

behind the second slit will record a graph of the magnified movement of the body relative to time.

The method of extracting the image reveals a further difficulty as the prismatic magnification is increased. For a given size of tetragonal prism the higher the magnification, the larger is the number of images which will have to be accommodated in half its width and hence the shorter is the length of the final slit image: this leads to difficulty in lining up the system. If the size of the tetragonal prism is increased, the final image will be further displaced from the axis of the system and will, therefore, decrease in brightness.

The prism system attached to the body is a roofed Porro, and not an ordinary Porro, in order that angular motion of the prism-system shall produce no errors in the record. Linear motion of the prism system parallel to the main optical axis will produce a slight out of focus effect, while a sideways movement,  $y$ , will produce a shift of the  $n$ th image along its own length of amount  $4ny$ .

The system was originally devised to measure the linear oscillations of one part of a body relative to another part of it, and is convenient for this work since, by increasing the focal length of the concave mirror, the distance between the prism system and the rest of the apparatus can be varied at will without affecting the record. (The relative aperture of the

concave mirror must, of course, be kept constant if the brightness of the image is to remain the same.)

Some typical results are given in Fig. 6. The roofed Porro prism system was attached rigidly to an elastic member which was caused to oscillate by a standard impulse. Records were taken of its motion using the second, third, and fourth images. The projector lens (13) was used to give a magnification of three times so that the final magnifications of the motions are  $\times 24$ ,  $\times 36$ , and  $\times 48$ . It will be noted that the width of the trace is no greater in the case of the  $\times 48$  magnification than in the case of the  $\times 24$  magnification; it does in fact appear to be thinner, this being due to the fact that the same exposure was given to all three traces in order to give an indication of the falling brightness of the images, in consequence of which the  $\times 24$  trace is over-exposed. The small breaks in the  $\times 36$  and  $\times 48$  records are due to images being lost when they fall exactly upon roof edges of the tetragonal prism. The speed of the film was again 0.73 in./sec.

The development of these methods was carried out at the Optical Laboratories of Tank Armament Research, Ministry of Supply.

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## Coppered-Tungsten Seals through Hard Glass

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**ABSTRACT.** A process is described for providing tungsten with an adherent and structurally sound sheath of copper, which flows into and fills up any cracks. This process can be used for making vacuum-tight seals through glass with split tungsten.

A difficulty often encountered in providing hard-glass apparatus with lead-through seals of tungsten is slow air leakage occurring along fine longitudinal cracks in the tungsten, and to avoid this when making seals of the orthodox type it is necessary to make quite sure that the tungsten to be used is sound and free from cracks. It has, however, been found possible to fill up any such cracks, at least to a considerable depth, with copper, and at the same time to provide the outer surface of the wire with a copper coating of any required thickness, and to do this in such a way that the copper is not only structurally sound but everywhere adheres firmly to the tungsten, making a vacuum-tight junction. It is then only necessary to seal the coppered wire into the glass apparatus, and if this is properly done there will be no possibility of leakage, however badly the tungsten core may be split. Not only so, but such seals have been found to remain vacuum-tight even after removal of the surface copper on both sides of the glass by nitric acid.

Since the copper sheathing increases the over-all thermal expansion of the wire radially, it is in general desirable to make the seal with a glass that expands somewhat more than one that would be suitable with bare tungsten. This presents no difficulty, several such glasses being available. If desired, the use of one of these glasses may be confined to the beading on the tungsten, the envelope being made of the usual C9, Nonex, or W1 glass.

The process consists of (1) mounting and cleaning, (2) preliminary plating, (3) fusion in hydrogen, (4) main plating, (5) boring, and (6) beading and sealing in. It will now be described in detail under the corresponding headings.

### MOUNTING AND CLEANING

The tungsten is first attached to a nickel handle, either by electric butt-welding or by fusion in the hottest part of a tiny

oxy-gas flame. It is then cleaned, either by deplating in caustic potash at moderate current density or by alternately heating to rather less than visible redness and plunging into a solution of sodium nitrite several times in succession. Sodium nitrite cleaning produces a smoother surface than does deplating. (Cleaning in the conventional manner with a *stick* of sodium nitrite is not recommended, as it has a tendency to widen excessively any cracks in the tungsten.) Finally, the tungsten is washed in running tap water before being transferred to the plating bath, care being taken to avoid contact with the fingers or other contamination.

