shaft carrying the indenter

[1] Figures in brackets indicate the literature references at the end of this paper.

must move in a bearing with very little friction. There must be no galling of the bearing at the elevated temperatures. Since a closed system must be used to prevent entrance of air, the clearance between the rod and the bearing must be small.

Bishop and Cohen maintained an inert atmosphere in their apparatus. The shaft carrying the indenter was connected to an annular oil seal. This type of construction is unsuitable for a microhardness tester for several reasons: Small variations in gas pressure would cause large variations in the load on the indenter; the apparatus could not be evacuated for the initial removal of air; the horizontal position of the indenter is not fixed; and cumbersome cooling coils must be used to chill the oil. Bens used an evacuated apparatus, and the shaft of the indenter moved in a closely fitting bearing, lubricated with oil to hold the vacuum. This latter arrangement would not be suitable with the type of apparatus designed at this Bureau, as the bearing became too hot to be lubricated with oil. Also a vacuum was out of the question, as the atmospheric pressure alone would have imposed a load of about 500 g on the indenter.

The problems referred to were met by using in the tester an inert atmosphere slightly above atmospheric pressure. The indenter shaft was a capillary of fused silica, which moved in a dry bearing lubricated with graphite. The clearance between the shaft and the bearing was about 0.001 in., and gas escaped slowly through this space during the test. The friction of the shaft in the bearing was equivalent to 1 or 2 g at room temperature and was probably less at elevated temperatures, as the clearance between the bearing and the indenter shaft increased. Figure 1 shows the details of the construction of the indenter assembly. The indenter fitting, B, was attached to a tungsten rod, A, which passed up through the capillary, G, and was held by a collar and set screw, C. A spring arrangement was provided to hold the tungsten rod under continuous tension and thus allow for its lengthening due to thermal expansion. This indirect method of securing the indenter to the shaft was necessary because a fused silica rod could not be anchored directly to the indenter fitting. A few trials were made to cement a silica rod in the cup of the fitting by using molten potassium bisulphate. Although the rod was satisfactorily anchored on two occasions, after 1 or 2 weeks the rod cracked just above the top of the cup.

It might be of interest to note two other methods of mounting the indenter that were tried without success. A stainless steel shaft was first tried, and although it operated well at room temperature, it would gall and seize at elevated temperatures. A sapphire rod 3 mm (⅛ in.) in diameter was also tried as an indenter shaft, but it was found to be too sensitive to thermal shock, and cracked.

The complete setup used for measuring hot hardness is shown in figure 2. A sketch of the cross section of the hardness tester in figure 3 shows the relation of the working parts. The details of construction of the important parts are shown in figures 1, 4, and 5. The tester is self-contained with the loading mechanism, indenter, and furnace all built into one compact unit. The load is applied to the indenter by dead-weight loading.

The main parts of the tester will now be described briefly. The muffle, before being wound with resistance wire, is shown in figure 4. It was made by welding heavy walled stainless steel pipes together. The anvil was a solid piece of stainless steel welded in tube A. It is provided with a small hole extending to within 3 mm (⅛ in.) of the upper surface to accommodate a thermocouple. The flange, B, was bolted to the top plate of the tester with a silver gasket inbetween to make the apparatus gas tight. A doughnut-shaped disk of insulating brick was placed in the space within the flange to decrease the heat transfer to the top plate of the tester. To provide uniformity of heating, the muffle unit was wound on all four limbs with a continuous length of No. 16 Nichrome wire on an Alundum form. The space between the furnace unit and the main shell was filled with insulation. By making the muffle assembly a single welded unit, and by supporting it from a single point, the relative motion between various parts resulting from thermal expansion was minimized.

The indenter assembly was raised and lowered at the rate of 2 cm (0.75 in.) per minute by a small motor that operated a simple screw mechanism similar to that used on the compound rest of a lathe. The vibration of the motor was damped by a mounting of rubber shock absorbers, and no difficulties were encountered from this source. The disk, E, figure 1, supporting the indenter assembly, rested on two pieces of rubber tubing, which served to make the application of the load more gradual when the supporting frame was lowered away from the assembly.

The indenter assembly weighed about 200 g. This was the maximum load applied in the tests, although larger loads could be applied by placing doughnut-shaped weights on the top of the indenter assembly. Usually lower loads were used at elevated temperatures. These were obtained by increasing the gas pressure in the tester above atmospheric. Each centimeter of mercury was equivalent to a decrease of 7 g in the load applied to the indenter. The smallest load used in the tests was about 30 g.

