

TUBE LABORATORY MANUAL

RESEARCH LABORATORY OF ELECTRONICS

MASSACHUSETTS INSTITUTE OF TECHNOLOGY

CAMBRIDGE, MASSACHUSETTS

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1951

FOREWORD

Some of the material presented here was retained from the Tube Laboratory Handbook issued by the Radiation Laboratory, Group 52.3 in 1943. Part of the data relating to wartime production was omitted as having little more than historical interest and as being repetitious from the general viewpoint. A considerable amount of new information has been included. Some of it is from original work performed by the Tube Laboratory personnel of the Research Laboratory of Electronics.

For other data we are indebted to the individual workers and to the companies and laboratories who have given us permission to use material that we have acknowledged in the text.

The manual has been prepared as a guide for workers in the Tube Laboratory of the Research Laboratory of Electronics. References to particular commercial items are not intended to indicate superiority, but simply to indicate what materials are used here.

Fred Rosebury

Tube Laboratory
Research Laboratory of Electronics
July 15, 1951

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CHEMICAL PROCEDURES

Component parts intended for assembly into high vacuum devices must always be scrupulously clean, and this is generally accomplished in part by chemical methods. Electroplating is useful not only for protecting surfaces against destructive atmospheres but as an aid in brazing. Electrolytic treatments are used where ordinary chemical treatments are inadequate to remove heavy oxide films and other effects of corrosion.

CAUTION: The use of some of the materials named in this section is attended with certain hazards. Many of the vapors of organic solvents such as carbon tetrachloride, trichlorethylene, methyl alcohol, acetone, etc. will produce toxic effects if inhaled, and should be used with adequate ventilation. Hydrofluoric acid (handled only in hard rubber, plastic or paraffined containers) is very poisonous and irritant, as are nitric, hydrochloric, sulphuric and phosphoric acids. The usual precaution in mixing sulphuric and phosphoric acids with water should be observed: the acid should be poured into the cold water slowly with stirring in order to prevent spattering. These acids are destructive of skin and clothing. The alkalis sodium and potassium hydroxide are very irritant and destructive of skin and fabrics. Ammonia (ammonium hydroxide) should be handled only in a hood with the face protected from fumes. All the cyanides are extremely poisonous, not only when ingested but also in contact with moist skin and in very small quantities. The inhalation of cyanide fumes should be strictly avoided. Mercuric chloride (bichloride of mercury) is very poisonous and should be used with extreme caution.

The labels on chemical containers usually indicate whether the material is poisonous or not, and it therefore is imperative to read the entire label on any such container.

DEGREASING, GENERAL

1. Rinse in acetone.
2. Rinse in three changes of trichlorethylene.
3. Rinse in two changes of methyl alcohol.
4. Dry in warm air blast or oven at 70-110°C.

See J. W. Walter: Solvent Vapor Degreasing, p. 173, Metal Finishing. (Finishing Publications, Inc., New York, N. Y., 1949)

ELECTROLYTIC CLEANING OF TUNGSTEN AND MOLYBDENUM

(These methods are not to be used for cleaning heaters or filaments which for the best results should have an unetched surface. See p. 2.)

A. C-2 Process

1. The material is electrolyzed for 30 seconds or longer in a 20 percent potassium hydroxide solution, using a 7.5 volt, 200 watt transformer with variac in primary, and a carbon electrode. The solution may be used repeatedly.
2. Rinse in hot water.
3. Rinse in cold distilled water.
4. Dry in warm air blast.

B. C-5 Process for Molybdenum only

1. Electrolyze as in A above.
2. Rinse in water.
3. Place in warm concentrated inhibited hydrochloric acid 50 percent for five minutes.
4. Rinse in running water to remove loose oxide.
5. Repeat 3 and 4 until oxide removal is complete.
6. Rinse thoroughly in water.
7. Rinse in clean methyl alcohol.
8. Dry in warm air blast.

CLEANING OF TUNGSTEN AND MOLYBDENUM HEATERS AND FILAMENTS

C-19 Process

1. Boil for five minutes in 20 percent potassium hydroxide solution.

| | |
|-------|---------|
| KOH | 300 g |
| Water | 1200 ml |

2. Rinse thoroughly in tap water.
3. Rinse in cold distilled water.
4. Dry in warm air blast.

DISSOLVING MOLYBDENUM MANDREL FROM TUNGSTEN COILS*

| | |
|--|------------|
| Nitric acid HNO_3 | 50 percent |
| Sulphuric acid H_2SO_4 | 30 percent |
| Water | 20 percent |

Temperature 90° C

1. Completely cover coils with solution. Cover vessel with watch glass to prevent spattering.
2. Allow coils to remain in solution until all evidence of chemical action has stopped.
3. Pour off solution and rinse thoroughly in distilled water.
Acid solutions may be used repeatedly until exhausted.

*So-called "coiled-coil" tungsten wire as used in incandescent lamps. Comes in various sizes.

PREPARATION OF NICKEL PARTS FOR CATHODE AND FLUORESCENT COATINGS

C-1A Process

1. Agitate in acetone.
2. Boil in the following solution for five minutes:

| | |
|------------------|------------|
| Sodium carbonate | 40 g/liter |
| Sodium hydroxide | 13 g/liter |
| Sodium cyanide | 13 g/liter |
3. Boil five minutes in distilled water.
4. Rinse in warm acetic acid 5 percent.
5. Agitate in three changes of boiling distilled water.
6. Rinse in clean methyl alcohol.
7. Dry in warm air blast or oven.
8. Just previous to spraying, the work should be vacuum or hydrogen fired at 900°C.

NICKEL CLEANING BATH, ELECTROLYTIC

| | |
|-----------------|--------|
| Water | 308 ml |
| Sulphuric acid | 172 ml |
| Phosphoric acid | 546 ml |

Temperature 60°C

Connect work as anode and use sufficient current density to cause gassing of the work. Use caution as this solution rapidly attacks and dissolves nickel.

REMOVAL OF FLUXES FROM METAL PARTS AFTER SOLDERING OR BRAZING

All soft solder fluxes are more or less corrosive with time and should be carefully and thoroughly removed with appropriate solvents. The acid or chloride type of fluxes are removed by application of very hot water preferably by boiling the piece in several changes. The oil or resin type of fluxes are removed by degreasing agents; rosin may be dissolved with methyl alcohol. The grease paste type of flux (Nokorode) contains chlorides. Removal of this type of flux is accomplished by a combination of the use of degreasing agents and boiling water, in sequence.

Hard soldering fluxes generally contain borax, boric acid, and sometimes fluorides and other compounds. These should be removed after brazing by immersing the work piece in several changes of boiling water. It may also be necessary to scrub with a wire brush or clean steel wool.

REMOVAL OF HEAVY OXIDE ON COPPER

1. Immerse piece in warm 75 percent inhibited hydrochloric acid (see p. 6).

2. Rinse thoroughly with water.
3. Dry.

ELECTROLYTIC POLISHING OF COPPER

Orthophosphoric acid 50 percent in water

Anode: sheet nickel

Large anode to cathode area ratio

Heavy current density, a-c or d-c

See below for two additional electropolishing methods for copper, brass and bronze.

BRIGHT DIP FOR BRASS

| | | | | |
|----|-------------------|--------|-------------|-------------|
| A. | Sulphuric acid | 435 ml | 800 g/liter | Sp. Gr. 1.8 |
| | Nitric acid | 71 ml | 100 g/liter | Sp. Gr. 1.4 |
| | Hydrochloric acid | 2.2 ml | 2.5 g/liter | |
| | Water | 49 ml | | |

Maximum temperature 35° C

B. Electrolytic Bright Dip for Brass. (This solution is useful for removing copper and silver plating from ferrous metals.)

| | | |
|--|------------------|---------|
| | Sodium carbonate | 75 g |
| | Sodium cyanide | 75 g |
| | Water | 1 liter |

Use solution hot. Voltage 6-12

Anode: sheet iron or steel

C. Two Methods for the Electrolytic Polishing of Copper, Brass, Bronze (Metal Finishing; Finishing Publications, Inc., New York, 1949)

| | | |
|----|-----------------|--------------------|
| 1. | Phosphoric acid | 63 percent by vol. |
| | Water | 37 percent by vol. |

Current density 0.02-0.05 amp/in²

Temperature 20° C

Time 5 minutes

| | | |
|----|-----------------|--------------------|
| 2. | Phosphoric acid | 58 percent by vol. |
| | Water | 42 percent by vol. |

Current density 0.5-0.7 amp/in²

Temperature 20° C

Time 10-15 minutes

Cathodes are unimportant from the work point of view as they are used only to complete the circuit because the work in this case is made the anode. Therefore a material is desired which will stand up in the solution. Generally cathodes can be

made of copper, lead, or in some cases, steel. Water-cooled copper tubing finds use when it is necessary to cool the baths in order to keep them at operating temperature. In some cases it is necessary to use cathodes that conform to the shape of the work, especially in the case of deep-drawn pieces.

CLEANING OF OXIDIZED IRON

1. Place material in warm 50 percent inhibited hydrochloric acid for approximately 5 minutes to remove oxides.
2. Rinse thoroughly in water.
3. Dry.

HYDROCHLORIC ACID DIP FOR STEEL, KOVAR, NICKEL

Hydrochloric acid (concentrated)
20 percent of standard 37 percent

PICKLE FOR CAST IRON

| | | | | |
|----|-------------------|--------|---|--|
| A. | Sulphuric acid | 1 pint | } | in hard rubber or paraffined glass container |
| | Hydrofluoric acid | 1 pint | | |
| | Water | 1 gal | | |

Use at room temperature or higher

| | | | |
|----|----------------|-------|-----------|
| B. | Sulphuric acid | 12 oz | by weight |
| | Nitric acid | 5 oz | by weight |
| | Zinc | 1 oz | by weight |
| | Water | 1 gal | |

PICKLES FOR IRON AND STEEL

- | | | |
|----|---|-----------|
| A. | Inhibited hydrochloric acid solution (see p. 6) | |
| | Sulphuric acid | 1/2 pint |
| | Water | 1 gal |
| | Temperature 20° - 79°C | |
| B. | Sulphuric acid | 3 oz/gal |
| | Potassium nitrate | 3 oz/gal |
| | Temperature 71° C | |
| C. | Citric acid | 10 oz/gal |

Ammonia is added until the solution is alkaline to litmus. Use at boiling point to remove rust without any measurable attack on the metal.

- | | | |
|----|-----------------|---------------|
| D. | Phosphoric acid | 10-15 percent |
|----|-----------------|---------------|

Water 90-85 percent
Temperature 79° -85 °C

INHIBITED HYDROCHLORIC ACID

Solutions are made up according to strength specified. For common pickling of iron and steel, fifty percent is used. The inhibitor is Rodine No. 50* and is added to the acid in the proportion of 1/4 percent by volume.

CLEANING SOLUTIONS FOR GLASS AND TANTALUM

- A. Potassium dichromate $K_2Cr_2O_7$ sat. sol. 35 ml
Sulphuric acid H_2SO_4 conc. 1 liter
- B. (Preferred because of absence of alkaline salts likely to remain in crevices)
Chromium trioxide CrO_3 chromic sat. sol. in hot conc. sulphuric acid.

Pour the acid slowly with stirring into the chromate or trioxide solution.

Use both solutions at 110°C. Solution should be red in color for best results. If muddy or greenish, solution should be discarded.

PICKLING OF MONEL

Sulphuric acid 1 pt/gal
Sodium nitrate 3/4 lb/gal
Sodium chloride 3/4 lb/gal
Temperature 82° -88 °C

PICKLING OF INCONEL

- A. Water 2 gal
Nitric acid 1 gal
Hydrofluoric acid 1 pint
Temperature 66° -74 °C
- B. Sulphuric acid 13 oz/gal
Rochelle salt 13 oz/gal
Temperature 71° -82 °C

PICKLING OF STAINLESS STEEL

Since the scales which form on stainless steel are very resistant to acid, it is often advisable to pickle in two operations, the first for loosening the scale and the second for removal, or for brightening the surface. Where only light scales are present, the scale loosening treatment may be omitted. Suitable inhibitors should be added to minimize pitting of the metal.

*American Paint Company, Ambler, Pa.

LOOSENING SCALE ON STAINLESS STEEL (All acids specified are concentrated)

| | | | |
|----|-------------------|------------|--------|
| A. | Sulphuric acid | 12 | oz/gal |
| | Temperature | 85° C | |
| B. | Sulphuric acid | 1 | gal |
| | Hydrochloric acid | 1 | gal |
| | Water | 8 | gal |
| | Temperature | 55° -60 °C | |

REMOVING SCALE ON STAINLESS STEEL

| | | | |
|----|-------------------|-----------------|--------|
| A. | Nitric acid | 1 | gal |
| | Hydrofluoric acid | 1 1/2 | gal |
| | Water | 2 1/2 | gal |
| | Temperature | 50° -65 °C | |
| B. | Sulphuric acid | 1/2 | pt/gal |
| | Hydrofluoric acid | 1/2 | pt/gal |
| | Chromic acid | 8 | oz/gal |
| | Temperature | 20° C or higher | |
| C. | Nitric acid | 1 | gal |
| | Hydrofluoric acid | 1 | qt |
| | Hydrochloric acid | 1 | pt |
| | Water | 9 | gal |
| | Temperature | 55° -60 °C | |
| D. | Hydrochloric acid | 5 | gal |
| | Nitric acid | 1 | gal |
| | Water | 14 | gal |
| | Temperature | 50° -70 °C | |

PASSIVATION SOLUTIONS FOR STAINLESS STEEL

Passivation or immunization should be employed after any pickling operation on stainless steel. Effectiveness of this treatment may be tested by application of copper sulphate solution, slightly acidified. No copper will be deposited on passivated surfaces.

| | | | |
|----|-------------|------------|-----|
| A. | Nitric acid | 1 | gal |
| | Water | 4 | gal |
| | Temperature | 50° C | |
| | Time | 20 minutes | |
| B. | Nitric acid | 1 | gal |
| | Water | 1 | gal |
| | Temperature | 20° C | |
| | Time | 1-2 hours | |

ELECTROLYTIC POLISHING OF STAINLESS STEEL (Metal Finishing; Finishing Publications, Inc., N. Y., 1949) (see p. 4 for remarks on cathodes) (All acids specified are concentrated)

A. Phosphoric acid 100-75 percent
 Water 0-25 percent

Current density 1-20 amp/in²

Temperature 40°-95°C

Time 5 minutes

B. Phosphoric acid 60-65 percent
 Sulphuric acid 15-20 percent
 Water 20-22 percent

Current density 0.35-5 amp/in²

Temperature 50°-95°C

Time 3-60 minutes

C. Phosphoric acid 30 percent
 Sulphuric acid 60 percent
 Water 10 percent

Current density 1.8 amp/in²

Temperature 50°C

Time 2 minutes

D. Phosphoric acid 45 percent
 Sulphuric acid 40 percent
 Water 15 percent

Current density 0.5-3.5 amp/in²

Temperature 30°-150°C

Time 5 minutes

E. Phosphoric acid 15 percent
 Sulphuric acid 60 percent
 Chromic acid 10 percent
 Water 15 percent

Current density 4 amp/in²

Temperature 50°C

Time 30 minutes

F. Phosphoric acid 44 percent
 Sulphuric acid 40 percent
 Chromic acid 6 percent
 Water 10 percent

Current density 3 amp/in²

Temperature 50°C

Time 60 minutes

- G. Sulphuric acid 60 percent
 Water 40 percent
 Current density 14 amp/in²
 Temperature 75°C
 Time 2 minutes
- H. Sulphuric acid 15-20 percent
 Citric acid 55-60 percent
 Water 25-30 percent
 Current density 2.5-40 amp/in²
 Temperature 50°-120°C
 Time 1/2-5 minutes
- I. Sulphuric acid 55 percent
 Hydrofluoric acid 7 percent
 Water 38 percent
 Current density 1/2-2 amp/in²
 Temperature 20°C
 Time 4-60 minutes

In some cases agitation is required in order to eliminate gas streaking.

ELECTROPLATING, GENERAL

Electroplating procedures have been chosen which seem most adaptable to laboratory scale. Large articles or those requiring heavy deposits should be handled by commercial plating companies. Information on other types of baths and special procedures may be found in The Plating and Finishing Guidebook published annually by the Metal Industry Publishing Company (The Journal of Metal Finishing). See also Chemical Abstracts.

The cleaning process and preparation of metal surfaces for plating are extremely important for proper adhesion and a good plate. In general the workpiece and the anode should have about equal size (see notes on cleaning and also section on Stainless Steel).

Drying of electroplated articles: To avoid thin tarnish films on plated surfaces (especially important in silver plating), as soon as the work is plated, rinse in hot running water, dip into clean alcohol, and dry in hot air.

It is generally considered desirable when applying more than one deposit on a given material to electroplate in a certain specified order:

STEEL: In the case of ordinary steels, the first deposit should be copper. This may be followed by either silver, nickel or gold, depending on requirements (see COPPER PLATING IRON AND STEEL, p. 11).

COPPER may be directly plated with gold, silver or nickel.

KOVAR may be silver plated directly, although for best results it should have an initial copper flash or strike.

BRASS may be silver, gold or nickel plated directly; however an initial copper strike is desirable.

STAINLESS STEEL and other high chrome alloys. (See special section devoted to this subject, p. 15.)

CHROMIUM plating is carried out in the order of copper, nickel, then chromium.

MOLYBDENUM: Best results are attained when this metal is first cleaned and nickel plated by methods similar to those described for stainless steel. (See special section, p. 15.)

1. Clean the work electrolytically in a 20-40 percent potassium hydroxide bath with a current density of 9 amp/ft² for one minute. Use d-c with a carbon or steel cathode. Rinse well in running water.

2. Nickel plate in Watts nickel bath (see below and section on stainless steel, p. 15) but reverse current, making the nickel anode the cathode. Current density is 3.6 amp/ft² for 3 to 4 minutes at room temperature.

3. Plate in usual manner (reverse of step 2) with current density of 11 amp/ft².

TUNGSTEN may be nickel plated directly, or if plated with copper, it must be hydrogen fired to produce good adhesion.

1. Clean electrolytically in a 20-40 percent potassium hydroxide bath using a-c and a carbon electrode in usual manner (p. 1).

2. Rinse in running water at least 3 minutes.

3. Nickel plate in Watts nickel bath (see section on stainless steel p. 15) at room temperature and very low current density. Plate for 20-30 minutes.

USE OF STOP-OFF LACQUER

This is a nitrocellulose-vehicle lacquer usually with a red dye added as identification. It is to be applied to the cleaned work-piece by dipping, spraying or brushing. The lacquer is removed after plating by immersion in ethyl acetate or acetone.

NICKEL PLATING

WATTS NICKEL BATH for Stainless Steel, Molybdenum, Tungsten (see section on stainless steel for procedure, p. 15).

Nickel sulphate $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$ 200 g/liter

Nickel chloride $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ 45 g/liter

Boric acid H_3BO_3 30 g/liter

Current density 25 amp/ft²

pH (electrometric) 2-5

Temperature 38°-42°C (may be operated at room temperature with slowing up of deposition)

This bath gives a matte finish and is to be preferred for the brazing and heat treating of chromium alloys (see p. 15).

Metal Finishing; Finishing Publications, Inc., N. Y., 1949; p. 274, gives the following data on WATTS type bright nickel bath.

| | | oz/gal | g/liter |
|--|---|--------------------|---------------------------|
| Nickel sulphate hexahydrate | $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ | 30-50 | 225-375 |
| Nickel chloride hexahydrate | $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ | 4-8 | 30-60 |
| Boric Acid | H_3BO_3 | 4-6 | 30-45 |
| Temperature | 49°-71°C | pH (electrometric) | 2-5 |
| Surface tension (as produced by a wetting agent) | | | 30-38 dynes/cm |
| Current density | | | 20-60 amp/ft ² |

Brighteners as specified by vendors

For further remarks on nickel plating, see "Practical Nickel Plating" by Dr. W. A. Wesley and W. H. Prine; The International Nickel Co., N. Y., 1949.

COPPER PLATING IRON AND STEEL (See also section on stainless steel, p. 15.)

1. Degrease in acetone-trichlorethylene-alcohol baths.
2. Electrolyze in 20-40 percent potassium hydroxide bath in usual manner (a-c with carbon electrode). (See p. 1.)
3. Rinse in running water.
4. If rust is present, pickle in warm 7 percent hydrochloric acid.
5. Rinse in running water.
6. Electrolyze in following bath:

| | |
|------------------|------------|
| Copper cyanide | 20 g/liter |
| Sodium cyanide | 60 g/liter |
| Sodium carbonate | 30 g/liter |

7. Rinse well. If necessary to paint with stop-off lacquer, first rinse in methanol and dry.

8. Copper plate in following bath:

| | |
|--|--------------|
| Copper cyanide | 12.2 g/liter |
| Sodium cyanide | 16.1 g/liter |
| Sodium carbonate | 15.2 g/liter |
| Potassium sodium tartrate (Rochelle salt) | 15.2 g/liter |

Bath is normally used at 60°-70°C, but may be used at room temperature with slower deposition.

Current density is 23 amp/ft². When bath is used at 65°C approximately 0.001" will be deposited per hour.

9. Peel off lacquer with alternate hot and cold water.
10. Rinse well in running water.
11. Rinse in methanol, dry in warm air blast.

SILVER PLATING

SILVER STRIKE

| | |
|-------------------|------------------------|
| Silver cyanide | 6.5 g/liter |
| Potassium cyanide | 68.0 g/liter |
| Current density | 15 amp/ft ² |

Work should be connected to current source before immersion in bath.

The strike solution has a low silver content and a high cyanide concentration. It is used only to obtain the first covering of silver; thickness is to be built up in the regular silver plating bath. The strike solution will prevent the deposition of silver by immersion which results in poor adhesion of the ultimate deposit. The work is kept in the strike just long enough to coat the surface.

REGULAR SILVER PLATING SOLUTION

| | |
|---------------------|--------------------------|
| Silver cyanide | 41 g/liter |
| Potassium cyanide | 40 g/liter |
| Potassium hydroxide | 11 g/liter |
| Potassium carbonate | 62 g/liter |
| Current density | 5-15 amp/ft ² |

Yield is 0.001" an hour at bath temperature of 48°C but may be operated at room temperature with reduced thickness of deposition.

Anode should be 0.9995 "fine" (pure) silver. Sterling is NOT suitable.

Remove anode from plating bath when not in use so as to prevent excessive solution of the metal in the bath.

GOLD PLATING

GOLD PLATING SOLUTION

| | |
|------------------------|--------------------------|
| Potassium gold cyanide | 3.9 g/liter |
| Potassium carbonate | 7.9 g/liter |
| Potassium cyanide | 3.9 g/liter |
| Current density | 5-15 amp/ft ² |

Remove anode from bath when not in use to prevent excessive solution of the metal.

Yield is 0.001" an hour at temperature of 50°-70°C. May be operated at room temperature with reduced yields.

PLATINUM PLATING SOLUTION (Baker and Company, U.S. Pats. 1,779,457 and 1,779,436)

| | |
|------------------|-------|
| Ammonium nitrate | 100 g |
| Sodium nitrate | 10 g |

| | |
|---|---------|
| Pt as diammino nitrite (platinum P-salt) | 10 g |
| Ammonia | 50 cc |
| Water | 1 liter |

Voltage 4.5

Current density 55-120 amp/ft² or 6-13 amp/dm²

Temperature 95°C

Covers quickly with fair throwing power. For the building-up of heavier deposits use 2.4 volts and 55 amp/ft² (6 amp/dm²).

Replenish bath with the platinum P-salt. Avoid contamination with base metals.

METAL COLORING (From Metal Finishing; Finishing Publications, Inc., New York, 1949; p. 352.)

BLACK COLORING ON BRASS

| | |
|------------------|--------|
| Copper carbonate | 1/2 oz |
| Aqua ammonia | 4 oz |
| Soda ash | 1/4 oz |
| Water | 1 gal |

Temperature 90°C

The copper carbonate should be as free from sulphate as possible. Freshly precipitated well-washed copper carbonate is preferable. The black color is more permanent on 80 percent Cu - 20 percent Zn than on common high brass.

BLUE COLORING ON BRASS

| | |
|----------------------|--------|
| Lead acetate | 2-4 oz |
| Sodium thiosulphate | 8 oz |
| Acetic acid, glacial | 4 oz |
| Water | 1 gal |

Temperature 80°C

This will produce a blue color on a nickel deposit and also on polished high carbon steel. The color will change if not lacquered.