### PRELIMINARY PLATING

The preliminary electro-plating may be done either with copper alone or with copper plus nickel. For copper-plating a bath consisting of 160 g. copper sulphate, 28 g. sulphuric acid and 1 l. of water is satisfactory. A suitable current density is 25–35 mA./sq.cm. If the wire is to be plated with nickel as well, it should be washed thoroughly immediately it is removed from the copper-plating bath and at once transferred to the nickelling bath. The nickel adheres well to the copper, and it is permissible for the current density used in its deposition to be about the same as in the deposition of the copper. Under these conditions the deposit formed usually appears black.

The copper or copper plus nickel should adhere to the tungsten well enough to withstand moderate rubbing with the fingers. If the coating rubs off easily an unsatisfactory condition of the tungsten surface is indicated, and the cleaning should be repeated. If the sodium nitrite cleaning method is used, particular care should be taken not to overheat the tungsten.

In carrying out the plating as described the copper will not, of course, be deposited inside the cracks. Also, the deposit neither makes a vacuum-tight junction with the underlying tungsten, nor is it, in itself, structurally sound. However, all these defects can be remedied by melting the deposit in

hydrogen. As in the manufacture of 'heavy alloy',\* the hydrogen, by keeping both the tungsten and the molten copper or copper-nickel alloy clean, acts as a flux between them, enabling the plating to 'wet' the tungsten. In consequence the junction between the two is rendered completely vacuum-tight. Also, if the fusion is carried out properly, the copper or alloy is found to flow into, and completely fill up, the cracks.

If the copper or alloy is deposited in such a way as to leave a gap between its upper edge and the nickel handle, then, on being melted in hydrogen, this will gradually creep upwards, until, finally, it reaches the nickel handle. Nickel from the handle may subsequently enrich the molten metal with nickel locally, or even, eventually, over the entire surface. This does no harm. It is, however, important not to allow such enrichment to occur too early, as it would if the plating were originally deposited over the whole surface of the tungsten, for an alloy over-rich in nickel has a high melting-point and does not readily flow into the cracks. To ensure filling of the cracks over the whole length of the tungsten the preliminary plating should stop short of the handle by a distance varying with the roughness of the tungsten surface and the nature of the plating. Copper-nickel alloy of composition, say, 10 of copper to 1 of nickel, appears to flow more readily than pure copper, and is, on the whole, to be preferred. With copper plus nickel in these proportions, and depleted tungsten, the gap between plating and nickel handle should be some 8-10 mm. long. With sodium-nitrite-cleaned tungsten it should be only 3-4 mm. If copper only is deposited the gap should be reduced to about one-third of these values.

Excess metal collects in a blob at the bottom. The formation of such a blob will be avoided if the metal deposited is only enough to give a final mean thickness of 0.13-0.16 mil. over the whole tungsten surface. On the other hand, if the preliminary plating is done too sparingly the cracks will be left open near the surface—just where it is most necessary to fill them.

#### FUSION IN HYDROGEN

To fuse the deposit, the tungsten wire may be held by its nickel handle along the axis of a vertical silica-tube furnace. Hydrogen is fed in at the bottom and burns with a small flame at the top. The silica tube must be lined internally with some suitable material, e.g. nickel sheet, to protect the silica from the harmful action of copper vapour.

To ensure proper flow of the copper or alloy into the cracks the plating should be kept molten, at a temperature of about 1130° C., for 10-15 min. Penetration tests to establish this consisted of removing the copper or alloy with nitric acid, grinding a hollow into the side of the tungsten wire where there was a crack, and finally deplating. In the deplating it is the tungsten, but not copper or copper-nickel alloy on it, that is removed, and so, presently, if the crack has been penetrated, a bright streak of copper or alloy becomes visible along it, right down into the hollow. It may easily be seen with the aid of a high-power lens.