The assembly for holding and moving the specimens is shown in figure 5. The specimens measure 6 by 6 by 12 mm (0.25 by 0.25 by 0.5 in.) and are polished by metallographic procedures. Six specimens may be indented in one heat. They fit into the carriage, B, which slides in a keyway on the inside of the muffle. The carriage is moved along by a stainless steel micrometer screw, which has been plated with cobalt-tungsten alloy to prevent seizing at elevated temperatures. The micrometer serves to locate an indentation within about 0.2 mm (0.008 in.) of a desired spot.

Because the indentations are microscopic in size, they are difficult to locate in the small field of a microscope when making the subsequent measurements of diagonals. For this reason a fitting (D, fig. 5) was designed to hold the carriage in a definite

position on the moving microscope stage. By setting the microscope stage at the same reading as that on the micrometer when the indentation was made, the impression appeared in the field.

The temperature of the furnace was measured by a thermocouple that was placed in the cavity extending up through the anvil. In preliminary checking the temperature within the muffle was measured also with a thermocouple inserted through the top bearing of the tester. The two thermocouples agreed within about 5 or 10 deg. centigrade. The temperature was automatically controlled by a commercial pyrometer.

In addition to its use for measuring the hot-hardness of electrodeposits, the microhardness tester may be used for testing bulk metals and has several advantages over the two instruments on which it was based.

1. It is a self-contained instrument, and incorporates within one unit both the furnace and the device for loading the indenter.
2. Six specimens may be tested in one heating cycle.
3. The micrometer screw permits indentation to be made at specified points and enables them subsequently to be readily located in the microscopic field.
4. The small loads are less likely to cause the diamond to shift in its mounting (usually consisting of monel metal) when measurements are made at very high temperatures.

III. Procedure for Making Measurements

The first step in using the hot-hardness tester, after placing the specimens in the instrument, was to evacuate the apparatus and then flush with hydrogen. Nitrogen or argon could also be used. Before turning the current on the furnace, the atmosphere in the apparatus was tested by heating the fused silica tube, G, figure 2, which contained a strip of bright steel. If the steel remained bright on heating and cooling, the tester was heated.

The gas was passed over heated copper (B, fig. 2) to remove oxygen, and then through a drying tube, before reaching the furnace. The flow of gas was measured with a flow meter, A, and was of the order of 60 ml/min, under a pressure of from 3 to 25 cm of mercury. The tester was kept under a positive pressure of gas during the heating, both to regulate the load on the indenter and to prevent the entrance of air, as the apparatus was not vaccum tight.

The pressure in the apparatus was kept constant without difficulty by regulating the needle valve on the gas tank and the needle valve, J, figure 2, on the exit end of the system.

After the heat was turned on the furnace heated up rapidly. About 10 min were required to reach a temperature of 600° C and about 15 min additional were required to reach a temperature of 800° C. The furnace was held at any one temperature for about 15 min before making indentations. At each temperature a set of three indentations were made about 0.01 in. apart, according to preselected micrometer settings. Along with each set of specimens, a molybdenum specimen was used as a check on the precision of the run. The apparatus has been used at 900° C, but most of the data were obtained at a maximum temperature of 800° C.

The accuracy of the hot-hardness tester could be determined only at room temperature. This was done by comparing with a standard type of microhardness tester. Measurements were made with the hot-hardness tester on a variety of different types of specimens, which were then measured with an Eberbach microhardness tester. The agreement of the results, on an average, was within 4 percent.

The reproducibility of the instrument was determined by measurements on a molybenum specimen. In four runs, coverirg the range of room temperature, 400°, 600°, and 800° C, the average deviation from the mean hardness at each temperature was less than 4 percent. Because of the reproducibility of its hardness, the molybdenum specimen was used as a check on the proper functioning of the hardness tester, and a molybdenum specimen was included along with each set of test specimens.

IV. Measurements

Some of the data obtained with the hot-hardness tester, chiefly on electrodeposits, are shown in figures 6, 7, and 8. These data are given to illustrate the use of the instrument and are not presented as part of a systematic investigation of the hot-hardness of metals. Figure 6 shows that soft chromium and hard chromium are about equally soft above 600° C. At 800° C. the hardness of both has dropped to about 100 Vickers, as compared with the high initial values of 600 and 1,000 Vickers.