DURABLE BROWN GUN BARREL FINISH FOR STEEL

| | |
|--------------------|---------|
| Copper sulphate | 20 g |
| Mercuric chloride | 5 g |
| Ferric chloride | 30 g |
| Nitric acid, conc. | 150 g |
| Denatured alcohol | 700 ml |
| Water to make | 1 liter |

1. Dip parts in above solution until thoroughly wetted. The solution can also be brushed or sponged on.

2. Place parts in hot-air oven for 30 minutes at 80°C.
3. Transfer parts to humidity (steam) cabinet at 65°C*. Relative humidity 55 percent. Wet bulb 54°C.
4. Turn off steam and permit parts to remain in cabinet until wet bulb reading has fallen to 38°C. A ferric oxide coating will be formed on the parts.
5. Put parts in boiling water until red rust is converted to black oxide.
6. Dry and scratch brush with a steel wheel.
7. Repeat operations 1 - 6 three times.
8. Oil with an oxidizing oil, such as linseed.

NOTE: The steel must be absolutely clean and free from coloring before beginning the process.

Stainless steel can be given the browning finish if it is first plated for about 20 minutes in a ferric chloride solution.

BLUE ON STEEL

| | | |
|----|---------------------|----------|
| A. | Sodium hyposulphite | 8 oz/gal |
| | Lead acetate | 2 oz/gal |

Use boiling

| | | |
|----|---------------------------|------|
| B. | Ferric chloride | 2 oz |
| | Mercuric nitrate | 2 oz |
| | Hydrochloric acid, conc. | 2 oz |
| | Alcohol, ethyl 95 percent | 8 oz |
| | Water | 8 oz |

Room temperature

1. Parts are immersed for 20 minutes, removed and allowed to stand in air for 12 hours.
2. Repeat, then boil in water for one hour.
3. Dry, scratch brush and oil.

| | | |
|----|---------------------------|-------|
| C. | Mercuric chloride | Parts |
| | Potassium chlorate | 4 |
| | Alcohol, ethyl 95 percent | 3 |
| | Water | 8 |
| | | 85 |

Use at room temperature

*A humidity cabinet is not absolutely necessary. A steel tank with a cover can be used and live steam introduced into the tank. It is important to get a good coverage of red rust, otherwise the finish will be inferior. A patchy finish will also result if the previous oxidized oil finish is not completely removed.

BRAZING OF STAINLESS STEELS AND OTHER ALLOYS CONTAINING CHROMIUM and/or NICKEL, COBALT. (From material furnished by A. Korbela and E. Okress, Engineering Department of Westinghouse Electric Corporation, Bloomfield, N. J., 1950).

Some of the nonmagnetic stainless steel alloys (e. g. types 304, 347) are very useful in making electronic tube parts. The presence of chromium in these alloys, however, makes ordinary hydrogen furnace brazing difficult because of the formation of oxides (1) owing to the moisture content of tank hydrogen. Any moisture in excess of one part in 7400 of hydrogen will oxidize metallic chromium at 895°C (2). In practice a dewpoint of -40°C and lower in a tight, nonleaky furnace has been found necessary to prevent oxide formation and to allow good brazed joints at higher temperatures with copper, gold-nickel and copper-nickel brazing materials.

To braze below 950°C a protective electrodeposited coating such as nickel has been found very satisfactory. Such a coating is necessary when brazing types 304, 307 etc., stainless alloy steels with silver and gold brazing alloys such as silver-copper eutectic (BT), Silfos and gold-copper materials.

Probably the most difficult metals to plate are those containing amounts of chromium and/or nickel, cobalt. Microscopic amounts of these alloying components either render the material completely passive or result in a loosely bonded electroplate which is likely to peel and blister. Two methods for overcoming this difficulty have been used rather successfully (see p. 25).

Anodic etching in a sulphuric acid bath followed by a direct transfer to an acid copper (sulphate) electrolyte.

The preferred method is an activation or "strike" in a chloride bath of the following composition.

| | | |
|--------------------------|---|--------|
| Nickelous chloride | $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ | 32 oz |
| Hydrochloric acid, conc. | | 500 cc |
| Water | to make one gallon | |

PROCEDURE FOR PLATING

1. The stainless alloy is first cleaned to remove surface contaminants. In the case of spinnings, contamination extends beneath the surface due to lapping or burying of amounts of spinning compounds, etc. The elimination of subsurface inclusions is best carried out by an electrolytic etch in 56°Be sulphuric acid (Assay 96.4 H_2SO_4 - 42 1/2 parts, water 57 1/2 parts), buffered by additions of boric acid to saturation. This etch is used at room temperature (21°C - 27°C), with the part being cleaned made the anode. Hard (commercial) lead or lead alloy cathodes with an area at least

(1) Chromium oxide is reduced to metallic chromium in vacuum at about 1000°C.

(2) "Scientific Foundations of Vacuum Technique" by S. Dushman; John Wiley and Sons, Inc., 1949; pp. 820-821.

equal to that of the anode are used. Anode to cathode spacing should be such as to insure uniform current distribution. At least twice the thickness of the article being treated should be allowed for such spacing, i. e. a piece of 2" pipe being treated should have a minimum spacing of 4" from the edge of the anode to the facing edge of the cathode. This electrolytic pickling should be long enough to remove completely all subsurface contaminants. Treatment times of 2 to 3 minutes have been found satisfactory on most parts. Badly contaminated pieces take considerably longer, and may even require mechanical scrubbing action to loosen the spinning compounds which tend to protect, through their nonconductivity, the underlying metal.

2. After electropickling, the parts are thoroughly rinsed in clean running water.

3. A rinse in 20 percent hydrochloric acid solution at room temperature followed by a clean water rinse.

4. Electroplate in the chloride bath given on p. 15. The activation of the alloy surface is apparently effected through the bombardment of the metal with atomic hydrogen - with a tightly bonded nickel film being deposited at the same time. The strike should be carried out for one minute at 6 volts d-c. (approximately 150-200 amp/ft² of cathode or work surface) at room temperature. The solution should be electrolytically purified and filtered to remove copper and iron contamination and then filtered to remove precipitates and other foreign materials. The electro-purification is performed by plating at a low current density (2-4 amp/ft²) using sheet cathodes of as large a surface area as possible. Frequent agitation of the solution during purification is advisable. The presence of copper and iron as bath impurities is indicated by a dark deposit under normal plating conditions.

5. Rinse in clean running water.

6. Transfer to the standard Watts nickel solution and plate to the required thickness. The Watts solution is made up as follows:

| | | |
|-----------------|---------------------------------------|-------|
| Nickel sulphate | NiSO ₄ · 7H ₂ O | 40 oz |
| Nickel chloride | NiCl ₂ · 6H ₂ O | 8 oz |
| Boric acid | H ₃ BO ₃ | 6 oz |
| Water | to make one gallon | |

The nickel salts should be dissolved in hot water (65°C) leaving out the boric acid which is added later. For each gallon of solution add 1 oz of nickel carbonate, made into a paste with hot water, and stir into the bath. After about one-half hour of stirring at 65°C, take a sample, filter, cool and measure the pH. If it is not 5.8 (electrometric), continue stirring until this value is reached. More nickel carbonate may be required. When the proper pH is reached, to the hot solution add 5 cc for each gallon of solution of 100 volume hydrogen peroxide (acid stabilized peroxide is preferred to the organic inhibited material). Continue stirring the solution for at least 4 hours after the addition of the peroxide. Allow the electrolyte to settle overnight and filter into a clean plating cell. Heat to 50°C, add 2 oz per gallon of activated carbon Darco S-51 (Darco

Corp., 60 E. 40th St., New York 17, N. Y.)* After the carbon is added the solution is stirred for about 30 minutes at 50°C and then filtered. The right amount of clean boric acid is then added to the solution, the pH measured, and if above 4.0, careful additions of sulphuric acid are made to adjust to that value. Operating temperature of the bath over a range of 50°-60° C at a current density of 30 amp/ft² will produce good, soft, ductile deposits of nickel. Hydrogen pitting may occur on some work pieces due to their contours; this can be overcome by the daily addition of hydrogen peroxide in the amount of 0.057 oz of 30 percent (100 vol) H₂O₂ per liter of electrolyte. Peroxide stabilized with organic agents such as acetanilide must not be used. An alternative method of pitting control is through the use of a wetting agent such as Duponal C, a grade certified by analysis to be free from arsenic and lead. Concentration used is between 0.5 and 0.75 grams per liter of plating solution. The plating solution is approximately 97 percent efficient and will plate at the rate of 0.0001" per minute at 125 amp/ft² at the temperature given. For example, at 25 amp/ft² every five minutes of plating will give a deposit of 0.0001". Generally, as a safety factor, to compensate for errors in computing surface area, etc., a factor of 10 percent is added to the above figure so that in actual practice 5 1/2 minutes of plating time would be allowed for each 0.0001" of plate thickness required when using a current density of 25 amp/ft².

For protection against oxide film formations on chromium alloys during treatment at elevated temperatures in hydrogen, the thickness of the electrodeposit required will vary directly with the temperature and the treating time. Thicker coatings will be required for higher temperatures and longer firing time. The detailed data are tabulated elsewhere in this section. It must be remembered that thin spots, such as will occur at the contact point areas, can only be prevented by shifting the work on the plating rack. A good practice is to shift the contact points when half the desired thickness is reached. Care should be taken to avoid finger print staining of the freshly plated surface by handling the work with clean rubber gloves. Reactivation of the work after handling is a good safety measure to observe.

7. After nickel plating, the parts are thoroughly rinsed and flash copper plated - 90 seconds at 30 amp/ft² - in a Rochelle salt-copper cyanide plating bath (see section under copper plating).

8. Thorough water rinses followed by a dip in a 5-10 percent sulphuric acid solution at room temperature, with another water rinse, assure a cyanide-free surface.

9. The parts are dried by any one of several suitable methods:

- a. boiling water rinse and air drying.
- b. hot water rinse, alcohol and forced air drying.
- c. alcohol rinse, acetone rinse and air drying.

*For detailed use of activated carbon in electroplating see W. A. Helbig, Metal Industry, December 1938; and W. A. Helbig, Proc. Am. Electroplaters Soc., 1941.

References:

- Modern Electroplating, Electrochemical Society; Finishing Publications, Inc., New York, 1949.
Trans. Electrochemical Soc. Vol 80.
Practical Nickel Plating, International Nickel Co., 67 Wall St., New York, 1949.

NOTES

For successive brazing operations above 900°C it was found necessary, on the basis of previous results on samples, to plate the nickel in thicknesses from 0.00075" to 0.001". The chromium in the stainless steel alloy is thus protected against the atmosphere in the furnaces.

PLATING DATA vs. FIRING TEMPERATURES AND TIME

The details of the results of the nickel plating experiments are as follows. Plating was done at a current density of 25 amp/ft².

| Nickel thickness inches | Hydrogen firing Temperature °C | Condition after firing | |
|----------------------------|-----------------------------------|------------------------|------------|
| | | 30 minutes | 60 minutes |
| 0.00025 | 900 | unsatisfactory | |
| 0.00025 | 1000 | poor | |
| 0.00025 | 1100 | bad | |
| 0.00025 | 1200 | very bad | |
| 0.0005 | 900 | good | doubtful |
| 0.0005 | 1000 | good | doubtful |
| 0.0005 | 1100 | doubtful | poor |
| 0.0005 | 1200 | poor | bad |
| 0.00075 | 900 | good | good |
| 0.00075 | 1000 | good | good |
| 0.00075 | 1100 | good | good |
| 0.00075 | 1200 | good | doubtful |
| 0.001 | 900 | good | good |
| 0.001 | 1000 | good | good |
| 0.001 | 1100 | good | good |
| 0.001 | 1200 | good | good |

Metallurgical examination has shown that the nickel at the interface begins to alloy with the base metal at 900°C producing a very tenacious and protecting surface. However, if the alloying process proceeds too far as a result of long time or high temperature, the nickel diffuses into the steel and no longer protects the chrome content portion of the stainless steel surface.

HEAT TREATING AND BRAZING

HYDROGEN BRAZING, GENERAL

Controlled atmosphere brazing and heat treating offer certain very definite advantages over other methods of performing these operations. In working with copper, nickel, monel, Kovar, silver and other metals and alloys the work generally comes out bright and clean, and usually stress-relieved as a result of uniform heating. When due consideration is given the design of surfaces at joints, dimensional tolerances of fits, disposition of the solder, jiggling or otherwise holding the parts to be brazed together and also the time and temperature cycles, no special skill is required to produce clean brazed joints with good vacuum and mechanical properties, and a high degree of uniformity. All materials to be brazed should be degreased and chemically clean, and should not be handled with the bare hands prior to firing. The secretions of the skin contain, in addition to oils, inorganic salts which are difficult to remove, and which cause staining of the work piece through the action of the heat.

Various materials, including those mentioned above, may be joined. Under certain conditions, tungsten, molybdenum, stainless and other steels and chrome-bearing alloys, as well as various ceramics and other nonmetallic substances, may be successfully brazed.

COPPER: Ordinary electrolytic copper may be satisfactorily brazed in a hydrogen atmosphere if porosity and lowered mechanical strength may be tolerated. These effects are caused by the reduction of the oxide particles always present in ordinary copper. Water vapor is formed in the intergranular spaces and the pressure due to expansion of the gas during heat treatment tends to rupture the metal. Articles made of electrolytic copper and subject to a hydrogen atmosphere at high temperatures are therefore not suitable for vacuum work. Where nonporosity and ductility are rigidly required, OFHC (oxygen-free, high conductivity) copper must be used. The most commonly used solder for copper is the silver-copper eutectic alloy, 72 percent silver, 28 percent copper (Handy and Harman Company "BT") which has a sharp melting point at 779°C. This material will enter into diffusion with the copper parent metal during brazing operations, resulting in alloys of different composition and higher melting point. An examination of the equilibrium diagram for silver-copper alloys, to be found in any metallurgy text, will illustrate this graphically. Advantage may be taken of this effect where two or even three brazing operations on the same piece are called for. If the first operation is done at some temperature above 779°C a considerable part of the joint will contain copper in excess of the 28 percent eutectic value, and will have a melting point approaching as a limit the temperature at which the operation was done. If then the second brazing operation is done at a temperature between 779°C and that of the first operation, the metal in the joint formed in the first operation will not flow. In this serial type of brazing the temperature must of course be accurately controlled; the more serial operations the more accuracy is

required.

Copper may also be joined by means of other solders in the hydrogen furnace. Two requirements must be met in normal operation: the solder should have no ingredients which will vaporize, such as lead, zinc, cadmium (the use of solders containing such volatile constituents is treated elsewhere in this section); and it should have a flow point safely (100°) below the melting point of the copper itself. These requirements are met by the silver-copper eutectic mentioned above, by other alloys comprising silver and copper, by a gold-copper alloy containing 80 percent gold, 20 percent copper, m. p. 890°C , by a gold-nickel eutectic, (Au 82.5 percent, Ni 17.5 percent, m. p. 950°C), by pure silver (m. p. 960°C) and by several solders containing varying amounts of gold and copper. Pure gold may also be used, sometimes advantageously by the "diffusion" method, in which the parts to be joined must be accurately machined and held together under pressure with the gold as foil, wire, or electroplate, making intimate contact. In this method the gold will diffuse into the copper if it is held for a long enough time under pressure, at a temperature below the melting point of the former. (Temperatures as low as 500°C have been used.)

A very successful method of joining copper parts is by the use of electro-deposited silver. Where joints can be made with good contact over the surfaces to be bonded, by press or close mechanical fit with allowance for plating thickness, the same procedure as for the silver-copper eutectic may be followed. The interface of the silver-copper is in some places a thin layer of eutectic composition. This will melt first and more silver and copper will dissolve in the liquid phase at 779°C as long as there is enough silver to maintain the eutectic composition. Higher temperatures and longer periods will result in higher melting point silver-copper alloys in the same manner as though the process were initiated with the eutectic. By judicious use of silver plate highly satisfactory vacuum tight joints can be made. The silver plating may, if necessary, be limited to definite areas by the use of stop-off lacquer during the plating process. In brazing, the silver may be prevented from flowing to surfaces where its presence is undesirable by coating these surfaces with aluminum oxide in a nitrocellulose binder. This is the same material which is used to coat tungsten heaters for vacuum tubes. The binder is burned off harmlessly during the firing and the aluminum may be brushed off after the work has cooled, since the soldering temperatures are not high enough to "set" the ceramic material.

In designing joints for controlled atmosphere brazing, consideration must be given to capillary and gravity flow. Since the solder in its molten condition will act like any other liquid, and since materials such as copper in a hydrogen atmosphere present a very "clean", i. e. wettable, surface, the solder will flow up into small cracks and gaps and even upward along exposed surfaces. The precautions to be taken are therefore: avoidance of the use of excessive solder or silver plate, stopping-off, and positioning of work in the furnace so that gravity may work to advantage.

Where the use of a jig is required in brazing copper or other metals in the

hydrogen furnace, it is common to make those parts of the jig which might, by being in contact with or close to the work and the solder, otherwise be brazed fast to the work, of stainless steel (No. 303) or of nichrome. The parts of the jig are to be prefired in tank hydrogen to produce a protective coating of green chromium oxide, to which the solder will not adhere as this oxide is very tenacious and not reducible at brazing temperatures.

STEEL, cold-rolled, hot-rolled and carbon steels: Many of the remarks about copper apply to other materials, especially in respect to design, machining and fit of parts to be joined. Chrome-free steels may be brazed with the solders mentioned for copper. One method for brazing steel to steel is that of copper brazing in which pure copper is used as a solder (m. p. 1083° C). The steel should be previously nickel and/or copper plated. Since neither copper nor silver diffuse to any extent into iron the remarks on diffusion of silver into copper do not apply. Considerable success in H₂ brazing steel with silver solders has been attained by a partial purification of the hydrogen. The gas is passed through one or more "De-Oxo" (Baker and Company) catalytic purifiers, which reduce any oxygen present to water vapor, the removal of most of which can be accomplished with a drying tube and a liquid nitrogen trap. *

The use of solders other than pure copper will be indicated by the kind of metals to be joined to the steel, copper brazing being obviously unsuitable for joining steel to copper.

NICKEL, MONEL and related alloys except those containing chromium and aluminum (see p. 15): These can all be brazed with silver-copper eutectic in the hydrogen furnace. No special problems are encountered with these materials. Nickel may be copper or silver brazed without special treatment.

PHOSPHOR BRONZE: This material may be handled in the same manner as copper, due regard being given to the subsequent heat treatment, if required.

BERYLLIUM COPPER should be copper or nickel plated before brazing.

BRASS and zinc-bearing bronzes: The copper-zinc alloys should not be brazed in the hydrogen furnace without special precautions. Zinc is vaporizable at silver brazing temperatures; this produces loss of zinc with consequent change in compositional qualities. The brasses and bronzes may be handled only in a hydrogen furnace which is NEVER used for any other materials (especially tube parts), and with a time/temperature schedule which will ensure fast heating and melting of the solder so as not

*This method of hydrogen purification is not suitable for stainless steel or other chromium or aluminum bearing alloys, which are, however, susceptible to treatment by the method outlined on p. 15. Chrome and aluminum free steels should be placed in a clean nickel or stainless steel box with a fairly tight cover in order to protect the material from traces of contaminants residual in the furnace muffles and which enter perhaps from the outside air.

to subject the volatile ingredients to excessive evaporation. The presence of these vapors, besides being noxious and indicating changes in composition, will contaminate the walls of the furnace and consequently all subsequent materials heated in it. Brass and other volatile-metal-bearing alloys may be handled by the method of "bottle" brazing described below.

KOVAR, an iron-nickel-cobalt alloy, presents no difficult problems and may be handled much like nickel and its alloys. In joining Kovar to Kovar, steel or nickel, copper-brazing is the preferred method. The gold-copper and gold-nickel alloys may be used with success in brazing Kovar to copper. Pure silver and pure gold can also be used. Silver-copper eutectic may be used provided the Kovar has previously been stress-relieved above 850°C and copper plated. These precautions are necessary to prevent intergranular penetration of the silver into the Kovar with the consequent liability of the Kovar to cracking. Kovar should always be brazed with quick-melting solder, i. e. pure metals or simple alloys such as eutectics. Intergranular growth occurs in Kovar when exposed to solders having a wide melting range, with resultant penetration of the solder. If the Kovar piece has not been previously annealed above 850°C, this penetration of the solder between grains may eventually result in fracture, especially if the part is to be repeatedly heated and cooled. In general the Kovar should be copper or nickel plated at the junction for any kind of soldering operation.

PREPARATION OF WORK FOR BRAZING: Indications have already been given regarding some of the steps to be taken in preparing work for brazing in the hydrogen furnace. The parts should be clean, and firmly held together by jigs, clamps, screws, wire or weights. Allowances must be made for differences in coefficients of expansion between work pieces and jig, etc., so that if, for example, the jig is of stainless steel and the work copper, the jig, which will expand less than the copper, does not bite into, crush, or squeeze the work as it heats. Copper, nickel and some other metals become quite soft at brazing temperatures. For this reason weights are sometimes to be preferred to clamps or screws.

Care should be exercised to see that nothing can loosen or fall or strike the walls of the furnace as the work is pushed through. The pieces should be loaded into a nickel or stainless steel box or tray. If a cover is used, it should be provided with vents large enough to allow free circulation of gas. Massive pieces or work with small interior spaces having access to the outside only through small orifices should be preheated in the front part of the furnace so that (a) both work and muffle are protected against thermal shock, and (b) the hydrogen has time to displace the air in the interior spaces. When such cavities are large it is advisable to purge them with dry nitrogen or forming gas immediately previous to preheating, to remove the possibility of explosion.

Temperature of brazing will depend on the kind of solder used as outlined above. The time must be determined by experiment, and can usually be done conveniently by placing a small piece of solder in some position where it can be seen while the work is in the heated part of the furnace, but where it will not drip on the

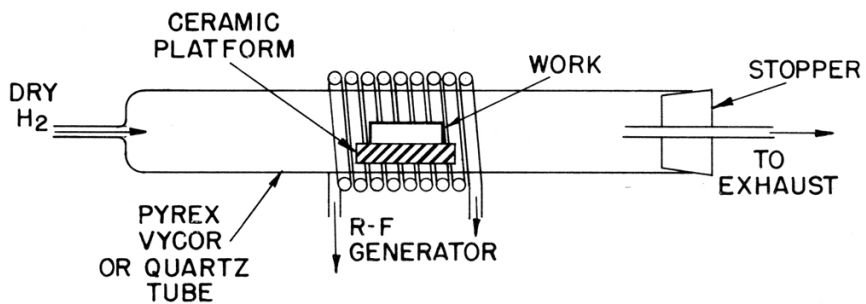
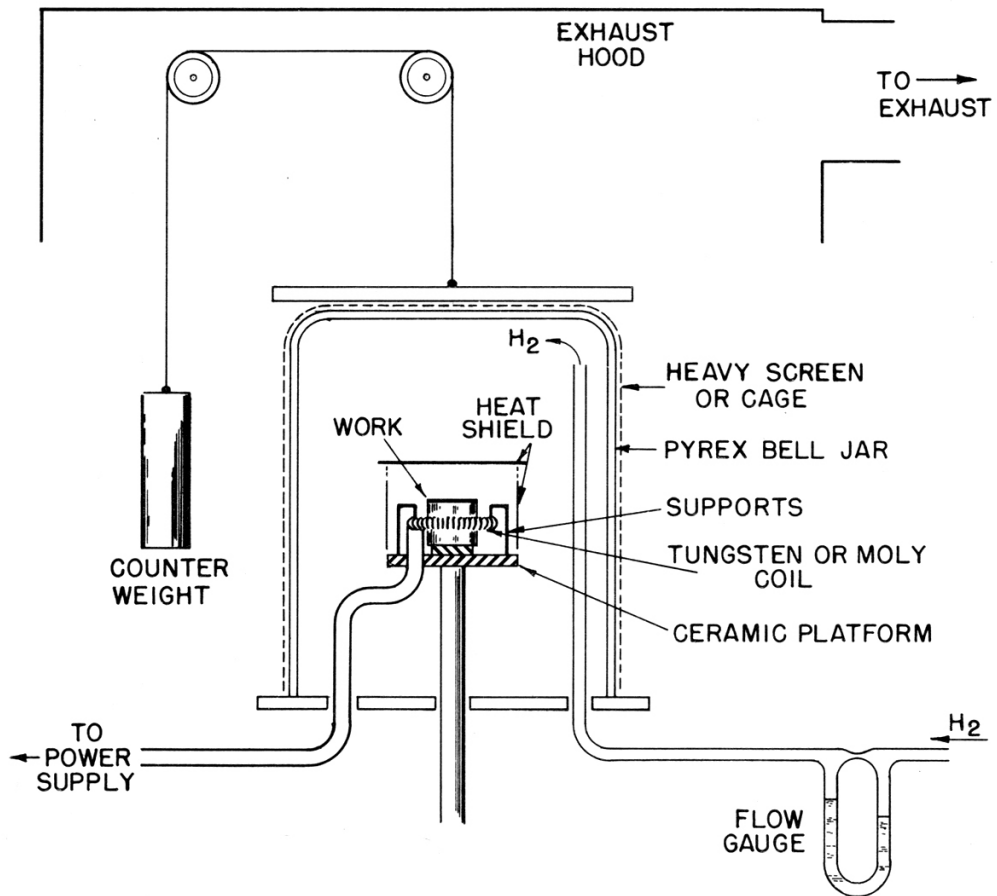
muffle or the work as it melts. Sometimes a piece of solder which is on the work can be observed directly, or a small piece may be attached where it will do no harm and where it can be conveniently seen. Experience will dictate how much time to allow after the solder melts for the work to remain in the heated part of the furnace. Where the pilot piece of solder is directly in contact with the main body of the work, only a short time will be required; a somewhat longer time will be necessary if the pilot is attached to or in contact with the jig or tray, where these are less massive than the work, since the more massive sections will heat more slowly.