#### MAIN PLATING

The furnace should be allowed to cool sufficiently before the wire, with its fused preliminary plating, is removed from it. If, on exposure to the air, it suffers any slight oxidation, it should be immersed for a few seconds in dilute sulphuric acid before the main copper-plating is started. Grease contamination should, of course, be avoided as before. It is a good plan to immerse the wire sufficiently far into the main plating bath for the deposit to overlap the nickel handle somewhat.

\* J. C. McLennan and C. J. Smithells, *J. Sci. Instrum.* **12**, p. 159 (1935).

For the main plating, as for the preliminary, a current density of 25-35 mA/sq.cm. is suitable. The thickness of the deposit should not be less than about 1 mil.; otherwise both copper and underlying tungsten are apt to suffer excessive oxidation in the borating and subsequent sealing operations. On the other hand, there is no point in making the deposit unduly thick. As in the case of Dumet, so also here, the radial expansion of the composite wire will be greater than the axial, which latter will (in the present case) be simply that of the core. Obviously, the greater the thickness of the deposit, the greater will be the mis-match, which will have to be taken up by plastic flow of the copper. In general, a thickness of 1-1.5 mil. will be found to give good results.

To render the plating structurally sound, and also smooth for the borating operation, it should first be sintered in hydrogen and then burnished with a rounded tool, e.g. a piece of glass tubing or rod. If difficulty is experienced in making the surface sufficiently smooth by this means, the plating may be done in two equal stages, each followed by sintering and burnishing, and in that case, of course, the first burnished surface should be scrupulously cleaned before the second stage of plating is proceeded with. This may be done by immersing for about 30 sec. in chromic acid and then washing thoroughly in running tap water. A similar cleaning is necessary after the second stage of plating as a preliminary to borating.

A satisfactory sintering has been found to result from 10 min. heating in hydrogen at about 950-1000° C.

#### BORATING

The borating operation is very simple. It is merely necessary to dip the cleaned coppered tungsten into a saturated solution of borax, and then, holding it horizontally, to heat it to bright redness from one end to the other, by passing it slowly through the neutral or slightly oxidizing part of an oxy-gas or air-gas flame. If the wire is thick, and so has a large heat capacity, it may be advantageous to quench it in water immediately after passing through the flame, and in that case it should at once be dried, as prolonged exposure to moisture is harmful to a borated copper surface. The right conditions must be found by trial, and for this purpose an ordinary copper wire, somewhat thinner than the coppered tungsten, may be used.

When cold, the borated surface should appear smoothly glazed, and of a brilliant red colour. If it is not right, the wire should be immersed in dilute sulphuric acid until it can be rubbed fairly clean with the fingers, then immersed in chromic acid, washed thoroughly, and finally reborated.

#### BEADING AND SEALING IN

The borated wire should be beaded with glass without undue delay, in any case before its surface has had time to deteriorate by exposure to the moisture of the atmosphere. The best kind of glass to use for the beading will depend on the diameter of the tungsten wire and the thickness of the copper, and it is not difficult, making what appear to be entirely reasonable assumptions, to calculate the optimum expansivity of the glass in any given case.

Let us consider a coppered-tungsten wire with a bead of glass sealed to it, such as is depicted in Fig. 1. We shall assume that both the hard tungsten core and the glass expand just as they would if entirely unconstrained, while the copper in between accommodates itself to the expansions and contractions of these two mainly by plastic flow. These assumptions will correspond closely to the truth where the glass is such that the stresses developed in it in the making of the seal

are small. The copper will not, in such a case, suffer any appreciable bulk strain, and we shall assume, for the purposes of calculation, that the bulk strain is nil. It will be further assumed that the condition for least stress is that no copper shall flow into, or be extruded from, the volume enclosed between the tungsten and the glass bead when the seal is heated or cooled. This volume is more precisely defined as that which is bounded radially by the tungsten and the glass bead and axially by the plane or conical surfaces (according to the temperature) that pass through both the ends of the bead and the circles formed by the intersections of the planes of the ends of the bead with the tungsten core when the seal is at room temperature ('cold').