It will be noted that chromium and nickel become permanently softened as a result of the heat treatment, that is, on cooling to room temperature the hardness is lower than initially. In contrast to this, cobalt and cobalt alloys, figures 7 and 8, regain a large part of their initial hardness on cooling, or may even increase in hardness, provided that the maximum temperature has not been excessive. The maintenance or increase in hardness of a metal after heat treatment cannot be considered as evidence of good hot-hardness. This is demonstrated particularly well by the data on the cobalt phosphorus alloys, figure 7, which have a hardness of only 5 Vickers at 800° C. but which become harder than initially after cooling.

The hot-hardness of the cobalt-tungsten alloys, figure 8, appears to increase with their tungsten content. The alloy containing 31 percent of tungsten is outstanding in hot-hardness. At 800° C. it is as hard as cobalt is at room temperature. Unfortunately, the higher the tungsten content, the more difficult the alloys are to deposit, and the more likely they are to be excessively brittle and to crack on heating, especially when deposited on a steel base.

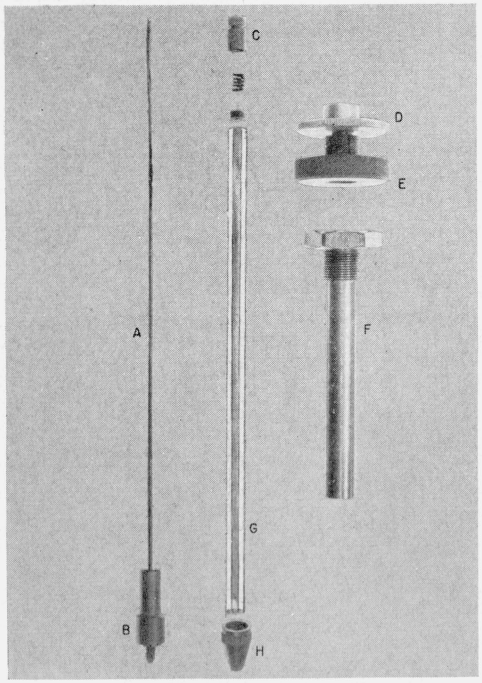

FIGURE 1. *Indenter assembly for hot-hardness tester.*

A, Tungsten rod, 2-mm diameter; B, stainless steel adapter for Vicker's indenter H; C, collar with set screw; D, chuck for gripping silica capillary, G; E, nut for tightening chuck; F, bearing for silica capillary; G, silica capillary, approximately 8 mm in diamter and approximately 2 cm long.