BOTTLE BRAZING: Although this method does not involve the use of a furnace, it comes into the picture of controlled atmosphere brazing as a convenient method of working with vaporizable materials, and of applying localized heating to assembled work.

In rough outline, the method is as shown in the diagram, p. 24. A pyrex glass bell jar covered with a protective screen is hung with suitable counterweights under an exhaust hood. Hydrogen is admitted through a flow gauge and metal tube to a point near the inside top of the bell jar. A molybdenum or tungsten heating coil is placed around or near the part of the work which is to be heated, and is supported in several places to prevent sagging or shortcircuiting. The work is mounted on a suitable refractory ceramic platform so as to be about halfway up inside the bell jar, and is surrounded by sheet nickel or molybdenum heat shields. Another heat shield is placed so as to protect the top of the bell jar. Adequate copper leads, heavy enough to dissipate the heat at the ends of the radiation coil, are brought out to a variable voltage supply. A space must be allowed at the bottom of the bell jar for the gas to flow out and away, where it can be drawn off by an exhaust hood or fan.

In operation hydrogen is allowed to flow at a moderate pressure for a length of time sufficient to displace ALL the air in the bell jar. The current is then turned on and the voltage raised slowly from zero. As the work receives the radiated heat the operation is closely observed. A face shield should be worn by the operator. When the solder reaches its flow point there will be, if vaporizable material such as zinc or cadmium is present, some clouding of the glass due to condensed vapors, but this is usually not sufficient to obscure the operation. No fluxes should be used. The work should be clean before setting up. Any brazing alloy or pure metal mentioned above may be used, although generally bottle brazing is confined to the lower melting point hard solders, such as Handy and Harman "Easyflow", "Sil-Fos" and Westinghouse "Phos-Copper". One advantage of bottle brazing is that work which has parts of less rugged materials such as glass already attached may be brazed by properly disposing the heating coil and by the use of auxiliary sheet nickel or molybdenum heat shields to protect such parts against excess heat. The hydrogen is allowed to flow at a reduced rate after the solder has flowed and the power shut off, until the work has cooled enough to be exposed to the air without risk of oxidation or cracking.

STAINLESS STEELS and other Chrome-bearing Alloys cannot ordinarily be



Above: Bottle brazing

Below: Induction heating of stainless steel in dry H_2

brazed in tank hydrogen without special treatment because the water-vapor and/or oxygen content is sufficient to form a protective film of chromium oxide on the metal which prevents the solder from wetting or adhering. This effect can be overcome in two ways.*

By properly purifying the hydrogen, i. e. removal of water vapor and oxygen by passage of the gas through suitable chemical trains, the effect of chrome oxide formation is reduced or eliminated. In order to conserve the flow of pure dry hydrogen, since the chemical or catalytic train will function at an efficiency which is inversely proportional to the rate of flow, the work is enclosed in a steel, stainless steel, or nichrome box, into which the pure dry hydrogen is fed. This box is closed with a piece of iron or nickel foil wired in place, so that a small leakage of gas will occur, and so that only a small flow of hydrogen will be required to attain a positive pressure inside. The box is previously filled after loading the work, with dry nitrogen (or some other dry inert gas), and placed in the preheat section of the regular hydrogen furnace; then the pure dry hydrogen is allowed to flow into the box. The small positive pressure within prevents the ordinary tank hydrogen from diffusing in. After preheating, the box is pushed into the heated section of the furnace in the usual manner. The dry hydrogen must continue to flow until the box is cool enough to be removed from the furnace.

Another dry hydrogen method for handling small work involving chrome-bearing materials is also shown in the diagram. The work is loaded on a ceramic refractory platform so as to center it in a horizontal pyrex, vycor or quartz tube of suitable dimensions. The pure dry hydrogen flows in one end and the loading end is closed with a stopper or gate having a small exhaust tube through which the hydrogen is led away to be burned or otherwise safely disposed of. Heating is accomplished by induction, (see p. 26) in which the water-cooled coil is placed around the outside of the tube as shown. Radiation heating may also be utilized as described in bottle brazing, with proper use of heat shields. The hydrogen must be allowed to flow until all air is displaced before applying heating power.

The second method of handling stainless steel and other chrome alloys is that used by Westinghouse in which the metal parts are carefully cleaned and nickel plated. The well-bonded nickel coat protects the chromium from oxidation so that brazing may be accomplished in the furnace with regular tank hydrogen. (See section on stainless steel p. 15.)

RESISTANCE BRAZING: It sometimes becomes necessary to do brazing operations on assembled work where, in addition to the presence of glass or other materials which must be protected from excess heat, there are parts such as unactivated cathodes which might be damaged by hydrogen. If the apparatus under construc-

*F. C. Kelley, Properties of Brazed 12 percent Chrome Steel; The Iron Age, November 2 and 9, 1939; also see reference no. 4 page 34.

tion is designed for it, one way of handling such work is by the use of the resistance brazer, in which the vessel or tube has two parallel sides which are placed between special graphite pieces machined to fit, and a low voltage high current is made to flow through the work piece sufficient to heat it to brazing temperature. The vessel or tube must be so designed that carbon dioxide bubbling through iso-propyl alcohol can be introduced inside and carried off at a moderate flow rate. At brazing temperatures this gas mixture provides a very good reducing atmosphere which is noninflammable and which does no harm to unactivated cathodes, although the outside of the metal will be oxidized. This latter effect can be removed later by mechanical and chemical cleaning, taking precautions against the entrance of water or chemicals into the tube by plugging up the openings.

A modification of this method, in cases where the shape of the work precludes the use of the resistance brazer, is by the use of a torch. It may be necessary, if the tube is of copper and massive, to use an oxy-hydrogen (reducing) flame. If the outside of the tube is degreased before starting, and thoroughly coated with Handy and Harman "Handy Flux" or other suitable hard soldering flux, the effects of exterior oxidation will be minimized.

NOTES ON INDUCTION HEATING*

The work to be heated is surrounded by a water-cooled coil or inductor of copper tubing which is connected by special water-carrying cables to the vacuum tube oscillator. For maximum energy transfer the inductor should be as close to the work as possible but not in contact with it. Where work is to be heated in a vacuum with the inductor outside, the vacuum vessel walls should be of nonconducting material such as glass or quartz. If the work is to be raised to a high temperature, e. g. tantalum to 1000°C or higher, the vessel must be of such size and the work so centered or positioned as not to cause softening and collapse of the walls by radiated heat.

Since the heating is roughly proportional to the coupling (i. e. for a given piece of work a surrounding coil of small diameter will transfer more energy than one of larger size), a coil appropriate to the work should be chosen. Where the work is not to be raised to a high temperature, and where closer control of heating is required, a coil of larger diameter may be used.

A rule of thumb for estimating the amount of power required to heat a given piece of work: 2KW output for each square inch of surface to be heated. This will vary considerably with size and shape of work.

*Some of the material in this discussion of induction heating is from "High Frequency Induction Heating" by F. W. Curtis, 1944. Courtesy of McGraw-Hill, N. Y.

Approximate formula for calculating heat transfer:

$$WST = \text{btu}/\text{min}$$

Where W = weight of material in lbs

S = coeff. of specific heat of the material*

T = temperature required in ° F.

No allowance is made in this formula for normal heat losses which may be 25 to 50 percent or more in air.

The frequency determines the heat penetration; the higher the frequency the less the penetration. For surface heating of metals and carbon frequencies of 200 to 500 kc are commonly used. Depth of penetration at 200 kc is about 0.020".

The power output rating of an induction heating device is given by:

$$Kw = 2.93 \times \text{wt (lbs)} \times \text{specific heat}^* \times \text{temp. rise (°F)} \times 10^{-4}$$

Magnetic materials, at temperatures up to the Curie point, heat more quickly than nonmagnetic materials because of hysteresis. Nonmagnetic materials heat by eddy currents only. The lower the resistivity the lower the eddy current losses and hence the less heating; i. e. more power is required to heat a piece of copper than a similar piece of nickel to a given temperature.

The induction heaters in this Laboratory require coils (for direct connection)

*SPECIFIC HEATS OF METALS

The values given are approximate for room temperature. In general the values increase slightly with temperature.

| Metal | Specific Heat | Metal | Specific Heat |
|-------------------|---------------|------------|---------------|
| Aluminum | 0.21 | Molybdenum | 0.07 |
| Brass, Bronze | 0.09 | Nickel | 0.11 |
| Carbon (gas) | 0.20 | Platinum | 0.03 |
| Carbon (graphite) | 0.17 | Silver | 0.06 |
| Copper | 0.09 | Tantalum | 0.04 |
| Gold | 0.03 | Tin | 0.06 |
| Iron, Steel | 0.12 | Tungsten | 0.03 |
| Lead | 0.03 | Zirconium | 0.11 |

- from the Chemical Rubber Handbook of Physics and Chemistry.

of about eight turns for approximate impedance match. Where localized heating is to be applied over a small area the current transformer should be used. This is essentially an impedance matching step-down r-f transformer which allows the use of a single turn work-coil, with reduced voltage but little loss in power.

Multiturn coils should be proportioned so that the length does not exceed four times the diameter. Where long lengths of work are to be heated the work should be progressively fed through the coil.

The turns of a coil should be closely spaced without actually touching. Asbestos cord or fiberglass sleeving (without binder) are useful for insulating the turns. Coils are commonly made of high conductivity copper tubing. Annealing and partial flattening are sometimes resorted to so as to bring the turns closer together; this results in more even distribution of the heat.

Coils can also be made from solid copper stock of various basic shapes by machining and brazing. Such coils are useful on specific jobs and therefore have a limited application.

Generally speaking, the coils should have full turns, especially those with only one or a few turns. In single turn coils for use with the current transformer the turn should actually be, in effect, a continuous ring with a small gap; the smaller the gap the more uniform will be the field. In coils with more than one turn the leads should be brought out close together near the coil. They may be separated beyond the coil. This does not apply to coils of many turns (i. e. five or more) since a high degree of uniformity of the field at the ends of the coil is not expected.

Since the voltages employed in induction heating are high enough to cause serious or fatal injury, adequate provision should be made for protection of personnel. Metal tables or frames which are part of or close to an induction heating set-up should be thoroughly grounded to a cold water pipe. The rubber covering on the cables used on the small and medium heaters is not sufficient insulation, and contact with them should be avoided.

Condensation of water sometimes occurs in humid weather on the work coils; breakdown troubles from this source can be reduced by increasing the spacing of the turns or by covering the coil with unsaturated fiberglass sleeving.

BRAZING METAL TO CERAMIC WITH ZIRCONIUM HYDRIDE (Condensed from "Metal to Nonmetallic Brazing," C. S. Pearsall and P. K. Zingeser; M. I. T., R. L. E. Technical Report No. 104, April 5, 1949)

The general procedure in making this type of joint involves coating the surfaces to be brazed with thin films of zirconium hydride. A water paste or a nitrocellulose-solution binder seem to work equally well in producing thin uniform coatings of the hydride. A piece of suitable solder (silver-copper eutectic, "BT", or pure silver) is then placed in contact with the hydride coated surface. The material is heated to a

temperature at which the solder flows freely, (900-1000°C) in a vacuum, in an atmosphere of pure, dry hydrogen, or of commercial (tank) dry nitrogen. When the proper temperature is reached, the brazing alloy will melt and flow over the hydride coated surface in a manner similar to that in brazing.

Titanium and other hydrides are sometimes used but the authors believe zirconium hydride has the greatest range of usefulness.

Alloys or intermetallic compounds of silver and zirconium (i. e. silver 85 percent, zirconium 15 percent) will wet and bond ceramics, diamond, sapphire, carbides etc. in much the same manner as the hydride method. Equally good results are obtained by placing pieces of zirconium metal and silver wire on the surface to be brazed and heating to the flow point. The alloy formed wets and bonds nonmetallic as well as metallic materials in tank dry nitrogen. Good bonds are obtained on stainless steels in either forepump vacuum or dry nitrogen. Thin oxide films on the metal do not inhibit the brazing process since the alloy readily wets and bonds oxides. This alloy also yields good brazes with molybdenum.

The bonds produced with zirconium in tank dry nitrogen, although not so clean as those produced in vacuum seem, however, to be equally sound. The zirconium brazing process has also been successfully performed in commercial tank helium and argon.

Consideration must be given in bonding metals to ceramics for vacuum and pressure seals to the proper matching of expansion coefficients. Improper matching of these values can lead to considerable stress with subsequent failure. In general, glass and ceramic materials are weak in tension and have maximum strength under compression; for example, Alsimag No. 243, used for vacuum seals, has a tensile strength of 2000 psi, while the compressive strength is 85,000 psi. It is also desirable that the expansion coefficients of the materials to be joined match reasonably well over the range of temperatures to be encountered.

It is necessary to control carefully the rates of heating and cooling metal/ceramic combinations during the brazing process, since the thermal conductivities of most ceramic materials are much lower than those of most metals. Steatites, (see p. 56A) useful in vacuum work, are much superior to glasses in their resistance to thermal shock.

Two metal/ceramic combinations which have been used successfully in preparing vacuum seals in our Laboratory (1) are chrome iron No. 430 alloy (see below) with Alsimag No. 243; and Kovar with Coors ZI-4.

(1) D. L. Eckhardt, M.I.T., R.L.E. Quarterly Progress Report; April 15, 1948.

METALS

COEFFICIENT OF EXPANSION (per °C; x 10⁻⁶)

| | Temperature Range | Coefficient |
|---|-------------------|-------------|
| No. 430 Chrome Iron (1) Cr: 14-18; C: 0.12; Mn 1.0; Si 1.0; P; S; Bal Fe | 0 - 600 | 10.5 |
| | 0 - 1000 | 12.0 |
| Kovar (2) | 30 - 500 | 5.71 - 6.21 |
| No. 52 Alloy (3) : Fe 50; Ni 50 | --- | 10.7 |
| No. 14 Alloy (3) : Ni 42; Cr 5.5; Bal Fe | --- | 9.5 |

CERAMICS

See Table, p. 56A.

(1) Distributors: Brown-Wales, Boston, Mass. 165 Rindge Ave. Ext. Boston, Mass., (p. 39)

(2) Stupakoff Ceramic and Manufacturing Co., Latrobe, Pa. See p. 73.

(3) Driver-Harris Company, Harrison, N. J. (See also R. J. Bondley, "High Temperature Metal-Ceramic Brazed Seals", General Electric Research Laboratory Report, Schenectady, N. Y., April 1, 1947.)

HEAT TREATING SCHEDULE FOR VARIOUS MATERIALS

| MATERIAL | PURPOSE OF FIRING | OVEN | TEMP. °C | HEAT TIME MIN. | COOLING TIME MIN. MINIM. | APPEARANCE | REMARKS |
|-----------------------------------|--------------------------------------|------------------------|--|----------------|--------------------------|-------------------------|--|
| Tungsten | Anneal on mandrel | H ₂ | 900 | 5-30 | 30 | Bright | Use SS mandrel prev. oxidized in H ₂ . |
| Tungsten | Anneal wire dead soft | H ₂ or vac. | 1400 | 5-10 sec | -- | Bright | --- |
| Molybdenum | Anneal on mandrel | H ₂ | 900 | 5 | 30 | Bright | Use SS mandrel prev. oxidized in H ₂ . |
| Tungsten or Molybdenum | Firing of alundum-coating on heaters | HiTemp H ₂ | 1600-1650 | 3-7 | 10 | White-hard | Wire should not be too brittle. |
| Nickel | Anneal and de-gas | H ₂ | 900 | 15-30 | 30 | Bright-clean | Comes out dead soft. |
| Copper OFHC | Anneal and de-gas | H ₂ | 700 | 15-30 | 30 | Bright-clean | Comes out dead soft. |
| Copper to Copper | Brazing with BT | H ₂ | 800 | Note A | 30 | Bright-clean | |
| Kovar | Anneal and de-gas | H ₂ | 900 | 15-30 | 30 | Bright-clean | Must be fired before glassing or BT soldering. |
| Kovar to Copper | Brazing with BT | H ₂ | 800 | Note A | 30 | Bright-clean | Kovar must be previously annealed; should be copper plated at junct. |
| Kovar to Steel (not SS) | Brazing with Cu | H ₂ | 1100max | Note A | 30 | Kovar bright | Kovar need not be prev. annealed; Cu plate. Steel may be lightly oxidized. |
| Tungsten or Moly. to Cu | Brazing with Au-Ni | H ₂ | 960 | Note A | 30 | Bright-clean | |
| Stainless Steels or Chrome alloys | Brazing to other metals | H ₂ | Note C | Note B | 30 | Bright-clean | Metals to be plated by Westinghouse method. |
| Lava No. 1137 | Hardening | Air H ₂ | 1100 | Note D | Note E | Lt. tan hard White hard | See notes elsewhere on this subject (Ceramics). |
| Metal to Glass seals | Cleaning | H ₂ | 450 | 60 | 30 | Fairly clean | A second firing may be required. |
| Brass etc. | Brazing | H ₂ Bottle | With radiation heating and adequate heat shields. See pp. 24-26. | | | Clean-bright | |
| Tantalum | Anneal and de-gas | Vacuum | 1000-2000 | Note F | Note G | Clean-bright | Tantalum cannot be heated in hydrogen or nitrogen. |

Note A: Will depend on mass of piece plus jigs. For small work 5 minutes; larger pieces may take up to 30 minutes.

Note B: See section on stainless steel for table on time/temperature

Note C: Will depend on what brazing solder is used.

Note D: Raise temperature at rate of 260 °C/hr max. (lower for large pieces.)

Note E: Allow furnace to cool to room temperature before opening.

Note F: RF heating.

Note G: Allow 30 minutes (min) in vacuum

TUBE LABORATORY PROCEDURES

OUTGASSING OF TUBE COMPONENTS BY VACUUM FIRING

In addition to the method of outgassing tube parts by firing in the hydrogen furnace, the firing of metal parts in vacuum is a very useful and often mandatory method of outgassing, especially in the case of tantalum and unplated stainless steel.

A special exhaust station is set up in this Laboratory for the purpose of outgassing tube parts prior to their assembly. This system is similar to the ones described in the section on processing tubes during exhaust, p. 40, except that there is no bake-out oven and no ionization gauge. Heating is done externally by means of an induction (r-f) coil. Since pressure changes are likely to be sudden and rapid, the range of pressures encountered will be too great for any of the usual type of gauges, and particularly injurious to the ionization gauge. A thermocouple gauge may be used to measure forepressures.

The work is suspended at about the middle of a pyrex tube of suitable bore connected to the pumping system through a ground joint, which should be cleaned and freshly greased at the start of each operation. Tantalum or molybdenum wire is used to hang the object in the tube. In the case of small parts such as screws, nuts, tabs, insulators and the like, use is sometimes made of a tantalum "bucket" or cylindrical box with a double wall and having baffled or staggered holes to allow the evolved gases to be drawn off. In some instances a single wall bucket is used in conjunction with some clean alundum sand. A quantity of this sand should also be poured into the bottom of the glass tube so that in case the suspension fails the work will not break the tube in falling.

All parts to be vacuum fired should be degreased and chemically cleaned; i. e. the firing schedule should be the last operation prior to assembly in the tube. The use of a tantalum bucket allows nonmetallic materials such as ceramics to be fired in this way.

After the work is suitably suspended in the glass tube (it should be centered axially) and the ground joint closed, the large stopcocks are opened and the forepump started. The pressure is checked with a sparker, and after the proper interval, as discussed under tube processing, the oil diffusion pump and blowers are turned on. A coil connected to the induction heater is arranged around the glass tube so as to enclose the work in its field. It should be borne in mind that the closer the coupling, that is, the nearer the coil to the work, the more power will be transferred and the greater the heating. Before starting the induction heater the water flow through the various circuits should be adjusted to a value of not more than one gallon per minute. Excessive water pressure is unnecessary and wasteful. A blower is provided to direct a stream of air on the glass tube, and this should now be turned on. The filaments of the oscillator tubes should be switched on at least 15 minutes before plate voltage is applied; in about the same length of time the oil diffusion pump will "take hold". Induction heater power should be applied cautiously at first because most materials give off copious quantities of gas as soon as the temperature rises. So much gas may be given off that the pumps will be unable to remove it fast enough, and a bright glow discharge will occur within the glass tube.

When this happens the plate voltage should be shut off and not reapplied for several minutes or until the sparker shows no further fluorescence.

The following table shows outgassing temperatures for various materials:

| Material | °C Max. | |
|--|-----------|--|
| Tungsten | 1800 | |
| Molybdenum | 950 | (embrittlement takes place at higher temperatures) |
| Tantalum | 1400 | |
| Platinum | 1000 | |
| Copper and alloys* | 500 | |
| Nickel and alloys (Monel etc.) | 750-950 | |
| Iron, Steel, Stainless steel | 1000 | |
| Graphite | 1500-1800 | |
| Lava (No. 1137 previously air or H ₂ fired) | 800 | |

The time required for outgassing will depend on the size and mass of the material. When the work can be held at its maximum temperature without glow discharge, the induction heater power should be reduced somewhat and allowed to run 5 to 15 minutes. Massive pieces will continue to outgas for some time since the heat will penetrate very slowly. Materials with high electrical conductivity will require more r-f power because the heating is proportional to the IR drop. The maximum safe temperatures should not be exceeded as to do so will result in evaporation and sputtering with possible injury to the work and to the glass tube.

After firing, the work should be allowed to cool in vacuum. Massive pieces may take as long as several hours because of poor heat losses. The system is arranged so that after a short cooling time, the stopcock between the ground joint glass tube and the diffusion pump is shut off, whereupon the work is isolated under vacuum and the pumps may be shut down in the manner outlined in the tube processing section. To open the glass tube allow air to enter slowly at the bleeder stopcock. The work should be handled only with tweezers or clean gloves and should be assembled in the tube as soon as possible after being exposed to the atmosphere. If for some reason, a considerable time must elapse between firing and assembly, the work should be placed in a clean covered petri dish or beaker, or a new cellophane bag, and kept in an evacuated desiccator until ready for use.

HEATER DESIGN AND PREPARATION

The purpose of the heater is to raise the temperature of the electron emitting materials of the cathode sufficiently to produce the optimum thermionic emission. In the case of cathodes coated with barium-strontium carbonates, this temperature is approx-

*Except zinc bearing alloys which cannot be vacuum fired at high temperatures owing to excessive evaporation of zinc. (See pp. 23-24).

imately 850°C. However, while the tube is being evacuated, it is necessary to reduce these carbonates to oxides (see section entitled "Processing of Tubes on Exhaust", p. 40) by heating to approximately 1150°C and maintaining it for several minutes at this temperature.

In order to accomplish this it has been found advisable to design the heater so that its maximum temperature will not exceed 1400°C in order to keep safely below the melting temperature of the alundum insulating coating on the heater wire. Table A gives the approximate current necessary to raise the temperature of either tungsten or molybdenum wire to 1400°C in vacuum, together with the voltage drop at this current per inch or centimeter.

The heater usually consists of a coated tungsten or molybdenum wire or ribbon which has been wound on a mandrel or otherwise shaped. For some shapes it is necessary to anneal the wire on the mandrel or form in hydrogen. See p. 36.

The heater is inserted in the cathode sleeve or otherwise mounted close to the cathode in such a way that there is no electrical leakage between them, or between adjacent turns of the heater. This requires that the heater coating be hard and tough to withstand the abrasion incidental to mounting. The alundum material which satisfies these conditions and which is used in this Laboratory is obtained from the Raytheon Manufacturing Company under the designation A41-4 and is made according to the following formula:

Approximately 0.08 pound 1000 second nitrocellulose (Hercules Powder Company) is dissolved in equal parts of butyl alcohol and butyl acetate (about 1 gal of mixed solvents) to give a viscosity of 17.5 ± 0.1 centipoises at 74°F. To 1000 cc of this binder 635 grams of 38-900 alundum is added. This mixture is diluted with 705 cc of a mixture of equal parts of butyl alcohol and butyl acetate. The mixture is rolled to be sure of complete wetting of the particles, and then strained through cheesecloth as an additional precaution to remove coarse particles. A small amount of aniline dye (Sudan Red III, oil soluble) is added as identification. The material is agitated on the roller one hour before using, and is applied by spraying with a DeVilbiss spray gun.