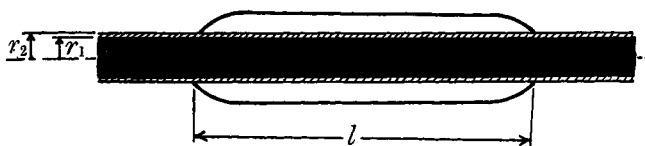


Fig. 1. Coppered tungsten wire with glass bead sealed to it

Let  $r_1$  and  $r_2$  be the radii of the tungsten core and coppered wire, respectively, and  $l$  the length of the glass bead, when the seal is cold, and let  $\alpha_1$ ,  $\alpha_2$  and  $\alpha_3$  be the mean linear expansivities of the tungsten, copper, and glass, respectively, between room temperature and the temperature at which the seal is annealed. Let us assume, in the first instance, that the seal is perfectly annealed at some annealing temperature, and then cooled, sufficiently rapidly for no appreciable further annealing to occur, to room temperature.

It would be logical, and correspond to the actual seal-making procedure, if we considered cooling from the annealing temperature to room temperature, rather than the reverse. As, however, it is easier to think in terms of heating, and it comes to exactly the same thing so far as the final result is concerned, this is done in what follows. On heating the seal from room temperature to the annealing temperature,  $t$  degrees higher, the cross-section of the tungsten core increases from  $\pi r_1^2$  to  $\pi r_1^2 (1 + 2\alpha_1 t)$ , while the internal cross-section of the bead (assumed to be free from stress) increases from  $\pi r_2^2$  to  $\pi r_2^2 (1 + 2\alpha_3 t)$ . Thus the cross-section of the copper sheath increases from  $\pi (r_2^2 - r_1^2)$  to

$$\pi (r_2^2 - r_1^2) + 2\pi t (\alpha_3 r_2^2 - \alpha_1 r_1^2).$$

The length enclosed is  $l$  when cold. Since in the cases in which we are interested the thickness of the copper coating is always small compared with the radius of the tungsten wire, the effective (or mean) length enclosed when hot may, with negligible error, be taken to be  $l \left( 1 + \frac{\alpha_1 + \alpha_3}{2} t \right)$ . Thus we find that the increase in volume of the copper sheath is

$$\pi l t [r_2^2 (\frac{1}{2}\alpha_1 + \frac{5}{2}\alpha_3) - r_1^2 (\frac{5}{2}\alpha_1 + \frac{1}{2}\alpha_3)],$$

and that the mean increase in volume per unit volume per degree rise in temperature is

$$\frac{r_2^2 (\frac{1}{2}\alpha_1 + \frac{5}{2}\alpha_3) - r_1^2 (\frac{5}{2}\alpha_1 + \frac{1}{2}\alpha_3)}{r_2^2 - r_1^2}.$$

The condition for least stress is, according to our assumptions, that this shall be equal to the cubical expansivity of the copper, i.e. to  $3\alpha_2$ . Equating these two quantities, then, and simplifying, we arrive at the final result

$$\frac{r_2^2}{r_1^2} = \frac{6\alpha_2 - 5\alpha_1 - \alpha_3}{6\alpha_2 - \alpha_1 - 5\alpha_3}, \quad (1)$$

giving the optimum thickness of copper coating to suit a predetermined glass to be used for the seal, or

$$\alpha_3 = \frac{6\alpha_2 (r_2^2 - r_1^2) + \alpha_1 (5r_1^2 - r_2^2)}{5r_2^2 - r_1^2}, \quad (2)$$

giving the optimum glass expansivity to suit a given composite wire.

In estimating optimum glass expansivities for practical sealing conditions we should, of course, make due allowance for finite rate of cooling and the difference between nominal and effective glass expansivities, and it would appear reasonable to make the same allowance here as that found necessary in the case of bare tungsten. It is found that ordinary tungsten seals made with glasses having a nominal expansivity in the region of  $0.8 \times 10^{-6}$  per  $1^\circ \text{C.}$  less than that of the metal are, when cooled at the usual rates, almost free from stress. If we make the same allowance here on the values of  $\alpha_3$  calculated according to equation (2) (with  $\alpha_1 = 4.5 \times 10^{-6}$  per  $1^\circ \text{C.}$ ,  $\alpha_2 = 17.5 \times 10^{-6}$  per  $1^\circ \text{C.}$ ), we arrive at the figures in the Table, which, each multiplied by  $10^{-6}$ , represent the estimated optimum nominal glass expansivities for a number of tungsten wire diameters and thicknesses of copper coating.