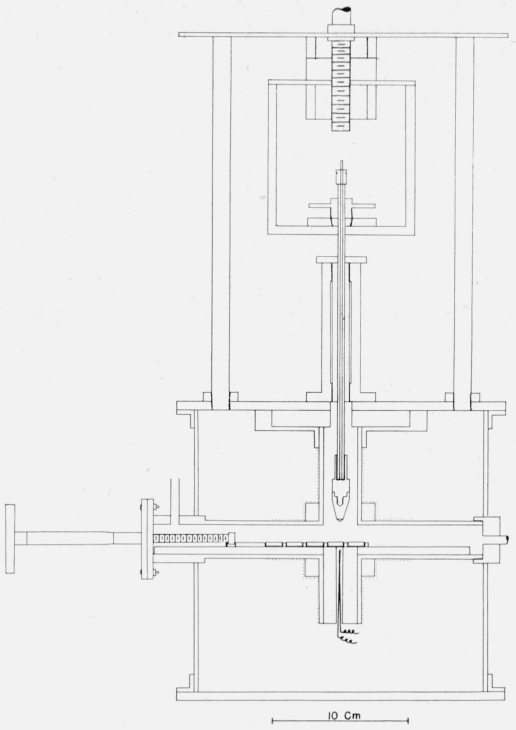

FIGURE 3. *Schematic cross section of hot-hardness tester.*

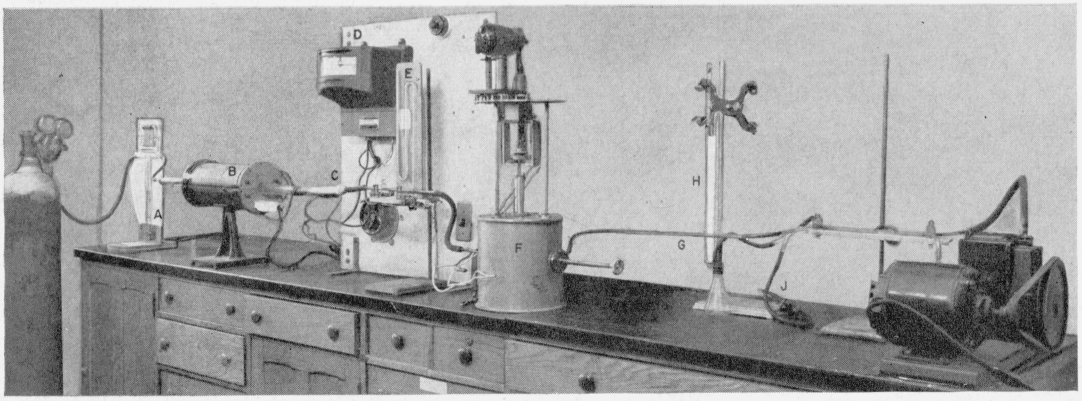

FIGURE 2. *Hot-hardness tester and auxiliary equipment.*

A, Flowmeter; B, apparatus for removing oxygen with hot copper; C, drying tube for gas; D, pyrometer and automatic temperature controller; E, manometer; F, hot-hardness tester; G, transparent silica tube, containing bright steel strip for testing purity of gas; H, open end mercury manometer; J, needle valve at exit of system.

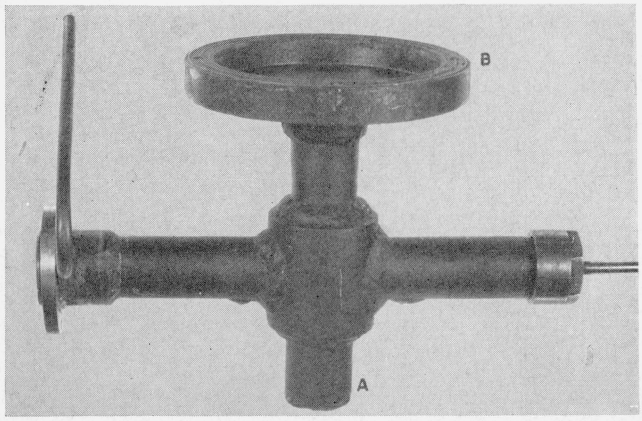

FIGURE 4. *Stainless steel muffle before being wound with heating element.*

A, Anvil; B, flange for bolting to top plate of outside shell.

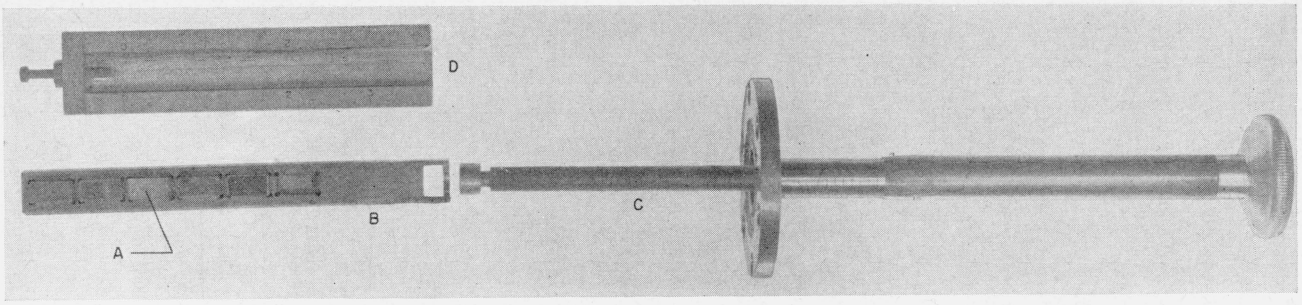

FIGURE 5. *Devices for holding and moving test specimens.*

A, Specimen; B, carriage, 13.3 cm long; C, micrometer screw, plated with cobalttungsten alloy; D, fitting for orienting carriage on moving microscope stage.