In winding helical heaters to a given O.D. the size of mandrel required will have to be determined by trial and error. The wire will always spring open to a larger diameter when tension is released. However, where heaters are to be annealed in the hydrogen furnace on the mandrel (which in this case should be grooved or threaded) there will be practically no change in dimensions. For this operation the wire is held firmly in place while firing. The mandrel must be so designed that the wire can be unscrewed or otherwise easily removed. Firing of tungsten is done in hydrogen at 900°C for 3-5 minutes, followed by regular cooling schedule. (See Table p. 31.)

After the heater has been formed to the required shape it is cleaned by boiling in 20 percent potassium hydroxide solution for five minutes* followed by thorough rinsing in

*Tungsten heaters and filaments should not be electrolytically cleaned or subjected to any strong chemical reagents.

distilled water and drying. The alundum coating is then sprayed on to the specified thickness (usually 0.003") and the heater is then baked at 100°C for one-half hour in an air oven to evaporate the vehicle. The heater should be examined at this point for defects in the coating, which should be remedied. The heater is now fired in the high temperature hydrogen furnace at 1600°C - 1650°C for 3 to 10 minutes, depending on the size of the heater, the exact time and temperature to be determined by trial. Too high a temperature will embrittle the wire or form an excessively hard coating which will break the wire if bent. Too low a temperature will produce a coating which is soft or which will change on standing due to incomplete decomposition of the spray constituents.

After firing, the heater is again examined for defects and if none are found it is ready for welding or assembly into position in the tube structure.

An alundum suspension (in water) may also be applied by cataphoretic deposition. The theory and process of this method are described in U. S. Patent No. 2,307,018 (Jan. 5, 1943). This method is useful in patching heaters on which the coating has become chipped, as alundum deposition occurs only on such portions of the wire as are bare or where the coating is extremely thin.

TABLE A

Volts per inch and current in amperes necessary to raise wire to 1400°C. Straight wire in vacuum

| WIRE SIZE inches | VOLTS | | AMPERES | |
|---------------------|--------|----------|----------|------------|
| | Per cm | Per inch | Tungsten | Molybdenum |
| 0.002 | 0.32 | 0.81 | 0.18 | 0.22 |
| 0.0025 | 0.28 | 0.71 | 0.26 | 0.31 |
| 0.003 | 0.25 | 0.63 | 0.34 | 0.41 |
| 0.0035 | 0.24 | 0.61 | 0.41 | 0.49 |
| 0.004 | 0.22 | 0.56 | 0.52 | 0.62 |
| 0.0045 | 0.21 | 0.53 | 0.60 | 0.72 |
| 0.005 | 0.20 | 0.50 | 0.72 | 0.86 |
| 0.0055 | 0.19 | 0.48 | 0.84 | 1.00 |
| 0.006 | 0.18 | 0.47 | 0.95 | 1.13 |
| 0.0065 | 0.18 | 0.46 | 1.05 | 1.26 |
| 0.007 | 0.17 | 0.44 | 1.20 | 1.44 |
| 0.0075 | 0.17 | 0.43 | 1.3 | 1.56 |
| 0.008 | 0.16 | 0.42 | 1.45 | 1.73 |
| 0.009 | 0.16 | 0.40 | 1.75 | 2.10 |
| 0.010 | 0.15 | 0.38 | 2.25 | 2.70 |
| 0.012 | 0.14 | 0.36 | 2.60 | 3.12 |
| 0.014 | 0.14 | 0.35 | 3.25 | 3.90 |
| 0.015 | 0.13 | 0.34 | 3.70 | 4.44 |
| 0.020 | 0.12 | 0.30 | 5.6 | 6.6 |
| 0.025 | 0.11 | 0.28 | 7.4 | 8.9 |
| 0.030 | 0.10 | 0.25 | 10.5 | 12.0 |
| 0.040 | 0.09 | 0.22 | 16.0 | 18.2 |

TUNGSTEN FILAMENTS

Tungsten wire or ribbon is used in making direct emitters which operate at temperatures considerably higher than the 1400° C value of the indirect heaters for oxide-coated cathodes (see p. 43). Consequently somewhat different heat-treatment and handling will be required. Filaments at high temperatures have a tendency to sag or warp unless heat-treated and supported adequately. Special consideration should also be given to lead wires.

Tungsten emitters for use at 2500° K are to be cleaned in the same manner as heaters (see pp. 2 and 33). The filaments should be formed and firmly held on an oxidized stainless steel jig or mandrel, and hydrogen fired for 20 to 30 minutes at 900° C. As this heat treatment produces considerable brittleness of the tungsten it should be handled carefully and mounted strain-free. Cleaning may take place after firing but it is preferable to assemble the cleaned straightened wire on the clean (but oxidized) mandrel. Hydrogen fire and assemble in the tube immediately after firing if possible; otherwise the filament should be stored in a dry, dust-free place, usually a vacuum desiccator. The filament should be handled only with gloves or tweezers.

It is obvious that high temperature tungsten filaments should not be spot welded to nickel leads of inadequate size or close to the high temperature zone. If nickel leads must be used they should be heavy enough to operate at a maximum temperature of 700° C. The best lamp-making practice requires tungsten filaments to be attached to leads by clamping, crimping, or the use of small screws, rather than welding, because of the tendency of welds to form low melting point eutectics or alloys. Welds can be made to tungsten and molybdenum leads by the interposition of a "flux" (see p. 59) of tantalum or other refractory metal, or the tungsten filament may be directly welded to tantalum lead wires.

Support wires for high temperature tungsten filaments are made of so-called tungsten "hook" wire which is a specially heat-treated material with somewhat more stiffness than annealed filament wire.

CATHODE COATING

The material used in this Laboratory for coating oxide cathodes is obtained from the Raytheon Manufacturing Co., Newton, Mass., under designation C51-2. It is composed of a binder (B71-5) consisting of 1000-second nitrocellulose (Hercules Powder Company) dissolved in equal parts of butyl alcohol and butyl acetate* to give a viscosity of 17.5 ± 0.1 centipoises at 74°F. To 1700 cc of this binder is added 1600 grams of "Radio Mixture" No. 3 (J. T. Baker Co.), which consists of 57.3 percent barium carbonate, 42.2 percent strontium carbonate and 0.5 percent calcium carbonate. This mixture is ball-milled for seven hours. For use, 1500 cc of the ball-milled suspension is diluted with 750 cc of

*Proportions are 0.08 lb. nitrocellulose to 1 gal. mixed solvents.

the 17.5 cps binder.

It requires about one hour of rolling in 1/2 gallon bottles at about 50 rpm to prepare the suspension for use in a spray gun. Spray gun bottles are cleaned with acetone and dried before the suspension is poured in. This Laboratory uses a DeVilbiss type CH spray gun. The fluid needle is turned down to a sharp point to allow fine adjustment. Compressed air is supplied through a DeVilbiss type HB air transformer. The pressure on the gun is set to 25 pounds with the gun operating. The fluid screw is set at about 1/2 to 3/4 turn, the spreader valve adjustment being zero. The gun is cleaned after using by removing the spray bottle and spraying clean acetone through the gun, which is to be used for this type of material only. Tank dry nitrogen may be substituted for compressed air if preferred

Spraying should be done in an exhaust booth to protect the operator and materials.

The cathode coating may also be applied to certain supports or bases with a brush (red sable No. 5 is a convenient size) which should be previously cleaned in acetone and dried. Cathodes may also be coated by dipping. Both brushing and dipping may be done repeatedly, allowing each coat to dry, in order to build up the required thickness. Any unevenness can be ameliorated by scraping with a sharp blade after the coating is dry.

PROCEDURE FOR ASSEMBLING TUBES

Assuming that the electrical requirements, and consequent geometry of the tube have been decided upon, the first step is to formulate a mechanical design with complete dimensions and kind of materials to be used. It is an excellent plan at this point to organize a step-by-step procedure, in which the order of cleaning, firing, machining, plating and all operations including those of assembly, are decided upon.

Because each new tube presents unusual problems, no exact procedure can be outlined. As a rough guide, however, the following points might be considered.

A. Parts to be machined. These include spinning, punching and forming, as well as the more usual machine shop operations. Materials handled under this head comprise all the metals used including the preparation of Kovar for glassblowing. The glassblower should be consulted regarding all Kovar-glass seals. The machine shop will also handle the winding of tungsten coils of wire larger than 0.030" diameter, and the machining of lava or other ceramics, and mica insulators.

B. Parts to be formed by hand. Certain parts such as nickel, tantalum or other sheet metal cylinders or other forms not readily machined, also small tungsten or molybdenum coils, will be made by the tube technician, who should be consulted before deciding upon a design.

C. If the envelope is to be glass, and if a press or presses are required, dimensional sketches are to be given to the glassblowers. Provision is to be made for exhaust tubulation and for gettering if required.

D. Any special parts that are to be made outside the laboratory, such as ground or machined quartz, castings and parts requiring operations for which the laboratory is not

equipped, are to be accounted for.

It is important that closed spaces, pockets or crypts be avoided in tube structures. Obviously such spaces are gas traps and therefore are difficult to evacuate, even though not completely gas tight: For example, when mica discs are used to center an electron gun in a glass tube, the micas should be provided with notches, holes or slots of dimensions ample enough to pump through. Likewise a ceramic disc used to close the end of a metal cylinder and to provide insulation for heater leads, etc., should be provided with a hole at least one-quarter the diameter of the cylinder itself. Screws should not be threaded into blind holes for the same reasons. When this type of assembly is unavoidable, provision should be made for evacuating the pocket so formed by means of another hole drilled at right angles or otherwise.

Cleaning.

A. Metal and other parts are to be degreased and cleaned according to the directions given in "Chemical Procedures", pp. 1-9.

B. Any chemical treatment or electroplating which is to be done before heat treating is performed at this stage.

C. Nickel, tungsten or Kovar leads on presses are to be chemically cleaned.

D. Nickel supports or bases for cathode and fluorescent coatings are to be chemically cleaned by the C-1A process (p. 3) with final rinses in pure, clean, anhydrous acetone or methanol.

E. Tungsten or molybdenum heaters and filaments are cleaned by the process given on p. 2. Other tungsten and molybdenum parts can be cleaned electrolytically. (p. 1)

Heat treating or firing.

A. All metal parts not attached to glass are to be fired either in the hydrogen furnace or in vacuum, for purposes of annealing and degassing. This operation can sometimes be combined with furnace brazing. Such metals as tantalum and stainless steels which are adversely affected by hydrogen firing are to be vacuum fired (see p. 32). Other metals may also be vacuum fired. Kovar parts must be hydrogen fired previous to glass sealing. See remarks on Kovar, p. 63.

B. In the case of parts made of steel or copper which are heavy or have thick sections or large areas, it is advisable to stress-relieve and degas in the hydrogen furnace after rough machining. This insures dimensional integrity when the final machining is done, and eliminates the risks of warping when the finished tube is being operated hot.

C. Tungsten or molybdenum heaters and filaments are often heat-formed. This process is described on p. 36.

Spray Coatings.

A. Heaters for use with indirectly heated cathodes, or which otherwise require an insulating coating, are to be processed according to the method given on pp. 33-35. These heaters can be stored in clean covered petri dishes.

B. Cathode spraying, outlined on p. 36, is to be done just prior to assembly in the tube. If necessary, sprayed cathodes can be stored in a vacuum desiccator with a drying agent. Moist air is harmful to the coating.

Assembly.

All parts are now ready for assembly. Glass parts and metal lead wires on presses have been cleaned and dried. All metal, ceramic and other parts have been cleaned and/or outgassed. Heaters and cathodes have been prepared. Wires of appropriate metals and sizes in short lengths have been stretched or straightened, cleaned and outgassed, for making connections and supporting structures, etc.

Cleaned parts should be handled only with clean tweezers or pliers and the operator should wear clean lintless gloves (some types of rayon are excellent). Assembly and all cathode preparation should be done in a dust-free room, preferably air-conditioned.

After assembly, the tube is closed up, by the glassblowers if the envelope is glass. It is then sealed on to an exhaust system and pumped according to the specifications given under "Processing of Tubes on Exhaust", p. 40. Glass tubes should be pumped and baked as soon after assembly as possible so that (a) if a coated cathode is present it will be protected from the atmosphere, and (b) the glass will be thoroughly annealed by the baking and subsequent slow cooling, with the internal parts protected from the atmosphere.

If required, tubes can be opened after processing for the purpose of making repairs, replacing heaters or filaments, or making changes in the structure. Provision for such contingency should be made at the time of the original design, and the glassblower and tube technician should be consulted beforehand. In general, when a tube with a coated cathode is opened to the air after activation, it will require removal of the coating, recleaning of the nickel base and respraying, because the oxides are very susceptible to reaction with water vapor, and to poisoning by other atmospheric vapors. Tungsten filaments are not affected and, if clean, may be used without change. When getters have been fired in a tube which is to be opened it is usually necessary to install fresh getters, and to remove the evaporated products from the glass by chemical cleaning.

In closing a tube either of glass or metal, it is sometimes necessary to protect the interior and structure of the tube against oxidation due to the heat of glassblowing or brazing. This can be accomplished by passing a reducing or inert atmosphere through the tube. The atmospheres used are dry nitrogen, "forming" gas (80 percent dry nitrogen, 20 percent hydrogen: nonexplosive and has some reducing action), carbon dioxide, either alone or passed over or bubbled through isopropyl alcohol (the gas and alcohol mixture has strong reducing action in contact with heated surfaces), helium or

argon. Mixtures of these gases may be used to obtain various effects. In general, tubes with unactivated oxide coated cathodes or barium getters should not be subjected to a strongly reducing atmosphere.

When the behavior of a cathode of any type which is to be assembled into a complicated structure cannot be accurately predicted from the viewpoint of emission and heat radiation, it is sometimes advisable to construct a simple diode with geometry similar to the final tube. Considerable information can be obtained in this way without going to the expense and trouble of building a more complex tube.

PROCESSING OF TUBES ON EXHAUST

A. General Description of Exhaust Stations (1)

The systems used in the Tube Laboratory consist of the following equipment:

1. A forepump (sometimes called mechanical or roughing pump).
2. An oil (or mercury) diffusion pump, or aspirator, usually consisting of two or more stages.
3. A cold trap with provision for baking out.
4. A low pressure measuring device, normally an ionization gauge with associated amplifier and controls (see detailed description, p. 45).
5. An all-glass manifold to which the tube or vessels to be pumped are sealed.
6. An adjustable electric oven for bake-out, with controls and temperature indicator.
7. A high vacuum stopcock inserted between the forepump and the diffusion pump; also a small bleeder stopcock (for admitting air and for glassblowing) inserted in a branch adjacent to the forepump.

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- (1) Space limitations prohibit even a superficial treatment of vacuum systems and pumps, and the measurement of low pressures. The reader is referred to the standard texts on these subjects, including the following:

"Scientific Foundations of Vacuum Technique," Saul Dushman; John Wiley and Sons, Inc., New York, 1949. Pumps: Chs. 3, 4, 5; pp. 126-257; Measurements: Ch. 6; pp. 258-386.

"Procedures in Experimental Physics," John Strong and Collaborators; Prentice-Hall, Inc., New York, 1942; pp. 93-150.

"Vacuum Pumping Equipment and Systems," H. M. Sullivan; R. S. I., 19, 1, January 1948; pp. 1-15.

"Vacuum Systems, Seals and Valves," F. N. D. Kurie; R. S. I., 19, 8, August 1948; pp. 485-493.

"The Production of Low Pressures," and "The Measurement of Low Pressures," B. B. Dayton; High Vacuum Apparatus (a periodical bulletin), published by Distillation Products, Inc., Rochester, N. Y., 1949.

"Vacuum Equipment and Techniques," A. Guthrie and R. K. Wakerling; McGraw-Hill Book Co., Inc., New York, 1949.

8. The r-f generator for induction heating (not part of the systems).

There are also controls, resistors, switches and blowers where required, also a Tesla coil or sparker for rough vacuum tests.

B. General Procedure for Exhausting Tubes.

The tube is sealed on to the manifold and after a few minutes to allow the seal to cool the forepump is started. A sparker is used to check for gross leaks in the glass seals* and also to determine when the diffusion pump should be turned on. The proper condition is indicated when a pale bluish diffuse glow or fluorescence occurs within the glass system. If no gross leaks are present, this pressure, corresponding to between 10^{-3} and 10^{-4} mm Hg, will be attained in roughly 5 to 30 minutes, depending upon the pumping speed as controlled by the bore and length of the pumping tubulation, as well as on the volume being pumped.

After starting the diffusion pump, allow 20 to 40 minutes, or until the oil in the pump is seen to drip off the ends of all the jets at a steady rate. The sparker should now indicate a "black" vacuum, i. e. only very slight fluorescence or none at all. The ionization gauge may now be turned on. When the pressure falls to about 5×10^{-5} mm Hg the tube is ready for baking out. The oven is lowered over the tube and the current turned on. The pressure will begin to rise immediately, due to water vapor and other gases being released from the glass walls and other parts of the tube. It may be necessary to shut off or lower the operating voltages of the ionization gauge during this initial bake-out period. Tubes are normally subjected to a temperature of 450°C to 500°C for a length of time necessary to bring the pressure down to 5×10^{-6} mm Hg at the maximum temperature. During this heating process the cold trap is also heated, usually with a separate heater, to drive off vapors and gases which may have formed from the previous exhaust operation. When this pressure has been reached the oven is shut off but not raised, thus allowing the tube to cool slowly or anneal. The pressure will fall as the tube cools and should drop to 2 to 5×10^{-7} mm Hg at room temperature. The oven may be raised when the temperature drops to 100°C or lower.

In tubes with oxide coated cathodes, (see p. 36) the next step is to activate the cathode. Actually the coating consists of barium and strontium carbonates with a binder, and the activation comprises elimination of the binder and conversion of the carbonates to oxides, in vacuum.

The regular Tube Laboratory procedure is to apply a small a-c voltage to the heater and to raise it by small steps while watching the ionization gauge reading.

BINDER. Where the cathode is visible, removal of the binder is accomplished by holding the temperature in the neighborhood of 550°C (below red heat). The pressure should not be allowed to rise above 5×10^{-5} mm Hg. If the temperature rises too fast there is risk of blistering, flaking or evaporation of the coating.

*See section on Hunting for Leaks, p. 46.

Where the cathode is not visible, it is a good plan to set up a test diode having similar geometry but with the cathode visible, upon which activation and electrical tests can be made, so that when the actual tube is being processed the cathode temperature measured by an optical pyrometer may be readily correlated with heater power. If such information is lacking, the progress of activation must be governed entirely by the pressure, which, in the process of binder elimination, should not exceed 5×10^{-5} mm Hg. A safe rule of thumb is to raise the cathode temperature slowly so as to keep below this maximum pressure for the first 30 minutes (for small heaters). If this procedure is followed it can be assumed that all binder removal products have been eliminated.

The cold trap is now used with liquid nitrogen, and is so kept during the entire activation program. It has been found in general that if the pressure is not allowed to rise above 10^{-4} during the activation, this process will take place in as expedient a manner as is allowable with safe practice. The rise in pressure is due largely to the evolution of carbon dioxide, with perhaps some water vapor and other gases. When the cathode becomes incandescent an optical pyrometer may be used for gauging the temperature. The normal operating temperature for an oxide coated cathode is between 800°C and 900°C , but during activation heater currents to produce cathode temperatures up to 1100°C may be applied for short periods of 30 seconds, at intervals of several minutes. This is called "flashing". The maximum chemical change occurs in the neighborhood of 900°C ; the higher temperature is applied so as to assure complete conversion of the carbonates to oxides, especially at the ends of the cathode sleeve where radiation losses are considerable.

During the activation, for each increase of applied heater power, the pressure will rise rapidly to a value approximately proportionate to the increase in temperature. It should be allowed to fall before applying a further increase of heater power. When the cathode temperature approaches 900°C the rate of gas evolution will rise considerably, indicating that the peak of the chemical conversion is being reached. Further increases of temperature, beyond the peak will result in a decreasing evolution of gas, and the pressure will fall to a lower level when the normal temperature of 800°C - 900°C is resumed after flashing. When this condition occurs, a small d-c voltage may be applied to the anode (or to the anode and grids or other electrodes tied together). It will be observed that the pressure will begin to rise again as soon as this voltage is sufficient to produce an electron current. This is owing to the evolution of gas from the anode etc. by electron bombardment. With tubes of applicable geometry considerable electron bombardment may be tolerated indeed with beneficial effects (outgassing of the structure), but with certain designs overheating may cause mechanical failures from expansion and warping of metal parts.

A small anode current should be drawn continuously until the pressure drops to about 2×10^{-7} mm Hg. At no time during the early stages of drawing current should the pressure be allowed to rise above 5×10^{-6} . Pressures in excess of this value may give rise to injurious discharges due to positive ion bombardment. Oxide coated cathodes are

particularly susceptible to damage from this source.

A tube is said to be "hard" when changes in d-c voltage produce little change in pressure.

In tubes where electron bombardment cannot be produced sufficiently from the cathode emission to outgas the structure, resort is had to the induction heater or "bomber". This piece of equipment consists of a vacuum tube oscillator of sufficient power and with necessary controls to permit heating of a tube structure by means of a water-cooled coil placed around the outside of the tube. This process is called "bombing" and must be used with understanding and caution.

Bombing of tubes without oxide coated cathodes is carried out at any time after bake-out. Ionization gauges may be bombed after installation in a system. Anodes and other outer structures of tubes with oxide coated cathodes can be bombed twice: before and after activation. Excessive heating should be strictly avoided because of the injurious effects of radiation and evaporation on the cathode, both before and after activation. In general tubes with oxide coated cathodes should be no more than lightly bombed, i. e. the temperature of the parts heated should not exceed an incipient red heat (about 550°C).

In tubes containing one or more getters, i. e. pellets or wires of barium or other metals having high gas-holding or "fixing" abilities (see Espe, Knoll and Wilder, Electronics, Oct. 1950, p. 80), the getter should now be flashed or activated by heating. The pellet type is heated by the induction coil, usually placed with its axis perpendicular to the tube. The wire type of getter is heated by applying a small a-c voltage to the leads supplying it. Some gettering action is attained when the active metal is plainly evaporated, that is, begins to form a mirror, on the adjacent glass. It is obvious that the getter should be located in the tube and shielded with nickel gates so as to preclude the possibility of metal being sputtered on the stem or structure where its presence would produce electrical failure. With the filament type of getter, some operators prefer to burn it out entirely, e. g. where the getter filament is attached to the leads of two electrodes.

The tube is to be sealed off immediately after firing the getters, or as soon thereafter as the pressure has dropped to 2×10^{-7} or better.

Some workers like to bake the tube again after drawing d-c and before flashing the getters. Where a very hard tube is required, the baking, both first and second, is extended many hours, with cathode at normal operating temperature and a small d-c being drawn.

In tubes with pure tungsten emitters, the procedure is the same except that the cathode or filament does not require activation beyond heating to the operating temperature. Some gas will be driven off since tungsten has considerable gettering action.

THORIATED TUNGSTEN

This discussion was taken from "Fundamentals of Vacuum Tubes" by A. V. Eastman, copyright 1937. Courtesy of McGraw-Hill, N. Y.

Thoriated tungsten emitters are "formed" or activated by being operated in vacuum

at a temperature considerably above normal, usually at about 2200°K. At this temperature some thoria is reduced to metallic thorium and works its way to the surface of the filament where it deposits in a layer one atom deep, the additional thorium merely displacing some which has already formed and so gradually reducing the supply of this metal in the cathode. The normal operating temperature for thoriated tungsten is about 1900°K, as compared with 2500°K for pure tungsten.

Procedure: Raise the temperature of the filament to about 2800°K and hold for 10 to 30 seconds. This rapidly reduces some of the thoria to fresh thorium, which diffuses toward the surface and evaporates. The temperature is then reduced to about 2200°K and maintained at that temperature from 10 to 30 minutes as required. This process is called "ageing", as it is during this period that the new surface is formed. This temperature is too low to provide much new thorium or to cause appreciable evaporation, but that thorium which was formed during the flashing process will continue to diffuse to the surface.

At intervals during the ageing process the filament temperature may be reduced to normal and the emission determined by applying a saturating plate voltage. When such tests show no further increase in emission, it may be assumed that the new surface is entirely formed.

| Normal filament voltage | Flashing voltage | Ageing voltage |
|-------------------------|------------------|----------------|
| 3.0 | 10.0 | 4.5 |
| 5.0 | 18.0 | 7.0 |

For tubes with filament voltages higher than 5, flashing is not recommended, but ageing may produce satisfactory results at 40 percent higher filament voltages and for somewhat longer time than is required for the 3 and 5 volt tubes.