Optimum nominal glass expansivities ( $\times 10^6$ )

Thickness of copper (mil.)	Diameter of tungsten wire (mm.)					
	1	1.5	2	3	4	5
1	5.5	4.9	4.6	4.3	4.2	4.1
1.5	6.2	5.5	5.1	4.6	4.4	4.3
2	6.9	6.0	5.5	4.9	4.6	4.4

These are ideal values. Actually, thanks to the property possessed by copper of accommodating itself to considerable differences of expansion by plastic flow, a fairly wide tolerance is allowable. Thus, using 1.5 mm. tungsten wire coated with copper to thicknesses of 1, 2 and 2.3 mil., the author has made successful seals with C9 glass (expansivity  $3.75 \times 10^{-6}$  per  $1^\circ \text{C.}$ ), uranium glass ( $4.1 \times 10^{-6}$  per  $1^\circ \text{C.}$ ) and Corning 704 ( $4.9 \times 10^{-6}$  per  $1^\circ \text{C.}$ ) respectively. In all these cases the actual expansivity of the glass used was less than the calculated ideal by more than  $10^{-6}$  per  $1^\circ \text{C.}$  There is evidence, however, that where the actual expansivity falls short of the ideal by appreciably more than  $10^{-6}$  per  $1^\circ \text{C.}$  the stresses set up in the glass are rather excessive, and it seems advisable in practice to arrange for any defect to be not greater than  $10^{-6}$ . From indirect qualitative evidence obtained with seals between lead glass and Dumet from which some of the copper sheathing had been dissolved off by nitric acid, it appears that some tolerance is allowable in the other direction also, i.e. that it is permissible for the glass to have a higher expansivity than the calculated ideal. The available evidence is not sufficient, however, to indicate how much. It would appear, then, that in most cases one of the glasses that seal well with molybdenum, such as Wembley HH ( $4.6 \times 10^{-6}$  per  $1^\circ \text{C.}$ ) and the Corning glasses 704 ( $4.9 \times 10^{-6}$  per  $1^\circ \text{C.}$ ), and 706 ( $5.0 \times 10^{-6}$  per  $1^\circ \text{C.}$ ) would be suitable. Thus, in the course of the present investigation a large number of seals have been made, all of them successful, using Corning 704 glass and 1.5 mm. tungsten sheathed with copper to thicknesses between 1 and 1.5 mil. With the heaviest wires uranium glass ( $4.1 \times 10^{-6}$  per  $1^\circ \text{C.}$ ) would probably be better.

In order to avoid over-oxidation of the borated copper in the sealing process, a sleeve of glass, of internal diameter not much greater than the diameter of the prepared wire, and not too thick in the wall, should first be shrunk on. The resulting bead should be left thin-walled everywhere except at the centre, where, in the interests of mechanical strength, it may

be thickened for joining to the envelope. Stresses are always greater at the ends of a bead than elsewhere, and it is important not to aggravate these either by allowing the glass to thicken

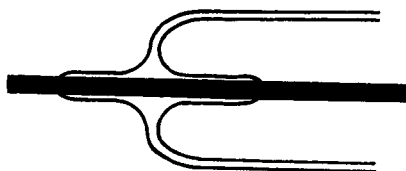


Fig. 2. A good form of seal

at the ends or (what amounts to the same thing) making the junction between bead and envelope at an end. A good form of seal is shown in section in Fig. 2.

It has been found that there is no objection to dissolving away surface copper from the unengaged ends of the wire with nitric acid after the bead has been made. Thanks to the deep penetration of the cracks by the copper, seals so treated are still completely vacuum-tight.

I am indebted to the Australian Council for Scientific and Industrial Research for leave to publish this article, which is based on work carried out in 1942 at the C.S.I.R. Radio-physics Laboratory in Sydney.