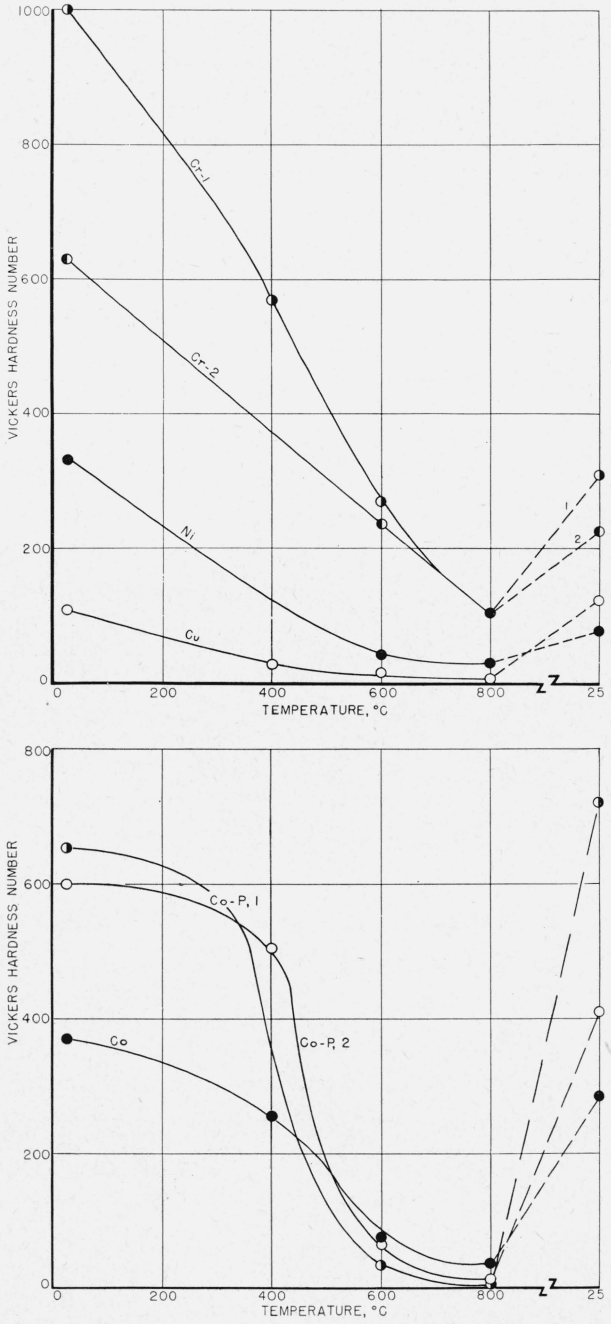

FIGURE 6. *Hot-hardness of electrodeposited chromium and nickel and bulk copper.*

Cr-1, Ordinary bright chromium, deposited from bath containing CrO_3, 250 g/liter; H_2SO_4 2.5 g/liter; current density 20 amp/dm² at 50°C. Cr-2, soft chromium deposited from same bath as Cr-1 but at 85°C and 80 amp/dm². Ni, Nickel deposited from an all-chloride bath, molar in nickel chloride and 0.5 molar in boric acid at 5 amp/dm² and a temperature of 55°C. Cu, Specimen prepared from a commercial bar of copper.

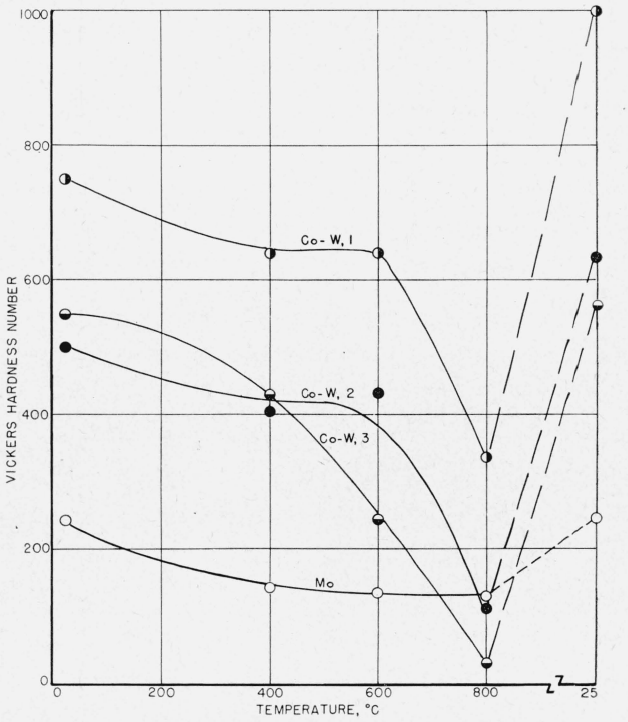

FIGURE 8. *Hot-hardness of electrodeposited cobalt-tungsten alloys and of bulk molybdenum.*

Co-W, Cobalt-tungsten alloy containing about 31 percent of tungsten; Co-W, 2, cobalt-tungsten alloy containing about 15 percent of tungsten; Co-W, 3, cobalt-tungsten alloy containing about 8 percent of tungsten; Mo, commercial specimen of molydenum bar.

FIGURE 7. *Hot-hardness of electrodeposited cobalt and cobalt-phosphorus alloys.*

Co-P, 1, Cobalt-phosphorus alloy containing about 9 percent of phosphorus; Co-p, 2, cobalt-phosphorus alloy containing about 1 percent of phosphorus; Co, cobalt deposited from a cobalt-chloride solution, molar in cobalt.

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V. References

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