Both thoriated and oxide coated cathodes are particularly subject to injury under positive ion bombardment, especially the latter, as pointed out earlier.

C. Shutting Down Vacuum Systems

After a tube has been sealed off, the system should be allowed to run until the glass is cool. The procedure for shutting down is as follows:

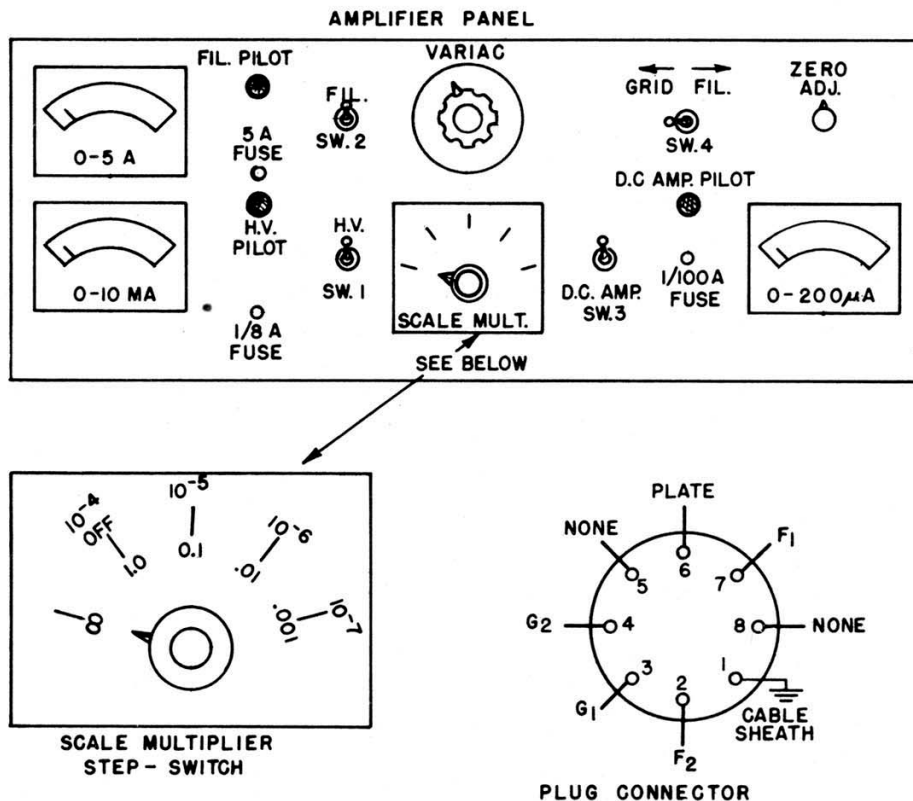
1. Remove liquid N₂ from trap and allow to reach room temperature.
2. Turn off heaters on the diffusion pump; blowers should be left running.
3. When oil in diffusion pump is cool (below 60 °C) or after about 30 minutes, close the stopcock between the forepump and the diffusion pump.
4. Turn off forepump and blowers.
5. The system remains under vacuum and the diffusion pump oil is protected against dust and oxidation.

The small bleeder stopcock to the forepump may be opened to prevent the oil from being drawn up into the rubber and glass parts adjacent to it.

THE IONIZATION GAUGE AND AMPLIFIER

When conditions are appropriate, i. e. when the sparker shows faint to no fluorescence in the glass parts of the system, the ionization gauge can be turned on.

1. Set variac at zero; scale multiplier on OFF (1.0 or 10^{-4}) position. Turn on switches S1, S2, and S3. Turn switch S4 to FIL position.
2. After five minutes turn variac up slowly until 0-10 milliammeter reads 5 ma. The ammeter will now indicate the filament current which should be from 2.5 to 4.5 amp.
3. Zero of microammeter (ion current) should be set from time to time as follows: for each position of the scale multiplier turn the variac to zero and adjust meter with ZERO ADJ. knob.
4. If microammeter now reads on scale with multiplier set at 1.0 the reading is translated into what it would be if full scale were 10 units and multiplied by 10^{-4} . As the pressure falls, the reading of the meter will diminish, and when it reads less than 1 unit, the scale multiplier can be turned to the next step, $0.1 = 10^{-5}$.



Read meter as though full scale were ten units; multiply by step-switch readings as shown in this figure. For example, half-scale reading on meter with step-switch on 0.01 would be 5×10^{-6} mm Hg.

5. The grid of the ionization gauge may now be outgassed. Turn the variac to zero, throw switch S4 over to GRID and bring up the variac until the ammeter reads 5 amp. This is the current flowing through the grid which is a double ended heavy tungsten wire spiral and is now connected as a filament. The grid should show a dull red to red color, and should be left in this condition for at least five minutes. Then turn the variac back to zero, throw switch S4 back to FIL and bring the variac up again until the milliammeter reads 5 ma. It will be observed that the pressure is somewhat higher than before outgassing the grid, but in normal operation it will drop back to a value even lower.

6. Do not leave the ionization gauge filament running at full current for long periods. If it is necessary to pump for several hours or overnight, turn off the ionization gauge amplifier entirely, with multiplier switch at OFF. For shorter periods the filament of the ionization gauge should be turned down so that it is operating at about 75 percent of normal. Excessive filament current serves no purpose as the amplifier is calibrated for the 5 ma electron current. For this reason it will be necessary to adjust the variac occasionally to compensate for line voltage fluctuations.

HUNTING FOR LEAKS

Defects in glass seals, small cracks, pinholes, etc., are detected directly with the sparker. When the electrode is passed near the leak a bright spot will indicate its position.

Leaks in all-metal systems and in glass-metal seals cannot be detected by means of the sparker; however, if there is a glass section in the system being pumped, the sparker may be used as follows:

Providing the leak is not so large as to preclude a discharge fluorescence, the sparker is held near the glass section so that the discharge can be observed, and the suspected places swabbed or brushed with carbon tetrachloride. Acetone or methyl alcohol may also be used, but since these are inflammable, caution should be exercised. Wherever the volatile liquid flows over a small leak the suction and expansion cause it to freeze at the site, thus momentarily closing the leak, whereupon the character of the fluorescence will undergo a marked change. Octoil and other low pressure oils are sometimes used. These materials will have a semi-permanent sealing action so that when applied to a small leak the pressure will drop to a lower value and remain so until the oil is removed with acetone. The use of Octoil (and similar materials) is to be discouraged in most instances because of the difficulty of removing it from small cracks and porosities and its subsequent oxidation and carbonization during bakeout.

The color and character of the fluorescence produced by the sparker is a rough indication of the condition of the vacuum. At relatively high pressures (down to 10^{-2} mm Hg) the glow will be bright, with a "snaky" character and a pink or violet color indicating the presence of air. As the pressure drops the glow becomes more diffuse, paler and more blue (less red) with falling pressure. The blue color is generally attributed to CO_2 and water vapor present in and on glass and metal parts which have not been thoroughly baked or outgassed. At pressures in the neighborhood of 10^{-4} only a faint local fluorescence may be seen, and at 10^{-5} the glow has practically disappeared.

When a system pumps down to pressures of the order of 10^{-5} mm Hg and does not improve on continued pumping, the chances are that something in the system is more or less steadily outgassing due to contamination or chemical action, and/or that there exists a very small leak or porosity. Since no useful visible discharge can be obtained at this pressure, the sparker cannot be used. The ionization gauge is to some degree useful in hunting such leaks, either with the liquids mentioned above, or with illuminating gas or hydrogen. The section under suspicion is covered with a box or other vessel open at the bottom and the gas is introduced. The presence of a leak or of porosity is indicated but not exactly located in this way. After the presence of a leak is definitely established, the open-bottomed vessel is removed and a small jet of gas used as a probe. The presence of a leak is indicated by a sudden increase of pressure.

For leaks which are so small or obscure as to defy all of the above measures, resort is had to the helium leak detector, which is a mass-spectrometer permanently adjusted for helium, and which is connected into the vacuum system in such a way that when the open-bottomed vessel or jet of helium is used (see above), the presence of minute amounts of helium entering a leak can be detected. Detailed instructions for using the helium leak detector are furnished with the instrument.

Moderately small leaks may sometimes be quickly located by covering the suspected area with apiezon Q wax, glyptal, or various mixtures of waxes and resins. This method while suitable for apparatus requiring only moderate vacuum, is to be discouraged for high vacuum tubes, since the presence of the substances mentioned will tend to prevent the attainment of pressures lower than their vapor pressures. Baking can of course not be carried out when these substances are used.

GENERAL INFORMATION ON MATERIALS
MISCELLANEOUS DATA

ALPHABETICAL LIST

- ACETONE:** CH_3COCH_3 ; A volatile solvent used extensively for cleaning and degreasing tube parts and diffusion pumps. It is miscible with water, alcohol and oils. Inflammable.
- ALCOHOLS:** **ETHYL ALCOHOL:** (Ethanol, grain alcohol); A volatile solvent used for cleaning and drying (see methyl alcohol which is preferred for this purpose as it is more nearly anhydrous). Miscible with water in all proportions. Inflammable.
METHYL ALCOHOL: (Methanol, wood alcohol); In pure form, a useful volatile solvent for cleaning and drying tube parts, etc. Preferred to ethyl alcohol because of lower water content and availability. Miscible with water in all proportions. Poisonous and inflammable. Avoid inhaling vapors.
ISOPROPYL ALCOHOL: In pure form, carbon dioxide is bubbled through it in brazing and soldering techniques, to protect copper surfaces from oxidation. At brazing temperatures, the isopropyl alcohol decomposes to form carbon monoxide and hydrogen which provide a reducing atmosphere.
- ALLOYS:** (by trade names) See Table, pp. 49, 50, 51, 52 and 53. See also Monels, p. 68 and Nickel Alloys, p. 69.
- ALUMINUM:** In pure form (Alcoa 2S), a ductile metal having good electrical conductivity, sometimes used in tubes, especially in gas discharge devices because of its freedom from sputtering. Difficult to outgas because of its low melting point (660°C). Aluminum alloys have a wide range of mechanical properties.
- APIEZON:** A trade name for various products including vacuum waxes, greases and plastic cements, made in England and sold in the United States by the J. G. Biddle Company of Philadelphia. Apiezon L is a low viscosity grease for regular stopcock or ground joint use; Apiezon M and N are for use where higher temperatures are encountered. Apiezon Q is a plasticene-like substance used as a temporary vacuum seal and for hunting leaks etc.

TABLE OF ALLOYS

| Alloy No. or Designation Manufacturer* | ADVANCE | ALUMEL | BALCO | BERYLLIUM COPPERS | | | | BRASSES | | | | CALIDO |
|--|----------------|---------------------------|----------------|--|-------------------|-----------------------------------|----------------|----------------|------------------------------|----------------|-------------------------|--------|
| | | | | DH | H | WBD | 25 | 40 | 50 | Low | Low Rich | |
| Composition Percent | Ni 43 Cu 57 | Ni 94 Fe 0.5 Mn 2.5 | Ni 70 Fe 30 | Be 2 Co trace Cu bal. | Same as No. 25 | Be 0.50 Co 1 Ag 1 Cu bal | Cu 80 Zn 20 | Cu 90 Zn 10 | Cu 60 Zn 39.25 Sn 0.75 | Cu 85 Zn 15 | Ni 59 Cr 16 Fe 25 | |
| Specific Gravity | 8.9 | | | | | | 8.6 | 8.8 | | 8.75 | | |
| Resistivity - ohms cir. mil./ft. | 294 | 177 | 120 | | | | | | | | 600 | |
| Temperature Coefficient of Resistivity | ± 0.00002 | | 0.0045 | | | | | | | | 0.00012 | |
| Electrical Conductivity Percent of Copper | | 0.0014 | | 12-15 ⁽¹⁾ 25-29 ⁽²⁾ | | 60-65 ⁽²⁾ | 33 | 44 | 25 | 38 | | |
| Approximate Melting Point °C | 1210 | 1380 | 1425 | 900 | | 950 | 1004 | 1050 | 899 | 1030 | 1400 | |
| Coefficient of Linear Expansion 10-6 per C | 14.9 | 12.0 | 15.0 | | | | 19.1 | 18.4 | 21.2 | 18.7 | 13.0 | |
| Tensile Strength 1000 psi 20°C | 60 135 | 85 | 80 | 150-180 | | 90-100 | 43 | 37 | 54 | 42 | 90 | |
| Magnetic | No | Yes | Yes | No | | No | No | No | No | No | | |
| Relative Machinability | Good | Good | Yes | Good | | Good | Good | Good | Good | Good | | |
| Soft Soldering | Yes | | | Yes | | Yes | Yes | Yes | Yes | Yes | | |
| Hard Soldering | Yes | Yes | | Yes | | Yes | Yes | Yes | Yes | Yes | | |
| Spot Welding | Yes | Yes | | Poor | | Poor | Fair | Fair | Yes | Fair | | |

Source of data (see Mfrs. below)

(1) unheat-treated

(2) heat-treated

*Manufacturers: B - Beryllium Corp., Reading, Pa.

DH - Driver-Harris Co., Harrison, N. J.

H - Hoskins Mfg. Co., Detroit, Michigan

WBD - Wilbur B. Driver Co., Newark, N. J.

P - C. E. Phillips and Co., Detroit, Michigan

TABLE OF ALLOYS

| Alloy | CHROMAX | CHROMEL | CHROMEL | CHROMEL | CHROMEL | CHROMEL | CHROMEL | CHROMEL | CLIMAX | COMET | CONSTANTAN | COPEL | CUPRON |
|---|--------------------------|----------------|-------------------------|-----------------------------|----------------|------------------------|------------------------------|----------------------|----------------|----------------|----------------|----------------|----------------|
| No. or Designation | | A | C | D | H | P | | | | | SEE ADVANCE | SEE ADVANCE | |
| Manufacturer* | DH | H | H | H | H | H | H | DH | DH | DH | H | H | WBD |
| Composition Percent | Ni 35 Cr 20 Fe bal | Ni 80 Cr 20 | Ni 64 Cr 11 Fe 25 | Ni 35 Cr 18.5 Fe 46.5 | Ni 90 Cr 10 | Ni 25 Fe 74 Mn 1 | Ni 30 Cr 4.75 Fe 65.25 | Ni 44-45 Cu 55-60 | Ni 45 Cu 55 | Ni 45 Cu 55 | Ni 45 Cu 55 | Ni 45 Cu 55 | Ni 45 Cu 55 |
| Specific Gravity | 7.95 | 8.4 | 650 | 600 | 425 | 550 | 8.15 | 8.9 | | | | | |
| Resistivity - ohms cir. mil/ft. | 600 | 650 | | | | | 570 | 302 | | | | 298 | 294 |
| Temperature Coefficient of Resistivity | | | | | | | | | | | | | |
| 15-35°C | | 0.000219 | 0.00026 | 0.000397 | 0.00032 | | 0.00088 | ± 0.00001 | | | | Nil | ± 0.00002 |
| 20-100°C | | | | | | | | | | | | | |
| 20-500°C | 0.00031 | | | | | | | | | | | | |
| 20-900°C | | | | | | | | | | | | | |
| Approximate Melting Point °C | 1380 | 1400 | 1400 | 1400 | 1400 | 1415 | 1480 | 1190 | | | | 1290 | 1290 |
| Coefficient of Linear Expansion × 10 ⁻⁶ Per °C | 15.8 | 13.6 | 13.2 | 13.3 | 13.1 | 17.1 | 15.0 | | | | | 14.4 | 14.9 |
| 15-35°C | | | | | | | | | | | | | |
| 20-100°C | | | | | | | | | | | | | |
| 20-500°C | | | | | | | | | | | | | |
| 20-1000°C | | | | | | | | | | | | | |
| Tensile Strength × 1000 psi (20°C) | 70 150 | 95 165 | 90 165 | 85 165 | 95 165 | 75 | 75 160 | 120 | | | | 60 | 88 |
| Annealed | | | | | | | | | | | | | |
| Hard drawn | | | | | | | | | | | | | |
| Magnetic | | No | Slightly | Slightly Tough | No | Yes | Yes | No | | | No | No | No |
| Relative Machinability | | Fair | | | | | | Good | | | Good | Good | Good |
| Soft Soldering | | No | | | | | | Yes | | | Yes | Yes | Yes |
| Hard Soldering | | Yes | Yes | Yes | Yes | | | Yes | | | Yes | Yes | Yes |
| Spot Welding | | Yes | Yes | Yes | Yes | Yes | | Yes | | | Yes | Yes | Yes |

Sources of Data (see Mfrs. below)

*Manufacturers: DH - Driver-Harris Co., Harrison, N. J.
H - Hoskins Mfg. Co., Detroit, Michigan
WBD - Wilbur B. Driver Co., Newark, N. J.

TABLE OF ALLOYS

| Alloy | DRIVER | DRIVER | DRIVER | DRIVER | DRIVER | DRIVER | DRIVER | DRIVER | EVANOHM | EUREKA | EXCELLO | GERMAN SILVER | IDEAL | INCONEL | INVAR | |
|--|---------------|---------------|----------------|----------------|--|--------|--------|--------|---------|--------|------------------------------------|-------------------------------------|-------------|--|--------------------------|--|
| No. or Designation | No. 30 | No. 60 | No. 90 | No. 180 | | | | | | | | | See Advance | | | |
| Manufacturer* | WBD | WBD | WBD | WBD | | | | | | | HMA | | DH | INCO | | |
| Composition Percent | Ni 2 Cu 98 | Ni 6 Cu 94 | Ni 12 Cu 88 | Ni 22 Cu 78 | Ni 74.5 Cr 20 Al 2.75 Cu 2.75 | | | | | | Ni 85 Cr 14 Fe 0.5 Mn 0.5 | Cu 60 Zn 25 Ni 15 Variable | | Ni 79.5 Cr 13.0 Fe 6.5 Si 0.25 Mn 0.25 Cu 0.2 | Fe 64 Ni 36 Mn 0.5 | |
| Specify Gravity | | | | | | | | | | | | | | | | |
| Resistivity - ohms cir. mil./ft. | 30 | 60 | 90 | 180 | 800 | | | | | | 550 | | | 9.03 590 | 8.0 | |
| Temperature coefficient of Resistivity | 0.0011 | 0.0005 | 0.0004 | 0.00016 | ± 0.00002 | | | | | | 0.00016 | | | | | |
| Approximate Melting Point °C | 1100 | 1100 | 1100 | 1100 | 1350 | | | | | | | | | 1390-1430 | 1495 | |
| Coefficient of Linear Expansion/°C | 16.5 | 16.3 | 16.1 | 16.1 | 14.0 | | | | | | | 18.4 | | 11.5 | 0.8 | |
| Tensile Strength × 1000 psi (20°C) | 48 | 50 | 51 | 62 | 88 | | | | | | 95 | | | 85 | | |
| Magnetic | No | No | No | No | No | | | | | | | No | | No | Yes | |
| Relative Machinability | Good | Good | Good | Good | Good | | | | | | | Good | | Tough | Fair | |
| Soft Soldering | Yes | Yes | Yes | Yes | Yes | | | | | | | Yes | | No | Yes | |
| Hard Soldering | Yes | Yes | Yes | Yes | Yes | | | | | | | Yes | | Yes | Yes | |
| Spot Welding | Yes | Yes | Yes | Yes | Yes | | | | | | | Yes | | Yes | Yes | |

Sources of data (see Mfrs. below)

*Manufacturers: DH - Driver Harris Co., Harrison, N. J.

WBD - Wilbur B. Driver Co., Newark, N. J.

INCO - International Nickel Co., New York, N. Y.

HMA - Handbook of Metals and Alloys

TABLE OF ALLOYS

| Alloy No. of Designation | KANTHAL | MANGANESE Bronze | MANGANIN (See Evanohm) | MONELS See table p. 68 | MUNTZ Metal | NICHROME I | NICHROME II | NICHROME III | NICHROME IV | NICHROME V |
|---|-----------------------------|--|------------------------|------------------------|---------------------------------|-------------------------|---------------------------------|------------------|------------------------------------|----------------|
| Manufacturer* | J | P | DH; WBD | | P | DH | DH | DH | DH | DH |
| Composition Percent | Cr 20-30 Al 5 Fe bal. | Cu 59 Zn 39 Sn 1 Fe 1 Mn tr. | Cu 87 Mn 13 | | High Brass Cu 61 Zn 39 | Ni 61 Cr 15 Fe 24 | Ni 66 Cr 22 Fe 10 Mn 2 | Ni 85 Cr 15 | Ni 80 Cr 20 See Chromel A | Ni 80 Cr 20 |
| Specify Gravity | 9.56 | | 8.192 | | | 8.247 | | | 8.5 | 8.412 |
| Resistivity - ohms cir. mil/ft. | 850 | | 290 | | | 675 | 660 | 540 | 620 | 650 |
| Temperature Coefficient of Resistivity | | | ± 0.000015 | | | 0.00017 | 0.00015 | 0.000187 | 0.000179 | 0.00013 |
| Electrical Conductivity Percent of Copper | | 25 | | | 28 | | | | | |
| Approximate Melting Point °C | (1) | 888 | 1020 | | 910 | 1350 | 1370 | 1390 | 1390-1400 | 1400 |
| Coefficient of Linear Expansion × 10 ⁻⁶ Per °C | | | 18.7 | | 20.8 | 17.0 | 13.0 | 13.2 | | 17.0 |
| Tensile Strength × 1000 psi (20°C) | | 60 | 40 90 | | 57 | 95 175 | 100 | 110 | 120 | 100 200 |
| Magnetic Relative Machinability | | No Good | No Good | | No Fair | Yes Fair | Yes Fair | Slightly Fair | No Fair | No Fair |
| Soft Soldering | | Yes | Yes | | Yes | Difficult | No | No | No | No |
| Hard Soldering | | Yes | Yes | | Yes | Difficult | Difficult | Yes | Poor | Poor |
| Spot Welding | | Fair | Yes | | Fair | Yes | Yes | Yes | Yes | Yes |

Source of data (see Mfrs. below)

(1) Safe operating temperature - 1150-1350 °C.

*Manufacturers: DH - Driver-Harris Co., Harrison, N. J.
 J - C. O. Jelliff Mfg. Corp., Southport, Conn.
 WBD - Wilbur B. Driver Co., Newark, N. J.
 P - C. E. Phillips and Co., Detroit, Michigan

TABLE OF ALLOYS

| Alloy No. or Designation | NICKEL See Table, p. 69 | NICKEL-SILVER | | PHOSPHOR BRONZE | | RAYO | SILICON BRONZE | STAINLESS STEELS See Table p. 83 | TOPHET | | |
|---|----------------------------------|-------------------------|-------------------------|---------------------------|-----------------------------|----------------|-----------------------|---|---------------------------------|---------------------|-------------------------|
| | | 18 percent | 18 percent | No. 16 | No. 28 | | | | Reg. | A | C |
| Manufacturer* | | INCO | INCO | PB | PB | | P | | WBD | WBD | WBD |
| Composition Percent | | Cu 65 Ni 18 Zn 17 | Cu 55 Ni 18 Zn 27 | Cu 95.7 Sn 4 P 0.30 | Cu 89.85 Sn 10 P 0.15 | Ni 85 Cr 15 | Cu 96 Zn 1 Si 3 | | Ni 61 Cr 10 Fe 26 Mn 3 | See Chromel A | Ni 60 Fe 28 Cr 12 |
| Specific Gravity | | 8.69 | | 8.9-9 | | 575 | | | 644 | | 8.19 |
| Resistivity - ohms cir. mil/ft. | | | | | | | | | | | |
| Temperature Coeffi- cient of Resistivity | 15-35 °C | | | | | | | | | | |
| | 20-100 °C | | | | | 0.000102 | | | | | |
| | 20-500 °C | | | | | | | | | | |
| 20-900 °C | | | | | | | | | | | |
| Electrical Conductivity Percent of Copper | | | | 16 | 11 | | 7 | | | | |
| Approximate Melting Point °C | | 1055 | | 1050 | 1000 | 1410 | 1025 | | | | 1350 |
| Coefficient of Linear Expansion × 10 ⁻⁶ Per °C | 15-35 °C | | | | | | | | | | |
| | 20-100 °C | | | 17.8 | | | 18.0 | | | | |
| | 20-500 °C | | | | | | | | | | |
| 20-1000 °C | | | | | | | 60 | | | | |
| Tensile Strength at 20 °C × 1000 psi | Annealed Hard drawn | | | 40-55 100 | 58-73 120-133 | 95 | | | | | |
| Magnetic Relative Machinability | | No Good | No Good | No Good | No Good | | No | | | | |
| Soft Soldering | | Yes | Yes | Yes | Yes | | | | | | |
| Hard Soldering | | Yes | Yes | Yes | Yes | | | | | | |
| Spot Welding | | Yes | Yes | Yes | Yes | | | | | | |

*Manufacturers: PB - Phosphor Bronze Corp., Philadelphia, Pa.
WBD - Wilbur B. Driver Co., Newark, N. J.
INCO - International Nickel Co., New York, N. Y.
P - C. E. Phillips and Co., Detroit, Michigan

ASBESTOS: A heat resistant mineral fiber. It is commercially combined with more or less small amounts of organic binders and other materials and sold as asbestos paper, textiles, millboard, etc. Transite is asbestos combined with portland cement. Asbestos products are not suitable for internal tube structures but are useful in glass-blowing and brazing procedures.