## Tests on Highly Non-magnetic Stainless Steels for use in the Construction of Weights

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**ABSTRACT.** In connexion with the use of stainless steel weights, tests have been made at the National Physical Laboratory on different types of stainless austenitic steel to ascertain to what extent they are non-magnetic. Several weights of the type (18% chromium, 8% nickel) used in this country proved to be appreciably magnetic and a few were even found to be permanently magnetized to an appreciable extent upon receipt at the N.P.L.

Some other types of austenitic steel exhibited low permeability and retentivity, and rigorous tests, including severe cold rolling, were made on three promising types. So far as non-magnetic requirements are concerned, austenitic steel is available which is far superior to the 18/8 type and is even better than much commercial brass. Details of permeability and retentivity values are given.

### 1. INTRODUCTION

Analytical weights of stainless steel have been on the market for several years. The kind of stainless steel used for their construction is one which purports to be reasonably non-magnetic, being of the austenitic variety; and up to the present time the '18/8' type of austenitic steel, viz. that containing 18% chromium and 8% nickel, has been employed for this purpose in this country. The quality of the finished weights, however, has not been very satisfactory in regard to their magnetic properties, and some have been found permanently magnetized to an appreciable extent. In view of the extended range of types of stainless austenitic steel which have become available, and the prospect that some of these might well exhibit improved non-magnetic properties, the tests described below were carried out. While no claim is made to have investigated systematically the general family of steels, it is thought that the results obtained, which relate to certain types of steel available at the time of test, will be of interest not only to users of weights but also to those who for other purposes may require a highly non-magnetic steel.

This paper relates solely to the magnetic properties of stainless austenitic steels and does not deal with other properties which are of importance in the choice of suitable material for the construction of weights, e.g. machinability, resistance to corrosion, stability of mass; nor does it purport to discuss the relative merits of stainless steel and non-ferrous alloys.

### 2. NATURE OF MAGNETIC ERRORS

It is desirable first to refer to the circumstances in which errors may be incurred in service owing to the action of magnetic forces on a weight containing magnetic material. Such forces may arise from the presence of other magnetic material forming part of the balance on which the weight is used, or from external sources. The effect on the weight as

used on the balance pan is dependent on the magnetic condition of the weight and on the characteristics of the magnetic field to which it is exposed.

If the magnetic field were uniform, e.g. the vertical component of the earth's magnetic field, it would exert no net force on the weight; but a stray field would most likely not be uniform and would therefore exert a force on a magnetic weight, whether it were in a magnetized or demagnetized condition. It can be shown that, in the conditions of weighing, the magnitude of this force is generally much greater for the former than for the latter condition. If, therefore, the weight were accidentally magnetized, as is sometimes found, the risk of error in weighing would be considerably increased.

Balances as used for weighing are by no means always entirely non-magnetic. Though the metal parts are chiefly of brass, the pan-stops and the pointer are sometimes found to be magnetic; these parts, on account of their proximity to the pans, are more particularly liable to give rise to appreciable magnetic forces on weights of a magnetic material placed on the pans. It is evidently not practicable to assess with any accuracy the magnetic field to which a weight might be exposed in service and it is therefore very difficult to indicate the order of magnitude of the magnetic errors which might be incurred in weighing. In certain circumstances attending the use of the 18/8 type of stainless steel weights, as stated later in the light of the results obtained, errors up to 0.2 mg. are not unlikely to occur in weighing and an error of 6 mg. is not impossible (see § 4).

As balances containing magnetic parts still continue to be used, it is considered advisable to stipulate that the weights themselves should be of a highly non-magnetic material, moreover, such material should have both a low permeability and a low retentivity, the latter being the more important.

### 3. MAIN STAGES OF THE TESTS

The tests were purely magnetic in character, being directed towards ascertaining which types of austenitic steel could be regarded as satisfactory for the construction of weights in spite of their potentially magnetic constituents. For each specimen examined, the magnetic permeability and retentivity were determined. In this report the permeability is expressed, as usual, in the form of a ratio, the value being unity for a perfectly non-magnetic material. The retentivity is taken to be the residual intensity of magnetization (i.e. magnetic moment per unit volume) found after magnetizing a specimen to saturation by the action of an appropriately large magnetic field. (The quantity measured is not the true