BAKELITE: A trade name for various phenolic and other synthetic resins usually combined with paper, textiles, etc. Not to be used in vacuum tubes because of its high vapor pressure and low heat resistance.

BARIUM AND STRONTIUM: As carbonates converted in vacuum to oxides, copious thermionic emitters used as cathode coating in so-called oxide coated cathodes. (See pp. 36 and 41.)

Metallic barium is also used extensively as a getter (residual gas clean-up) material in the form of KIC-Barium wire (Kemet Laboratories, Cleveland, Ohio). This is made by filling an 0.080" iron tube with liquid barium in vacuum, drawing down the composite wire thus formed to 0.020" - 0.030" and then grinding one side flat to a thickness of about 0.003" of iron over the barium core. This wire may be heated by the passage of current through it, or, if suitably disposed, by induction heating. The getter is degassed by heating to about 750 °C and the barium diffuses through the thin side of the wire when the temperature reaches about 850 °C (Ehrke and Slack, Journal of Applied Physics 11, 129 (1940)). One centimeter of 0.020" getter wire mounted on 0.020" nickel leads yielded on an average 2.35 mg of barium under the flashing conditions used. Barium and alloys are also used in the form of pellets or buttons. See p. 43.

BENZENE: A volatile inflammable solvent used for degreasing but not recommended because of the toxicity of its vapors. (See trichlorethylene.)

BERYLLIUM COPPER: A heat-treatable copper-rich alloy used for making springs, contacts, diaphragms, bellows, etc. It is used for certain thermionic tube elements because of its good secondary emission properties where required, such as in electron multipliers. Should be vacuum fired for outgassing as the beryllium content is very sensitive to oxidation by the small amounts of oxygen and/or water vapor present in the tank hydrogen used in the furnaces. See Table p. 49.

BERYLLIUM OXIDE: A refractory ceramic material (mp 2570 °C) sometimes used in tube structures. The powder is very poisonous so that ceramics containing beryllium oxide should be ground only under an efficient hood.

BRASSES and BRONZES: Strictly speaking the brasses are copper-zinc alloys, while the

bronzes are copper-tin alloys. However, in practice, many of the brasses contain various other metals, and many commercial bronzes contain zinc and other metals. Common alloys containing zinc, cadmium, lead, antimony or bismuth should be used in thermionic tubes with caution because of the low vapor pressures of these materials. Brasses containing zinc etc. should not be run through the regular hydrogen furnaces, nor should they be vacuum fired, as the zinc and other low melting point metals mentioned above vaporize easily and not only contaminate the furnace walls but change the compositional integrity of the alloy. Brass is useful for making diffusion (except mercury diffusion) pump parts, special valves, ports, fittings, etc. on high speed kinetic systems. (See Table of Alloys, p. 50.)

CADMIUM: (See BRASSES above); Cadmium plated parts should not be tolerated in vacuum tubes which are to be outgassed with heat, because of the high vapor pressure of cadmium. (See also remarks under SOLDERS, also table, "Vapor Pressure of Metals", p. 89.)

CARBON: (Graphite); Pure carbon is difficult to outgas. The minimum temperature for removal of most of the sorbed gas appears to be in the neighborhood of 1800°C. However, colloidal carbon in distilled water (Aquadag) is useful for coating the inside of vacuum tubes, as a shielding electrode, with subsequent baking at 450°C. It is sometimes advantageous to mix three or four percent by volume of sodium silicate with the Aquadag to improve its adhesion to glass. Carbon is also useful in making jigs for glassblowing and air brazing, either with torch or induction heating.

CARBON DIOXIDE: CO_2 ; As "Dry Ice", a useful refrigerant for pumping traps. Ordinary tank carbon dioxide bubbled through isopropyl alcohol is useful as a protective atmosphere for the inside of copper tubes, cavities, or other vacuum vessels to be brazed or heat treated. The alcohol saturated gas is fed into the vessel and allowed to bleed out through a small opening at only slightly above atmospheric pressure. In closing up a magnetron, for example, this mixture is fed through, and does no harm to the unactivated barium-strontium cathode coating. (See ALCOHOL Isopropyl.)

CARBON TETRACHLORIDE: CCl_4 ; A volatile, heavy, noninflammable solvent for cleaning and degreasing. Should always be used with adequate ventilation as its vapors are toxic. Carbon tetrachloride is useful for hunting leaks in vacuum systems (see special section on this subject, p. 46) with the sparker, owing to its noninflammable nature.

CEMENTS and ADHESIVES: See Tables, pp. 57 and 58.

CERAMICS: (See "High Vacuum Technique", J. Yarwood, 2 ed. 1945, p. 106; also "Special Ceramic Materials" General Ceramics and Steatite Corp., Keasbey, N. J. Pamphlet 1947.) Also see special section herein "Treatment of Ceramic Insulators", below and Table p. 56A. At this date a much used ceramic material for making tube insulators and spacers is a natural magnesium silicate or soapstone of high purity (American Lava Corporation, Lava Grade 1137) which in its unfired state may be readily machined to close tolerances. It has a shrinkage of about 2 percent on firing. The properties of this and other materials are given in the table, p. 56A.

TREATMENT OF CERAMIC INSULATORS (Lava No. 1137 - American Lava Corporation)

This material is a domestic natural talc or soapstone, low in iron oxide and suitable for vacuum tube insulators. It can be obtained in prisms up to 20 cubic inches with the longest dimension not over 3 inches; the maximum size being $2\frac{1}{2}'' \times 2\frac{1}{2}'' \times 3''$. Articles made from it should have a maximum wall thickness of $\frac{1}{4}''$.

Lava can be machined like wood or brass with standard machine tools. After machining a firing operation is necessary to harden the material.

During the firing operation, chemically bound water is driven off and the material undergoes volume changes (linear shrinkage 2 percent, see below), which necessitates careful heating and cooling.

The pieces are placed in a cool furnace protected against direct flame by boxes or baffles. The rate of heating should not exceed 260°C an hour. For sections above $\frac{1}{2}''$ the rate should be reduced considerably, to approximately 150°C an hour. The maximum or curing temperature is between 1010°C and 1093°C . This maximum temperature should be held 15 minutes for $\frac{1}{4}''$ sections and 45 minutes for $\frac{3}{4}''$ sections, with intermediate sizes held in proportion. After this period, the heat is shut off, and the furnace allowed to cool before opening. The fired pieces are taken from the furnace close to room temperature. Firing schedules may be arranged so that cooling takes place overnight with the furnace door closed.

Hairline cracks are sometimes noticed in fired pieces. Some of these are unavoidable and are caused by cracks in the original stone. Large cracks, on the other hand, are usually caused by too rapid heating.

Lava does not change in size until a heat of 870°C is reached, at which temperature it begins to shrink rapidly. Insulators for vacuum tubes should be fired in dry hydrogen at 1000°C subsequent to the air firing operation. Air fired lava is light brown in color, while hydrogen fired lava is likely to be white or very pale.

**PROPERTIES OF VARIOUS CERAMIC AND REFRACTORY BODIES
FROM MANUFACTURERS' DATA**

| Type Body | STEATITES | | | | | | | | | | | ALUMINAS | | | | | CORDIERITES | | | | ZIRCON PORCELAIN |
|--|------------------|-----------------------|----------------------|--|------------------------|----------------------|-----------------------|---------------------------------|-----------------------|-------------------|---------------|------------------|-------------------------------------|----------------------|-----------------------|----------------------|----------------------|---------------------------------------|----------------|-----------------|------------------|
| Body Number | 35 | 196 | 197 | 211 | 228 | 2532 | 2858 | 3030 | 302 | 29 | SI-5 | AD-177 | AD-178 | AI-200 | 491 | 393 | M-244A | 202 | 400 | A-23 | ZI-5 |
| Manufacturer | Am. Lava | Am. Lava | Am. Lava | Am. Lava | Am. Lava | General Ceramics | General Ceramics | General Ceramics | Globe Union | H. L. Crowley | D. M. Steward | General Ceramics | General Ceramics | Coors | Am. Lava | Am. Lava | General Ceramics | Am. Lava | Globe Union | Mich. Porcelain | Coors |
| Specific Gravity | 2.5 | 2.6 | 2.6 | 2.7 | 2.6 | 2.7 | 2.5 | 2.8 | 0-0.005 | 0-0.05 | 2.7 | 3.2 | 2.5 | 3.4 | 3.3 | 2.4 | 2.3 | 2.1 | 0-0.007 | 7-8 | |
| Water Absorption percent | 0-0.05 | 0-0.05 | 0-1 | 0.08 | 0-0.05 | 0-0.05 | | 0-0.05 | | | 0 | 0-0.5 | 13.6 | | 0-0.05 | 12-18 | 0-0.5 | 10-15 | | | |
| Colors Standard Alternate | White Gray-Brown | White | White Gray-Brown | White | White | | | | | | | | | | | White | | Gray-Brown Lt. Gray | | | |
| Softening Temp. °C | 1450 | 1440 | 1445 | 1400 | 1440 | | | | | 1325 | 1286 | | | | 1500 | 1650 | | 1430 | | | |
| Safe Limit for Constant Temp. °C | 1000 | 1000 | 1000 | 1000 | 1000 | 980 | | 980 | | 1000 | | | | | 1350 | 1400 | | 1250 | | | |
| Hardness (Moh's Scale) | 7.5 | 7.5 | 7.5 | 7.5 | 7.5 | 7.5 | 7.5 | 8.0 | | 7.5 | 7.0 | 9.0 | 9.0 | | 9.0 | | 8.5 | 7.0 | | | |
| Linear Coefficient of Thermal Expansion per °C (x 10 ⁻⁶) | 6.9 | 7.3 | 7.7 | 7.5 | | 6.5 | 8.7 | 6.0 | | 6.3 | | 5.1 | 2.2 | 6.67 | 5.4 | 5.1 | 2.03 | 1.6 | 1.9 | | 3.7 |
| | 25-100°C | | | | | 7.8 | | 7.3 | | | | 6.3 | 5.7 | 7.26 | | | | | | | |
| | 25-700°C | 8.7 | 8.6 | 10.2 | 9.3 | 9.0 | | | 8.3 | 8.3 | 8.13 | | | 7.82 | 7.5 | 7.2 | 3.1 | 2.8 | 3.1 | 2.6 | 4.7 |
| | 25-800°C | | | | | 8.2 | | | | | | | | 8.47 | | | | | | | |
| Strengths psi x 10 ³ | 8.5 | 10 | 8.5 | 7.5 | 10 | 9.5 | 6.8 | 9 | | 10 | 7.2 | | | 13 | 70 | 11 | 4.0 | 3.5 | | | 9.0 |
| Tensile | 75 | 85 | 75 | 65 | 85 | 81.5 | 74 | 80 | | 75 | 96 | | | 108 | 35 | 10 | 35 | 40 | | 45 | 85 |
| Compressive | 18 | 20 | 20 | 18 | 20 | 19 | 17 | | | 20 | 10 | | | 18 | | | | 8 | 13 | 4.0 | |
| Flexural | | | | | | | | | | | | | | | | | | | | | |
| Impact Resistance (Charpy) | 4.5 | 5 | 4.5 | 4.5 | 5 | 2.30 | 1.6 | 1.9 | | | | 2.1 | 1.0 | | 3 | 0.9 | 0.9 | 2.5 | | | |
| Thermal Conductivity (cgs) | 0.006 | 0.006 | 0.006 | 0.006 | 0.006 | 0.006 | | 0.006 | | | | | 0.004 | | 0.004 | 0.003 | 0.003 | 0.003 | | | |
| Dielectric Strength 60 cy v/mil | 225 | 240 | 210 | 240 | 240 | 240 | 280 | 200 | 247 | | 235 | 160 | | | 95 | 100 | 100 | 100 | | | 250 |
| Volume Resistivity ohms/cc | 25°C | >10 ¹⁴ | >10 ¹⁴ | >10 ¹⁴ | >10 ¹⁴ | >10 ¹⁴ | 12 x 10 ¹³ | >10 ¹⁴ | | >10 ¹⁴ | | | 33 x 10 ¹³ | | >10 ¹⁴ | >10 ¹⁴ | 3 x 10 ¹⁰ | >10 ¹⁴ | | | |
| | 100°C | 21 x 10 ¹¹ | 1 x 10 ¹³ | 81 x 10 ¹² | >10 ¹⁴ | >10 ¹⁴ | | | | | | | | | 21 x 10 ¹² | 5 x 10 ¹² | | 3 x 10 ¹³ | | | |
| | 300°C | 6 x 10 ⁷ | 18 x 10 ⁸ | 25 x 10 ⁹ | 9 x 10 ¹² | 1 x 10 ¹³ | | 31 x 10 ¹¹ | | | | | 10 ¹² | | 25,000 | 10 ¹⁰ | | 2 x 10 ¹⁰ | | | |
| | 500°C | 32 x 10 ⁴ | 9 x 10 ⁶ | 88 x 10 ⁶ | 35 x 10 ⁹ | 2 x 10 ⁹ | | 18 x 10 ⁹ | | | | | 10 ⁹ | | 35 x 10 ⁸ | 75 x 10 ⁶ | | 9 x 10 ⁷ | | | |
| | 700°C | 23 x 10 ³ | 5 x 10 ⁵ | 42 x 10 ⁵ | 48 x 10 ⁷ | 17 x 10 ⁶ | | 16 x 10 ⁸ | | | | | 13 x 10 ⁸ | 52 x 10 ⁶ | 9 x 10 ⁷ | 36 x 10 ⁵ | | 3 x 10 ⁶ | | | |
| | 900°C | 7 x 10 ³ | 7 x 10 ⁴ | 68 x 10 ⁴ | 25 x 10 ⁶ | | | | | | | | | | 35 x 10 ⁵ | 56 x 10 ⁴ | | 35 x 10 ⁴ | | | |
| Te Value ⁽¹⁾ °C | 440 | 640 | 840 | 1000 | | 700 | | 700 | | | | 700 | 700 | | 980 | 835 | 700 | 780 | | | |
| Dielectric Constant 50 percent Rel. Humidity | 60 cy | 6.1 | 5.9 | 6.3 | 5.8 | 6.3 | 5.85 | 6.5 | 6.3 | 5.8 | 5.9 | 6.4 | 8.4 | 5.6 | | 5.4 | 5.4 | 5.0 | 5.4 | | |
| | 1 mc | 5.9 | 5.8 | 6.0 | | | | | | | | | | | | 5.4 | | 5.0 | | | |
| | 10 mc | 5.8 | 5.7 | 5.8 | 5.7 | | | | | | | | | | | 5.3 | | 4.9 | | | |
| | 100 mc | 5.7 | 5.6 | 5.7 | 5.7 | | | | | | | | | | | | | | | | |
| Power Factor percent | 60 cy | 0.015 | 0.0022 | 0.002 | 0.0004 | 0.0012 | 0.0012 | 0.05 | 0.00065 | 0.06 | 0.07 | 0.003-0.004 | 0.001 | 0.0002 | 2KC 0.079 | 0.0008 | 0.0025 | 0.012 | 0.004 | | |
| | 1 mc | 0.0035 | 0.0021 | 0.0012 | | | | | | | | | | | | | 0.0025 | 0.003 | | | |
| | 10 mc | 0.003 | 0.0015 | 0.0009 | 0.0003 | | | | | | | | | | | | 0.002 | 0.003 | | | |
| | 100 mc | 0.0028 | 0.0014 | 0.0009 | 0.0003 | | | | | | | | | | | | | 0.003 | | | |
| Loss Factor ⁽²⁾ percent | 60 cy | 0.09 | 0.013 | 0.0126 | 0.0023 | | 0.007 | 0.33 | 0.004 | 0.40 | 0.41 | 0.016 | 0.0084 | 0.012 | 2KC | 0.014 | 0.065 | 0.020 | 3.0 max | | |
| | 1 mc | 0.021 | 0.012 | 0.0072 | 0.0017 | | | | | | | | | | | 0.014 | | 0.015 | | | |
| | 10 mc | 0.017 | 0.008 | 0.0058 | 0.0017 | | | | | | | | | | | 0.011 | | 0.015 | | | |
| | 100 mc | 0.016 | 0.008 | 0.0051 | 0.0017 | | | | | | | | | | | | | 0.015 | | | |
| Capacity Change per °C (ppm) | +160 | +160 | +160 | +120 | +160 | | | | | | | | | | +160 | +100 | | | | | |
| Remarks | | | | Low loss material; for tube spacers etc. | High strength material | General grade. | | Low losses at high frequencies. | Low water absorption. | | | Dense body. | Porous body; high water absorption. | | | Porous body. | Low expansion. | Low expansion; withstands heat shock. | Low expansion. | | |

| Type Body | ZIRCON PORCELAINS | | | | PORCELAINS | | | | MULLITE FORSTERITE | | | NATURAL LAVA | STONES Mg Si | Al Si | MAGNESIUM SILICATE | TITANIUM DIOXIDES | | | TITANATE | QUARTZ | GLASS | |
|--|-------------------|-----------------------|----------------------|----------------|-----------------|-----------------------|---------------------|----------------------|-----------------------|----------------------|------------------------------|-----------------------|----------------------|----------------------|--------------------|-------------------|-------------------|-----------------|----------|-----------------------|-------------------|---------------|
| Body Number | M-81-A | 475 | ZI-4 | 452 | 527 | BV-572 | BV-580 | 157 | 203 | 243 | 1137 fired | Gr. M | Gr. A | 222 | K15 | K90 | 192 | 193 | K-1500 | (fused) | 7740 | |
| Manufacturer | General Ceramics | Am. Lava | Coors | Globe Union | Wisc. Porcelain | General Ceramics | General Ceramics | Am. Lava | Am. Lava | Am. Lava | Am. Lava | Am. Lava | Am. Lava | Am. Lava | Am. Lava | General Ceramics | General Ceramic | Am. Lava | Am. Lava | General Ceramics | | Corning Pyrex |
| Specific Gravity | 3.1 | 3.7 | 0 | | | 2.45 | 2.35 | 2.38 | 2.1 | 2.8 | 2.8 | 2.8 | 2.3 | 2.0 | 5.5 | 7.2 | 4.0 | 4.0 | 8.9 | 2.653 | 2.23 | |
| Water Absorption percent | 0-0.5 | 0-0.5 | 0 | 0-0.005 | 0-0.05 | 0-0.5 | 0-0.5 | 0-0.5 | 10-15 | 0-0.05 | 2.5 | 1.0 | 2.5 | 14-18 | | | 0-0.06 | 0-0.06 | | 0 | 0 | |
| Colors Standard Alternate | | | | White to Ivory | | | | White | White | Buff | White (H ₂ fired) | Brown | Pink | Lt. Brown | | | Tan | Blue-Black | | | | |
| Softening Temp. °C | | 1440 | | | | | | 1440 | 1650 | 1440 | 1475 | 1475 | 1600 | 1625 | | | 1450 | 1450 | | 1470 | 820 | |
| Safe Limit for Constant Temp. °C | 1196 | 1000 | | | | 1196 | 1196 | 1000 | 1350 | 1000 | 1200 | 1200 | 1100 | 1300 | | | 1000 | 1000 | | | 230-490 | |
| Hardness (Moh's Scale) | 7.0 | 8.0 | | 7.5 | | 8.5 | 8.5 | 6.5 | 6.0 | 7.5 | 6.0 | 6.0 | 6.0 | 6.0 | | | 8 | 8 | | 7 | | |
| Linear Coefficient of Thermal Expansion per °C (x 10 ⁻⁶) | 25-100°C | 2.95 | | 3.36 | 3.3 | 3.7 | 3.6 | 3.2 | 3.5 | 9.1 | 10.7 | 8.9 | 2.9 | 8.0 | | | 7.3 | 7.3 | | 0.50 | 3.2 | |
| | 25-400°C | 5.15 | | 4.27 | | 4.9 | 4.6 | | | | | | | | | | | | | | | |
| | 25-700°C | | 4.9 | | 4.0 | 5.5 | | 4.3 | 5.0 | 10.6 | 11.6 | 10.5 | 3.6 | 10.0 | | | 8.7 | 8.7 | | 0.546 | | |
| | 25-800°C | | 5.6 | 5.43 | | | | | | | | | | | | | | | | | | |
| Strengths psi x 10 ³ | 70 | 12 | 8-12 | 64-76 | | 4.5 | 3.5 | 50 | 2.0 | 10 | 2.0 | 2.0 | 2.5 | 2.5 | | | 7.5 | 7.5 | | 1.6-7.0 | | |
| Tensile | | 90 | | | 50 | 40 | 38 | 9 | 10 | 85 | 20 | 30 | 20 | 25 | | | 80 | 80 | | | | |
| Compressive | | 22 | 19 | | 7 | | | | 6 | 20 | 7 | 9 | 9 | 8 | | | 20 | 20 | | 190 | | |
| Flexural | | | | | | | | | | | | | | | | | | | | | | |
| Impact Resistance (Charpy) | 1.2 | 5.5 | 3.1 | | | 0.80 | 0.75 | 2.8 | 2.1 | 4.0 | 2.6 | 3.0 | 3.3 | 1.9 | 1.7 | 3.1 | 6.5 | 6.5 | | 0.0024 | 3.1 | |
| Thermal Conductivity (cgs) | | 0.012 | | | | 0.004 | 0.004 | 0.005 | 0.003 | 0.008 | 0.005 | 0.005 | 0.003 | 0.005 | 0.005 | 0.005 | 0.009 | 0.009 | | | | |
| Dielectric Strength 60 cy v/mil | 250 | 250 | 450 | 215 | 200 | 90 | 90 | 200 | 100 | 240 | 50 | 100 | 80 | 50 | 170 | 210 | 100 | 100 | 95 | 500-650 | | |
| Volume Resistivity ohms/cc | 25 °C | >10 ¹⁴ | >10 ¹⁴ | | | 26 x 10 ¹² | | >10 ¹² | >10 ¹⁴ | >10 ¹⁴ | >10 ¹⁴ | >10 ¹⁴ | >10 ¹⁴ | >10 ¹⁴ | >10 ¹⁴ | | >10 ¹² | 10 ⁶ | | >5 x 10 ¹⁸ | 10 ¹⁵ | |
| | 100°C | 56 x 10 ¹³ | 2 x 10 ¹³ | | | 27 x 10 ⁸ | | 7 x 10 ⁷ | 15 x 10 ¹² | 5 x 10 ¹³ | 65 x 10 ¹¹ | 54 x 10 ¹¹ | 6 x 10 ¹¹ | >10 ¹⁴ | | | | | | | 10 ^{8.1} | |
| | 300°C | 10 ¹³ | 55 x 10 ⁷ | | | 57 x 10 ⁶ | 10 ⁷ | 15 x 10 ⁶ | 56 x 10 ⁸ | 7 x 10 ¹¹ | 45 x 10 ⁸ | 5 x 10 ⁷ | 2 x 10 ⁶ | 6 x 10 ¹¹ | | | | | | | | |
| | 500°C | 12 x 10 ⁸ | 55 x 10 ⁷ | | | 38 x 10 ⁵ | 4 x 10 ⁶ | 35,000 | 17 x 10 ⁶ | 12 x 10 ⁹ | 43 x 10 ⁷ | 7 x 10 ⁷ | 5 x 10 ⁶ | 46 x 10 ⁸ | | | | | | | 106.6 | |
| | 700°C | | 14 x 1 | | | | | | | | | | | | | | | | | | | |

CEMENTS AND ADHESIVES

| Name or Trade Name | Composition-Properties | Preparation-Source | Temperature Properties | Application-Remarks |
|--------------------------------------|---|--|--|---|
| ALUNDUM CEMENT | Aluminum oxide and water. A refractory cement; sets on firing. At lower temperatures it may be reworked. | Mix to consistency of thick mud. | Firing temperature 1100° C | Useful for setting nichrome or other wires on furnace muffles, etc. |
| APIEZON Q | Paraffin oil residues with graphite. Modeling-clay consistency. Low vapor-pressure. | Made in England. Obtained from J. G. Biddle Company, Philadelphia. | Not usable beyond 30° C. Liquefies. | Temporary closing of leaks in vacuum systems. Sealing of belljars, etc. |
| BEEWAX AND ROSIN | Equal pts. Beeswax and Rosin. Adheres to clean, cold metal. Can be removed with putty knife. Shrinks on solidifying. Some-what sol. in equal pts. CCl ₄ and eth. alcohol. | Smoking indicates vaporization of beeswax, making it harder. Retempered by addition of more beeswax. | MP 57° C. Softens at 47° C. | Not very strong, but useful for sealing bell jars and for making other temporary seals. Can be applied smoking hot with eye-dropper or spatula. |
| DEKHOTINSKY CEMENT (see SEALSTIX) | Shellac plus 20-40 percent wood tar. Not affected by water, CS ₂ , benzol, benzine, or turpentine. Only slightly affected by ether, chloroform, and the strong mineral acids. Sl. sol. in alcohol. Made in grades: hard, med., soft. | Central Scientific Co. | MP 140° C. Softens at 100° C. | Sticks well to clean, hot (150° C) surfaces. Fairly low vapor-pressure. Useful for making semi-permanent seals in vacuum systems. |
| GLYCOL PHTHALATE (GLYPTAL) | Phthalic anhydride condensed on glycol or glycerol, etc. Long heating causes further curing and hardening (embrittlement). Has low vapor-pressure. | As "Glyptal", General Electric Co., a paint either with or without a pigment (ZnO) plus a solvent such as xylol or toluol. | Do not heat above 150° C. | Adheres well to clean metals. Useful for sealing quartz windows to metal or glass apparatus, and for sealing small leaks. |
| LITHARGE AND GLYCERIN | Lead monoxide (PbO) and glycerin. Inert to water, most acids and alkalis. Irreversible. | The pulv. litharge is first heated thoroughly at 400° C. Mix with pure glycerin to consistency of a paste. | Holds well up to 260° C. | A useful tube basing cement. Not used for vacuum work. |
| PICEIN | A black wax of low vapor-pressure (10 ⁻⁶ mm Hg at 20° C) sol. in benzol and turpentine. Unaffected by alcohol. Immune to short immersion in dichromate cleaning fluid. | Schrader and Ehlers, 239 Fourth Ave., N. Y. C. | One variety softens at 50° C; liquefies at 80° C. Another variety liquefies at 105° C. | Very good electrical insulator (equal to amber if not overheated). Adhesive. Useful for sealing mica windows and for closing small leaks. |
| PLASTER OF PARIS | Calcium sulphate. Irreversible. | Susp. in water to consistency of paste. Salt speeds setting, dil. acetic acid (vinegar) retards it. | | Useful for supporting glass bulbs containing Hg in wood or metal box. Oil or grease on glass prevents sticking. Tube base cement. |

CEMENTS AND ADHESIVES (continued)

| Name or Trade Name | Composition-Properties | Preparation-Source | Temperature-Properties | Application-Remarks |
|-------------------------------------|---|---|---|---|
| MYVAVAX S | Soft hydrocarbon wax with low vapor-pressure. Dark amber color. | Distillation Products, Inc. 1 oz. sticks. | M. P. 72.5°C. Sp. Gr. 0.95. | Adheres well to glass and metal surfaces. Soluble in petroleum ether, CCl ₄ and benzene. Unaffected by acetone, alcohol, or Octoil. |
| SAUERISEN CEMENT INSALUTE CEMENT | A suspension of ceramic powders in sodium silicate solution. Somewhat refractory. Sets very hard. Sol. in boiling water and alkali solutions. Low vapor-pressure. | Central Scientific Co. Thin with sodium silicate solution. (Water glass). Irreversible. | Withstands temps. up to 590°C. | Anchoring structures in tubes. Cementing glass or ceramics to metal. Not vacuum tight. |
| SEALSTIX | A modified DeKhotinsky cement; has lower vapor-pressure and higher strength. | Central Scientific Co. | Not as inflammable as DeK. Has greater working temperature range. | Same as DeKhotinsky cement. |
| SHELLAC (pure orange only) | A natural gum. Sol. in alcohol; has high tensile strength (about 3800 psi). | The gum is held in a cloth bag and soaked in alcohol for many hours. | Polymerized by heat to yield a material with higher softening temp. and less solubility in alcohol. (Complete transformation at 150°C for 3 hours.) | As a varnish in alcohol, and for temporary sealing of small leaks. The dry gum (as sealing wax) is useful for cementing small parts to a faceplate for machining. |
| SILVER CHLORIDE | AgCl. Insoluble in water, alcohol, benzol and some acids. Sol. in solution of sodium thio-sulphate. Most metals are wet by the fused salt. | | M. P. 455°C. | Useful for sealing optical windows. Must be cooled slowly to prevent cracking. |
| ZINC OXYCHLORIDE | Dental cement: 60 percent zinc chloride solution with zinc oxide (ZnO) powder, mixed to consistency of thick paste. The oxide should be calcined to a yellow color to free it of carbonates. Hard and adhesive. | | | |

REFERENCES: Strong: "Procedures in Experimental Physics", Prentice-Hall, Inc., New York 1942

Yarwood: "High Vacuum Technique".

The Handbook of Chemistry and Physics, Chemical Rubber Pub. Co., Cleveland, Ohio.

DATA ON LAVA GRADE 1137 UNFIRED

| Type | Density | Volume | Color | Hardness | Linear shrinkage |
|----------------------------|--------------------|---------------------|-----------------|----------|------------------|
| | lb/in ³ | in ³ /lb | | (Mohs) | Factor |
| Hydrous magnesium Silicate | 0.091 | 11.0 | Vari-mottled | 1 | 1.02 |
| AFTER FIRING | | | | | |
| Anhydrous | 0.102 | 9.76 | Lt. brown-white | 6 | ----- |

CHROME IRON: A general designation for chrome steels having carefully selected expansion coefficients to match those of glasses or ceramics to which direct fusion seals are to be made, for windows in vacuum tubes, etc. (See Table, Expansion Coefficients of Glass and Ceramic Sealing Metals, p. 30.)

CONSTANTAN: Also known as ADVANCE, CUPRON, COPEL, EUREKA and IDEAL; A nickel-copper alloy having a very low temperature coefficient of resistivity (0.00001). It has the composition: nickel-40-45 percent; copper-60-55 percent. (Advance contains 0.5 percent iron.) May be readily soft or hard soldered. Useful for making thermocouples with iron, copper and other metals. (See Table, "Alloys".)

COPPER: For making tube bodies or vacuum vessels, this Laboratory uses a highly purified, vacuum-worked copper designated as "Oxygen-free-High Conductivity" (OFHC) copper. Ordinary electrolytic copper contains enough oxide inclusions so that in firing operations the hydrogen which diffuses into the metal reacts with the oxide to form water vapor. Small cracks and porosities are thus caused since the water vapor is under sufficient pressure to produce rupture at the grain boundaries. Also, for this reason, ordinary copper is likely to become quite brittle after passing through the hydrogen furnace.

Copper can be readily brazed using eutectic silver-copper alloy (BT) or other solders, in the hydrogen furnace. It is difficult to spot weld copper directly. (See FLUXES below.)

FERNICO: A glass sealing metal manufactured by General Electric Company. Its properties are similar to but not identical with Kovar. (See KOVAR.)

FLUXES: This term is used to designate two different classes of materials. Various chemical compounds and mixtures are used in soft and hard soldering and brazing (and sometimes in welding) to keep the surfaces of the metals to be joined clean and free from oxidation. It must be remembered that

practically all chemical fluxes used in soldering and brazing are more or less corrosive, and if not thoroughly removed (see p. 3), tend to undermine the joint in time. (See end of Table, "Soldering and Brazing Alloys and Fluxes", p. 81.) The term is also used to designate certain metals which, in the form of foil or thin sheet, are interposed between two other metals difficult to spot weld directly, such as tungsten to tungsten or tungsten to molybdenum. The usual requirements for such a flux are that it should spot weld easily to both pieces being joined, and that it should have a melting point high enough to meet the conditions of service. Metals commonly used as fluxes in this connection, in the order of increasing melting points are: nickel, platinum, zirconium and tantalum. Thin sheet hard solders can be used as fluxes for spot-welding copper and silver.

GLASSES: Those in most common use in this Laboratory are the hard (borosilicate or Pyrex type) glasses. Occasionally there is a need for a high silica (Vycor) glass, or quartz, and sometimes for a soft glass. It is often required that combinations of these glasses occur in a single envelope; e. g. where seals are to be made with Kovar, tungsten and molybdenum in the same tube, each metal requiring a glass with approximately matching coefficient of expansion if good seals are to be obtained. This requires graded seals consisting of one, two or more intermediate glasses, the number of intermediates required depending on the difference in the thermal expansions of the two outside glasses (see Table, p. 62). To achieve a successful seal between different glasses they must be close together in expansion coefficients over a wide range of temperatures.

Wires which are to be sealed into glass, such as lead wires in presses, must be beaded with the appropriate type of glass before sealing in.

Copper can be sealed to glass, but since it has a larger coefficient of expansion than the glasses used, it must first be turned down in a lathe to a feather edge which can expand or contract with the glass.

Platinum wires and small tubes can be sealed into Corning 707 glass and into soft glass.

Some other alloys which are suitable for sealing into various glasses are as follows: DUMET or COPPERCLAD, a copper covered nickel steel alloy having the required expansion characteristics for sealing into lead and lime glasses. Sometimes used with a borated copper surface. SA-50 ALLOY (Callite) is suitable for use as a lead-in wire through soft glass. It is not copper clad, and is adaptable for mercury switches or vapor tubes as it does not readily amalgamate with mercury. See also KOVAR and FERNICO.

Windows of Corning 7052 glass can be sealed into Kovar. This glass which comes as tubing, is cut in short lengths and in half lengthwise, then

flattened between carbon plates in a furnace. The pieces are then ground and polished on both sides to the required thickness by a lens grinding process, after which they can be sealed to the Kovar on the vertical glass sealing machine. Overheating, which causes bubbles, can sometimes be avoided with the use of induction heating, in which the metal is heated directly, the glass only by conduction. Corning 707 glass can also be used for windows in Kovar frames when, upon cooling, it is under compression.

All windows, as well as most other pieces of glass work, must be thoroughly oven annealed. In the case of assembled tubes, the annealing is accomplished by the baking out process at the start of evacuation; the elements inside the tube are thus protected from oxidation.

The glassblower can very often offer valuable suggestions for the design of tube envelopes, presses, lead-ins and related matters, and should always be consulted when these aspects of tube design are under consideration.

In making seals between different glasses and between glasses and metals such as Fernico, Kovar, tungsten, molybdenum and certain types of glass-sealing ferrous alloys, careful consideration should be given to the stresses that might be set up in such seals. (This applies also to metal-ceramic seals; see p. 28). No stress figures are given here because of variable factors influencing them, such as design of the seal (including direction of compression or tension), glassing technique, subsequent heat treatment and thermal conditions in actual functioning of the finished apparatus. The advice of the glassblowers should be sought when such seals are contemplated.

The following table gives pertinent information on physical and mechanical properties of glasses and data relative to glass-working. (p. 62.)

GOLD: Useful as a brazing material, usually alloyed with copper, nickel and/or other metals. In pure form (24 carat) it can be used to effect the so-called diffusion seal on copper and other metals, in which the clean, accurately machined surfaces to be joined are gold plated, or separated by a thin gold foil, and pressed together at a moderate temperature well below the melting point of gold.

Gold has very low secondary emission.

GREASES, STOPCOCK: (See also APIEZON.) Greases for stopcock use should have vapor pressures of not more than 10^{-4} mm Hg, at 35°C, and should maintain adequate viscosity at this temperature. The grease in most common use (on the low vacuum or forepump side only) in this Laboratory is "Lubriseal" supplied by the A. H. Thomas Company.

PROPERTIES OF SOME CORNING GLASSES

(Manufacturer's 1949 Bulletin B-83)

| Corning Glass No. | 0120 | 3321 | 7050 | 7052 | 7070 | 7720 | 7740 | 7900 | 7900 |
|---|---------------------|---------------------|---------------------|---------------------|---------------------|---------------------|---------------------|--------------------|-----------------------------|
| Type | Potash Soda Lead | Hard Green Sealing | Boro-Silicate | Boro-Silicate | Boro-Silicate | Boro-Silicate | Boro-Silicate | 96 percent Silica | Multiform 96 percent Silica |
| Specific gravity | 3.05 | 2.27 | 2.25 | 2.28 | 2.13 | 2.35 | 2.23 | 2.18 | |
| Forms available (1) | TM | T | T | BMPT | BMPT | BPT | BPSTU | BPTU | M |
| Coefficient of thermal expansion (0-300°C) | 89×10^{-7} | 40×10^{-7} | 40×10^{-7} | 46×10^{-7} | 32×10^{-7} | 36×10^{-7} | 32×10^{-7} | 8×10^{-7} | 8×10^{-7} |
| Operating temperature limit (annealed)°C | 380 | 470 | 440 | 420 | 430 | 460 | 490 | 1090 | 1090 |
| Thermal shock resistance $6'' \times 6'' \times 1/8''(2)''$ °C (approx) | 65 | 135 | 125 | 125 | 180 | 160 | 180 | 1250 | 1250 |
| Thermal stress resistance (3) °C | 17 | 39 | 34 | 34 | 70 | 45 | 48 | 200 | 200 |
| Strain point (4) °C | 400 | 497 | 461 | 438 | 455 | 484 | 515 | 820 | 820 |
| Anneal Point (4) °C | 433 | 535 | 496 | 475 | 490 | 518 | 555 | 910 | 910 |
| Softening point (4) °C | 630 | 780 | 703 | 708 | 1100 | 755 | 820 | 1500 | 1500 |
| Working point (4) °C | 975 | | | 1115 | | 1110 | 1220 | | |
| Impact abrasion resistance (5) | | | | | | | | | |
| Modulus of elasticity, psi | | | | | 4.1 | 3.2 | 3.1 | 3.5 | 3.5 |
| Log ₁₀ of volume resistivity (Data for 25°C are approximate) | 17+ 10.1 8.0 | | 16 8.8 7.2 | 17 9.2 7.4 | 17+ 11.2 9.1 | 16 9.8 7.2 | 15 8.1 6.6 | 17 9.7 8.1 | 17 9.7 8.1 |
| Power factor percent | 0.16 | | 0.33 | 0.26 | 0.06 | 0.27 | 0.46 | 0.05 | 0.05 |
| Dielectric properties at 1 Mc/sec Dielectric constant and 20°C | 6.6 | | 4.9 | 5.1 | 4.0 | 4.7 | 4.6 | 3.8 | 3.8 |
| Loss factor percent | 1.1 | | 1.6 | 1.3 | 0.24 | 1.3 | 2.1 | 0.19 | 0.19 |
| Refractive index (Na D line - 5893A average) | 1.560 | | 1.479 | 1.484 | 1.469 | 1.487 | 1.474 | 1.458 | 1.458 |
| Comments | Lamp tubing | Sealing | Series sealing | Kovar sealing | Low loss electrical | Electrical | General | High temp. clear | High temp. white opaque |

Notes:

- (1) Forms: B - blown ware; M - multiform ware; P - pressed ware; S - plate glass; T - tubing and rod; U - panels.
- (2) Based on plunging sample into cold water after oven heating. Resistance of 100°C means no breakage if heated to 110°C and plunged into water at 10°C.
- (3) Resistance in °C is the temperature differential between two surfaces of a tube or a constrained plate that will cause a tensile stress of 1000 psi on the cooler surface.
- (4) Strain point is the highest temperature from which glass can be cooled haphazardly without becoming permanently strained. Anneal point is the temperature at which internal strains can be quickly relieved. Softening point is the temperature at which the glass begins to deform noticeably under its own weight. Working point is the temperature at which the glass is soft enough for hot working by ordinary methods.
- (5) Relative resistance to sandblasting.

INCONEL: A nonmagnetic alloy of nickel (79.5 percent), chromium (13 percent), and iron (6.5 percent), having fair machinability and good corrosion resistance at high temperatures, used for heating elements and sometimes for tube elements. Brazing to Inconel is accomplished the same as for stainless steel. (See special section on Stainless Steel, p. 15.) (See Alloys.)

INVAR: An alloy consisting of iron (64 percent), nickel (36 percent), having very low coefficient of thermal expansion (1×10^{-6} per °C between 0 and 100°C). (See Alloys.) Invar is magnetic. Nilvar is an alloy having closely similar properties, made by Driver-Harris Co., Harrison, N. J.

IRON AND ALLOYS: (See also special section on Stainless Steel, p. 15.) An especially pure form of iron known as SVEA metal is sometimes used in tube structures. However, because of the low cost, availability and excellent machining properties of COLD-ROLLED (low carbon) steel, much use is made of it in tube structures. (See Materials - Steel, p. 78.) It should be previously vacuum fired. Hydrogen firing causes a blue oxide to form due to the presence of small amounts of oxygen and/or water vapor. Cold-rolled steel can be copper and silver brazed if previously copper or nickel plated. Cadmium plated parts should not be used in vacuum tubes because of the high vapor pressure of cadmium, which should be removed from small parts such as screws by heating to redness in air, then vacuum firing in a tantalum bucket to 1000°C. Cold-rolled steel is much used for making mandrels for winding and annealing heaters in the hydrogen furnace, where its tendency to form oxide prevents the heater wire from adhering. It should not, however, be used to make brazing jigs, because the oxide film is not adequate to prevent sticking of molten brazing materials. (For brazing jigs, stainless steel types 302, 303 or 304 should be used.)

TOOL STEEL (drill rod, ground stock) though not so easily machinable as cold-rolled steel, has the advantage of being heat-treatable to obtain various degrees of hardness and temper, so that it is useful for making punches, dies, mandrels, etc. Iron and steel are not attacked by mercury. All of the iron and steel materials are strongly magnetic, except certain types of stainless steel.

KOVAR: GLASS SEALING METAL: (Stupakoff Ceramic and Mfg. Co., Latrobe, Pa.)

The composition of Kovar is:

| | | |
|-----------|------|---------|
| Nickel | 29 | percent |
| Cobalt | 17 | percent |
| Iron | 53.7 | percent |
| Manganese | 0.3 | percent |

Melting point 1450°C approximate
Coefficient of thermal expansion (after annealing)
30° to 500°C 5.71 to 6.21 × 10⁻⁶/°C

The surface of the Kovar should be smooth, bright and free from defects. Sharp edges of the metal in contact with glass should be avoided. The metal should not be subjected to successive heating in oxidizing and reducing atmospheres or in carbonaceous atmospheres.

After machining and polishing, the piece is degreased, rinsed in methyl alcohol, dried and hydrogen fired to outgas and anneal it at 900°C for 15 to 30 minutes. Where torch soldering operations are required this should be done after hydrogen firing. The Kovar should be previously copper plated at the site of the joint. Kovar may be hydrogen brazed to copper using eutectic silver solder (BT) but this should be done after annealing.

Kovar may be brazed to steels or nickel with pure silver or pure copper in the hydrogen furnace, but in both cases it should be previously copper plated. In the cases of pure silver and copper brazing, no separate annealing is necessary as the brazing temperature is high enough to accomplish this effect. Do not heat Kovar above 1100°C because grain size becomes enlarged beyond that temperature.

Attempts to braze Kovar with eutectic silver solder (or with solders having lower melting points) before annealing are likely to result in cracking, due to silver penetration of grain boundaries. Besides, the subsequent anneal will cause failure of the solder joint because of the higher temperature.

Kovar may be soft soldered with the same fluxes used for nickel (Nokorode salt solution is suitable). The surface of the Kovar must be clean. When brazing to brass or copper, it is advisable to apply the heat to these rather than to the Kovar, which should be previously annealed, as discussed above, and should preferably be copper plated at the site of the joint.

LEAD: Not to be used in tubes which are to be outgassed by heating, because of its high vapor pressure (see p. 89) and low melting point (328°C). Lead is useful as gasket material for high speed kinetic systems.

MAGNESIUM: Sometimes used as a getter material. Oxidizes rapidly in air; must be kept in a vacuum desiccator or under alcohol if to be used in tubes.

MERCURY: This liquid metal has wide usefulness in tube work, as in manometers (McLeod gauges), diffusion pumps, switches, discharge devices. When a mercury manometer or a mercury diffusion pump is to be used in connection

with a vacuum device, an adequate trap immersed in dry ice or, still better, liquid air or nitrogen, is to be installed between the tube being evacuated and the manometer or pump, to prevent mercury vapor from reaching the tube. When an ionization gauge is used, it should be on the same side of the trap as the tube being pumped.

Mercury readily combines with copper, brass and bronze to form amalgams, so that it should not be used in conjunction with these substances. Mercury vapor is poisonous when inhaled.

METALS: (See also ALLOYS) See Table "Properties of Some Metallic Elements", p. 66.

MICA (1): This is a useful material in tube fabrication because of its resistance to moderately high temperatures, to thermal and mechanical shock, and because of its good electrical properties. It can be accurately cut, drilled and slotted.

The preferred type of mica for tube and electrical work is Muscovite ($H_2KAl_3(SiO_4)_3$) which is nearly colorless in thin sheets.

| Hardness Mohs | Sp. Gr. | Thermal Conduct- ivity | Refrac. Index | Dielectric Strength 20°C volts/mil | Sp. Heat | Vol. resis. Ohms per cm cube |
|-------------------|-------------------------|------------------------------|--------------------------|--|----------|---|
| 2.8-3.2 | 2.6-3.2 | 1.8×10^{-3} cgs | 1.56-1.60 | 3000-6000 | | 0.2072×10^{13} - 1×10^{17} |
| Dielec. Const. | P. F. ($\frac{1}{Q}$) | Max. Coeff. Expansion | Water of Constitution | Mod. of Elasticity | | |
| 6.5-8.7 | 0.0001- 0.0004 | $36 \times 10^{-6}/^\circ C$ | 4.5 percent | 0.010 thickness lb/in 25×10^6 | | |

Affected only by hydrofluoric acid.

Heavily colored or striated mica should be avoided as the inclusions are chiefly iron compounds which are likely to be gassy. Pieces selected for tube work should be free of interlaminar defects, bubbles, inclusions, cracks or pinholes, and after cutting to shape should be handled carefully to avoid fraying the edges. For vacuum work the mica pieces, after machining, should be rinsed in two changes of acetone, soaked in methyl alcohol for 30 minutes, followed by thorough drying in an air oven at 200°C.

Mica should not be subjected to temperatures in excess of 625°C as the

(1) Some of this data is from the Handbook on Fabricated Natural Mica, Mica Fabricators Association, New York, 1949.

PHYSICAL PROPERTIES OF SOME METALLIC ELEMENTS

| | | | | | | | | | | | | | | | |
|---|-------|------|---------|---------|------|-------|------|---------|-------|------|------|---------|-------|--------|-------|
| Element Symbol | Al | Ag | Au | Ba | Be | Bi | C | Cd | Co | Cr | Cu | Fe | Hg | In | Ir |
| Elec. Resistivity Ohms cir. mil/ft. | | | | | | | | | | 15.6 | | | | 50.22 | 36.1 |
| | | | | | | | | | | | | | | | 0.16 |
| Temp. Coeff. of resistivity per °C | | | | | | | | | | | | | | 0.0047 | |
| | | | | | | | | | | | | | | | |
| Thermal Conductivity egs* approx. | | | | | | | | | | | | | | | |
| Approximate melting point °C | 0.5 | 1.0 | 0.7 | 850 | 1284 | 271 | 3500 | 321 | 1480 | 1615 | 1083 | 1535 | -39 | 155 | 2350 |
| Coeff. of linear thermal expansion 10 ⁶ per °C | 24 | 18.9 | 14.2 | | | | | | 12.3 | | 16.6 | 11.7 | | 41.7 | 5.71 |
| | | | | | | | | | | | | | | | |
| Tensile strength 1000 psi at 20 °C | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | |
| Specific Gravity | 2.7 | 10.5 | 19.3 | 3.78 | 1.85 | 9.75 | 2.25 | 8.64 | 8.9 | 6.92 | 8.92 | 7.86 | 13.55 | 7.28 | 22.42 |
| Magnetic | No | No | No | No | No | No | No | No | Yes | No | No | Yes | No | No | No |
| Annealing Temperature °C | 350 | 400 | 540-620 | | | | | | | | 550 | 650-815 | | | |
| Element Symbol | Mg | Mn | Mo | Ni | Os | Pb | Pd | Pt | Rh | Si | Sn | Ta | W | Zn | Zr |
| Elec. Resistivity Ohms cir. mil/ft. | 26.10 | 30 | 34.27 | | | | | | | | 39.0 | | | 35 | |
| | | | | | | | | | | | | | | | |
| Temp. coeff. of resistivity per °C | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | |
| Thermal Conductivity egs* approx. | | | | | | | | | | | | | | | |
| Approximate melting point °C | 0.376 | 650 | 1250 | 1455 | 2700 | 327 | 1555 | 1773 | 1960 | 1420 | 232 | 2900 | 3400 | 419 | 1900 |
| Coeff. of linear thermal expansion 10 ⁶ per °C | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | |
| Tensile Strength 1000 psi at 20 °C | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | |
| Specific Gravity | 1.74 | 7.42 | 10.2 | 8.9 | 22.5 | 11.34 | 12.0 | 21.45 | 12.44 | 2.42 | 7.3 | 16.6 | 19.3 | 7.14 | 6.4 |
| Magnetic | No | No | No | Yes | No | No | No | No | No | No | No | No | No | No | No |
| Annealing Temperature °C approx. | | | 1000 | 650-815 | | | | 700-800 | | | 20 | 1400 | 950 | | |

*Quantity of heat in calories which is transmitted per second through a plate 1 cm. thick across an area of 1 cm² when temperature difference is 1 °C.

cleavage planes separate and its mechanical properties are partially or largely destroyed.

Aluminum oxide (alundum), the material which is used to coat heaters, may be used to coat the surface of mica to enhance its ability to resist the effects of electrical leaks from evaporated metals in tubes. However, metal films evaporated on mica do not adhere very strongly, and may be removed by rubbing.

MOLYBDENUM: A nonmagnetic refractory metal, machinable in some forms; very useful in tube work. It is somewhat more ductile than tungsten and is suitable for heaters, grids, anodes, heat shields and supports. Molybdenum wire can be sealed to pyrex glass. It has a high melting point (2620 °C) and a low vapor pressure. It can be gold-nickel or platinum brazed (nickel plate the parts for best results in gold-nickel brazing), and may be readily spot welded to most metals used in tubes except to itself and to tungsten, to both of which it spot welds only with great difficulty. If the clean metal is given a thin layer of stopcock grease, some adhesion can be obtained. Molybdenum can be either vacuum or hydrogen fired for outgassing. Heaters made of molybdenum sag more than those of tungsten.

MONELS: This name is given to a series of nickel-copper alloys having various properties (see Table, "Monels", p. 68). The monels containing aluminum are subject to oxidation in the hydrogen furnace, but can be nickel plated like stainless steel to protect them for brazing.

NEOPRENE: A group of chloroprene (synthetic) rubber-like materials made by E. I. du Pont de Nemours and Co., Inc., having good resistance to chemicals and considerably more resistance to oils and greases than natural rubber. Various hydrocarbon and other solvents, including benzene, carbon tetrachloride, chloroform, amyl and ethyl acetates, toluene and xylene, have a marked softening and swelling effect upon them. Closely related materials, i. e. those having either similar chemical or mechanical properties, are listed by the Hycar Chemical Company*. The neoprenes are useful in vacuum work as tubing, gaskets, "O" rings, etc., on kinetic systems, or on the low-vacuum side of exhaust stations. Attempts to seal off vacuum devices with rubber or synthetic seals are usually not successful because of the large amounts of hydrogen and other gases given off. In using "O" rings, the metal members and their grooves should be so designed that a minimum of the rubber is exposed. In static seals metal-to-metal contact

*Chemical Names and Terms, Hycar Chemical Co., Akron, Ohio (1944).

PROPERTIES OF MONEL ALLOYS

(from International Nickel Company 1949)

| Type, designation and condition | MONEL wrought | "R" MONEL | "K"(1) MONEL | INCONEL | 326 (2) MONEL | "KR"(1) MONEL |
|--|--|---|---|---|--|--|
| Limiting composition Percent | Ni + Co 63-67 Cu Balance Fe 2.5 max Mn 2.0 max C 0.3 max Si 0.5 max Al 0.5 max S 0.02 max | Ni + Co 63-70 Cu Balance Fe 2.5 max Mn 2.0 max C 0.3 max Si 0.5 max S 0.025-0.060 | Ni + Co 63-67 Cu Balance Fe 2.0 max Mn 1.5 max C 1.0 max Si 1.0 max Al 2-4 S 1.0 max | Ni + Co 72 Cu 0.5 max Fe 10 max Mn 1.0 max C 0.15 max Si 0.5 max Cr 14-17 85-120 | Ni + Co 58-61 Cu Balance Fe 2.5 max Mn 2.0 max C 0.3 max Si 0.5 max S 0.02 max | Ni + Co 63-70 Cu Remainder Fe 2.0 max Mn 1.5 max C 0.35 max Si 1.0 max Al 2-4 S 0.010, max 150-175 |
| Tensile strength 1000 psi | 75 110 | 85 90 | 155 150 | | | |
| Condition | annealed cold-rolled hot-rolled cold-drawn hot-rolled, age hardened sand-cast, age hardened | | | | | |
| Melting point °C | 1300-1350 | 1300-1350 | 1315-1350 | 1390-1430 | | 1315-1350 |
| Coeff. of thermal expansion per °C × 10 ⁶ | 7.8 | 7.8 | 7.8 8.9 | 6.4 | | 7.8 8.9 |
| Electrical resistivity ohms cir. mil/ft. | 290 | 290 | 350 | 590 | | 350 |
| Magnetic | Yes | Yes | No | No | | No |
| Relative magnetic permeability μ/μ_0 (Air = 1) | 20° C 10° C | | | 1.007 | 1.025 1.1 | |
| Magnetic transformation °C | 43-60 | 43-60 | -80 | -40 | | -80 |
| Soft soldering | Yes | Yes | Difficult | Difficult | Yes | Difficult |
| Hard soldering | Yes | Yes | Poor (1) | Poor (1) | Yes | Poor (1) |
| Machinability | Good | Good | Tough | Tough | Good | Tough |

(1) The alloys containing aluminum or chromium are subject to oxidation in the hydrogen furnace and must be treated like stainless steel by nickel plating or otherwise, if brazing is to be done (see p. 15).

(2) 326 Monel is similar to wrought Monel but has slightly lower nickel content, making it substantially nonmagnetic at and above room temperatures. Magnetic properties are not appreciably influenced by hot or cold working. Fabricating and welding characteristics same as for Monel. Can be fired and bright annealed at lower temperatures and in H₂ atmosphere having a higher dew-point.

is often desirable.

NICHROME: See Table of Alloys, p. 52.

NICKEL: This metal is one of the most useful for vacuum tube construction. It is easily outgassed, has a fairly high melting point (1455°C), has good corrosion resistance, is very ductile when annealed, spot welds, hard and soft solders, machines and forms easily. It is a very satisfactory base for oxide coated cathodes. The nickel in use in this Laboratory is known as Electronic grade "A" Nickel*, and is obtained from the International Nickel Company through Whitehead Metal Products Company, Cambridge, Mass. The composition of grade "A" nickel is given below.

NICKEL ALLOYS: HIGH NICKEL ALLOYS: SUMMARY OF PROPERTIES (From International Nickel Company, Inc.)

ELECTRONIC GRADE "A" NICKEL*

| Limiting composition percent | |
|------------------------------|-----------|
| Ni (plus Co) | 99.00 min |
| Cu | 0.20 max |
| Fe | 0.30 max |
| Mn | 0.35 max |
| C | 0.20 max |
| Si | 0.20 max |
| S | 0.008 max |

"A" Nickel is commercially pure wrought nickel produced by melting and deoxidizing electrolytic nickel. Easily drawn and worked, welds and silver solders readily, has excellent resistance to corrosion. Sufficient strength is retained at bombarding temperatures to prevent deformation during outgassing. Modulus of elasticity and damping factor are high, minimizing vibrational and microphonic effects. Electrical resistivity is about 57 ohms circular mil/foot at 0°C. High magnetostriction coefficient of nickel makes it useful in devices employing this principle. Nickel is magnetic at room temperatures and becomes nonmagnetic near 350°C. Useful for support wires and rods, getter tabs, anode plates and cylinders, cathode shields, wire mesh for grids, lead-in wires.

*These are trade designations of the International Nickel Co., New York, N. Y.

| | |
|---------------|------------------------------|
| "220" NICKEL* | Limiting composition percent |
| Ni (plus Co) | 99.10 min |
| Cu | 0.20 max |
| Fe | 0.20 max |
| Mn | 0.20 max |
| C | 0.15 max |
| Mg | 0.01-0.10 |
| Si | 0.01-0.05 |
| S | 0.008 max |

"220" Nickel is used in hot cathodes where fairly easy activation is required, but without the liberation of excessive amounts of free barium. This falls in the proposed ASTM classification as "Normal" Cathode Material. As in all cathode grades, the Cu, Fe, and Mn contents are closely controlled and held to low levels. Si is the principle deoxidizing agent. Magnesium is controlled to 0.1 percent maximum as sublimation of this element is sometimes objectionable. Useful for all types of hot cathodes.

| | |
|---------------|------------------------------|
| "224" NICKEL* | Limiting composition percent |
| Ni (plus Co) | 99.00 min |
| Cu | 0.20 max |
| Fe | 0.20 max |
| Mn | 0.20 max |
| C | 0.15 max |
| Si | 0.12-0.20 |
| Mg | 0.01-0.10 |
| Ti | 0.02 max |
| S | 0.008 max |

An active cathode material by virtue of high silicon content. Mg is limited to avoid difficulties which might arise from the sublimation of this element. It is believed by some that Ti contributes to the cathode interface and for this reason is kept low. Used for hot cathodes.

| | |
|---------------|------------------------------|
| "225" NICKEL* | Limiting composition percent |
| Ni (plus Co) | 99.00 min |
| Cu | 0.20 max |
| Fe | 0.20 max |
| Mn | 0.20 max |
| C | 0.15 max |
| Si | 0.15-0.25 |
| S | 0.008 max |

*These are trade designations of the International Nickel Co., New York, N. Y.

Exhibits the highest activity of the three grades. Used for barium-strontium coated cathodes where activation under adverse conditions is required. The high deoxidizing agent content (Si) probably tends to liberate appreciable Ba. This is sometimes a limitation.

| | |
|-----------------|------------------------------|
| " 330 " NICKEL* | Limiting composition percent |
| | Ni (plus Co) 99.00 min |
| | Cu 0.15 max |
| | Fe 0.20 max |
| | Mn 0.30 max |
| | C 0.15 max |
| | Si 0.10 max |
| | S 0.008 max |

Designed primarily for use as anode material. Can also be used for cold cathodes and structural parts.

| | |
|---------------|------------------------------|
| " D " NICKEL* | Limiting composition percent |
| | Ni (plus Co) 93.70 min |
| | Cu 0.25 max |
| | Fe 0.75 max |
| | Mn 4.5-5.0 |
| | C 0.20 max |
| | Si 0.15 max |
| | S 0.015 max |

The strength and base hardness of "D" Nickel when fully annealed are slightly above those of "A" Nickel. Annealing temperature is also higher, hence greater hardness is retained after degassing. Oxidation somewhat greater than with "A" Nickel or "E" Nickel, so that the latter is more generally used for structural parts of tubes. The electron emission, even when contaminated with Ba, is lower than that of Ni, making it useful for grid wires.

| | |
|-------------|------------------------------|
| "E" NICKEL* | Limiting composition percent |
| | Ni (plus Co) 97.00 min |
| | Cu 0.25 max |
| | Fe 0.75 max |
| | Mn 1.75-2.25 |
| | C 0.20 max |
| | Si 0.15 max |
| | S 0.015 max |

*These are trade designations of the International Nickel Co., New York, N. Y.

The lower Mn content than "D" Nickel gives this material properties intermediate between "A" Nickel and "D" Nickel. Has good corrosion resistance at moderate temperatures. Useful for lead-in wires and supports.

DURANICKEL ("Z" NICKEL)*

Limiting composition percent

| | |
|--------------|-----------|
| Ni (plus Co) | 93.00 min |
| Cu | 0.25 max |
| Fe | 0.60 max |
| Mn | 0.50 max |
| C | 0.30 max |
| Si | 1.00 max |
| Al | 4.00-4.75 |
| S | 0.01 max |
| Ti | 0.25-1.0 |

An age-hardenable wrought nickel-aluminum alloy with mechanical properties between "K" Monel and Inconel "X". Useful for spring parts subjected to relatively high stress up to 340°C for prolonged times, and may be employed up to 400°C at low stress and short time. Slightly magnetic at room temperature in the soft condition, and magnetic after age hardening. Has good corrosion resistance. For best surface conditions ageing in dry hydrogen is recommended, but even in this gas a thin tenacious film of aluminum oxide is formed which must be removed prior to welding or soldering.

PERMANICKEL ("Z" NICKEL-TYPE B)*

Limiting composition percent

| | |
|--------------|-----------|
| Ni (plus Co) | 97.00 min |
| Cu | 0.25 max |
| Fe | 0.60 max |
| Mn | 0.50 max |
| C | 0.40 max |
| Si | 0.35 max |
| S | 0.01 max |
| Ti | 0.2-0.60 |
| Mg | 0.2-0.5 |

An age-hardenable high-nickel alloy developed for high strength, corrosion resistant applications. Requires different annealing and ageing treatments than Duranickel and has different physical properties. Magnetic at room

*These are trade designations of the International Nickel Co., New York, N. Y.

temperature in all conditions. Electrical conductivity higher than Duranickel but lower than "A" Nickel. Mechanical properties the same as for Duranickel. After ageing its surface must be cleaned before welding or soldering. Resistivity is about 260 ohms circular mil/foot. Used for grid wires, springs, diaphragms, thermostat contact arms.

LOW CARBON NICKEL ("L" NICKEL)*

Limiting composition percent

| | |
|--------------|-----------|
| Ni (plus Co) | 99.00 min |
| Cu | 0.25 max |
| Fe | 0.40 max |
| Mn | 0.35 max |
| C | 0.02 max |
| Si | 0.35 max |
| S | 0.01 max |

A low carbon material which is soft, ductile and suitable for spinning operations. Used under stress at temperatures higher than those suitable for "A" Nickel. Resistant to molten caustic soda, nitrates, and certain chemicals. Useful for getter tabs, heating element sheathing.

NILVAR: See INVAR.

"O" RINGS: Synthetic rubber (or neoprene) gaskets of round cross section, accurately made in a large variety of sizes, which are very useful as packing and seals in kinetic vacuum systems. The sizes range from 1/16" section, 1/8" I. D. and 1/4" O. D. to 1/4" section, 15 1/2" I. D. and 16" O. D. Information on the design of parts to receive these rings is contained in the catalog of the Goshen Rubber and Manufacturing Company, "O" Rings - Hydraulic Packings.

PALLADIUM: A metal having properties similar to platinum. A closed-end palladium tube heated to about 700 °C will pass hydrogen, i. e. if ordinary illuminating gas is fed into it, the other side being at reduced pressure, small quantities of pure hydrogen will diffuse through. This phenomenon provides a useful method for filling hydrogen discharge tubes at a controlled rate. The remaining inflammable gases comprising the illuminating gas are led out through a small nozzle and burned.

PHOSPHORUS: A nonmetallic element. Formerly used as a getter material in the form

*These are trade designations of the International Nickel Co., New York, N. Y.

of a coating on filament wire. Red phosphorus is suspended in alcohol with a little sodium silicate, and the wire dipped into it. Will absorb the ordinary atmospheric gases.

PHOSPHORUS PENTOXIDE: (P_2O_5): A rapid drying agent used in gas drying trains, desiccators, etc.

PHOSPHORS: Various chemical substances and mixtures which fluoresce in vacuum under the influence of a stream of electrons. Several characteristics as to color and persistence can be obtained. **WILLEMITE**, used in this Laboratory, is a zinc orthosilicate, having a greenish-white color and medium short persistence. It provides a useful general-purpose type of cathode-ray screen. Such a screen is prepared by mixing a small quantity of the dry powder, as a suspension, with distilled water and allowing it to settle undisturbed on the backing, which may be glass, nickel or other materials (and must be scrupulously chemically clean). When the settling is complete, the water is carefully partly decanted so as not to disturb the settled material, and the screen is allowed to dry in warm (not over 35°C) air, and protected from drafts and dust, for several hours, after which it may be put in an oven for 30 minutes at about 70°C. For additional information on phosphors, see Table, p. 75, also RCA Tube Handbook HB-3, Vol. 1-2, Cathode Ray Tube Section.

PLATINUM: May be used in the same way as palladium for admitting hydrogen to gas discharge tubes. Platinum foil is useful as a flux for spot welding tungsten or molybdenum. Pure platinum can be used as a brazing material at its melting point (1774°C) for molybdenum, tungsten, and for joining tungsten metallized thorium oxide cathodes to their supports. Platinum and platinum-rhodium alloys are useful as high temperature thermocouples, and platinum is employed in making high temperature resistance thermometers. The pure metal is very ductile, and has a notable resistance to corrosion at high temperatures. It can be sealed directly to certain glasses, and can be easily spot welded.

QUARTZ: (Silica) has the following physical properties:

| Coeff. lin. expan. per °C | Dielectric const. | Volume resistivity ohm/cm ³ | Refractive index |
|------------------------------|------------------------------|--|---------------------|
| 0.42×10^{-6} | 4.5 (fused silica 3.7) | 20°C- 5×10^{18} 600°C- 6×10^7 | 1.45843 |

FLUORESCENT COMPOUNDS

(From "Electronics" Buyers' Guide, June 1949)

| Compound | Activator | Fluorescent color | Luminous Efficiency candles/watt below | | Application and Remarks |
|--|------------|-------------------|---|------|--|
| | | | 1 Kv | 6 Kv | |
| Cadmium borate | Manganese | Red | 1 | 3 | For night viewing navigation indicators; short persistence, not sensitive to UV. |
| Cadmium sulfide CdS:Cu | Copper | Deep red | | | Not very active. CdS: Ag used with ZnS: Ag gives bright yellow, used for radar in daylight without filter and with tricolor red filter at night. |
| Cadmium tungstate CdWO ₄ | None | | 0.3 | 0.8 | Fluoresces without activator, but of little importance in CR tubes. Very sensitive to UV. Activated by Mn. |
| Calcium phosphate | Dysprosium | White | 0.4 | 1.5 | Long persistence; not sensitive to UV. |
| Calcium tungstate CaWO ₄ :Mn | Manganese | Blue | 0.3 | 1 | Stable photographic screen. Without an activator, CaWO ₄ is RMA-P5 used for observing and photographing transients; blue-violet, very short persistence (5 μsec). Also used in intensifying screens for X-ray. |
| Zinc Be silicate ZnBeSiO ₄ :Mn | Manganese | Yellow-green | 1.2 | 5 | Alternate to ZnSiO ₄ for general use; medium persistence. RMA-P3 used in early TV tubes. |
| Zinc Cd sulfide ZnCdS:Cu | Copper | Orange-yellow | 1.5 | 6 | Medium to long persistence; phosphorescent; component of RMA-P7 radar screen. Has orange afterglow. |
| Zinc Cd sulfide ZnCdS:Ag | Silver | Yellow | 2.5 | 10 | Short persistence; for daylight viewing and photography. Component of RMA-P6. |
| Zinc orthosilicate 2ZnO-SiO ₂ :Mn | Manganese | Green | 3.5 | 9 | Commonly called willemite. Used in RMA-P1 screen. Popular for scopes because of high light output, stability and ease of maintaining uniform characteristics in production. High efficiency at low voltage. Less apt to burn under electron bombardment than sulfides. Adding about 0.4 percent arsenic oxide plus 3 percent manganese activator increases persistence. Insensitive to UV. |
| Zinc phosphate | Manganese | Red | | | Sometimes used as special purpose persistent screen in tubes for radiography. |
| Zinc sulfide ZnS | None | Pale blue | | | Sulfides are the most important materials used for CR tube screens. Unactivated ZnS is relatively inefficient. Activity depends chiefly on impurities. |
| Zinc sulfide ZnS:Cu | Copper | Yellow-green | 2 | 12 | Addition of 0.001 percent Cu gives long persistence RMA-P2 screen for daylight viewing, for photographing traces and for holding transients. |
| Zinc sulfide ZnS:Mn | Manganese | Orange | 1.5 | 1.5 | For low voltage tubes. |
| Zinc sulfide ZnS:Ag | Silver | Actinic-blue | 2 | 0.4 | As little as 0.00001 percent Ag gives short persistence screen most suitable for photographic recording. Used with ZnCdS:Ag in RMA-P6 white, medium persistence screen. Used with ZnCdS:Cu in RMA-P7 to give medium persistence in blue and long persistence in yellow. (Known as AB screen in England). Used with nickel quencher in RMA-P11, 10 microsecond persistence screen. |
| Zinc silicate 2ZnO-SiO ₂ -H ₂ O | Manganese | Green | 3.5 | 9 | Medium persistence for general oscilloscopic work; commonly called calamine. |

Quartz is sometimes used as an envelope for vacuum devices where low coefficient of expansion, high working temperatures or sudden temperature changes, and/or passage of ultraviolet radiation (as windows which can be sealed on with cements), are required. Graded seals can be made to hard glasses.

RESISTANCE ALLOYS: See table of alloys, p. 49.

RUBBER: (See also NEOPRENE) Natural rubber has been almost entirely replaced by synthetic materials in vacuum work because of the superior chemical and oil resistance. Natural rubber tends to absorb somewhat more gas than neoprene, although it does have some mechanical properties which are superior to neoprene.

SILICA: (See QUARTZ)

SILICONE MATERIALS: See table, p. 77.

SILVER AND ALLOYS: In combination with other metals, and sometimes alone, silver is one of the most useful brazing materials. It is an excellent conductor of heat and electricity. (See section on Hydrogen Brazing for a discussion of silver brazing methods.) Silver may be readily evaporated in vacuum to form metallic reflecting or conducting films, and can also be deposited chemically by the Rochelle salt, Brashear, or silver paste process. Silver hard and soft solders easily, but is almost impossible to spot weld. (See FLUXES p. 59.)

COMMON SILVER ALLOYS

| Composition | Name of alloy | Sp. gr. | Coeff. therm. exp. per °C | Melt. pt. °C | Flow pt. °C |
|----------------|---------------|---------|---------------------------|--------------|-------------|
| Ag 99.9+ | Fine Silver | 10.5 | | 960 | |
| Ag 92.5 Cu 7.5 | Sterling | | | 804 | 910 |
| Ag 90 Cu 10 | U. S. Coin | 10.3 | | 890 | |
| Ag 80 Cu 20 | Jewelry | | 10×10^{-6} | 820 | |
| Ag 72 Cu 28 | Eutectic (BT) | | | 779 | |

(See Table: Soldering and Brazing Alloys, for other silver brazing materials.)

SOLDERING AND BRAZING ALLOYS AND FLUXES: (See Table, p. 79)

A general discussion of hydrogen atmosphere and other types of brazing will be found in the section HYDROGEN BRAZING, p. 19. SOFT SOLDERING, in which the lead-tin alloys are used, is useful for joining metal parts of

TABLE OF SILICONE MATERIALS
(From Literature Furnished by Dow Corning Corporation)

| Type and Name of Product | Use | Viscosity in Centistokes at | | | Freezing Point, °C. | Boiling Point °C. | Point mm Hg | Flash Pt. Min. °C. | Sp. Grav. 25°C. | Coeff. Exp. K × 1000/°C. 25-100°. | Ref. Index at 25°C. | | | | |
|--|---|---|--|---|---|--|---|--|---|---|--|-------------------------------------|--------------------------|--------------------------------|--------------------------|
| | | 0°C. | 25°C. | 100°C. | | | | | | | | | | | |
| FLUIDS | | | | | | | | | | | | | | | |
| Dow Corning 200 | Damping media; instrument and hydraulic fluids; antifoam and antislaking additives; water repellents; polishing agents; liquid dielectrics with dielectric constants at 1000 cps., 25°C ranging from 2.18 for the 0.65 centistoke fluids to 2.77 for the 30,000 centistoke grade, dielectric strength at 10 mils in the order of 250-300 V/mil, power factors ranging from 0.0001 at 10 ³ to 0.0006 at 10 ⁸ cycles. | 0.83 1.38 2.09 2.85 4.20 7.75 16.5 31 92 180 370 580 870 1700 55000 | 0.65 1.0 1.5 2.0 3.0 5.0 10 20 50 100 200 350 500 1,000 30,000 | Boils 0.54 0.70 0.86 1.15 1.78 3.3 6.0 16.8 33 67 98 147 270 9500 | -68 -86 -76 -84 -65 -65 -65 -60 -55 -55 -53 -50 -50 -50 -44 | 99.5 152 192 230 70-100 120-160 200+ 200+ 250+ | 760 760 760 760 0.5 0.5 0.5 0.5 0.5 | -1.11 43 71 79 101 135 162 271 273 | 0.761 0.818 0.852 0.871 0.896 0.918 0.940 0.950 0.955 0.968 0.971 0.972 0.972 0.973 0.973 | 1.598 1.451 1.312 1.247 1.170 1.095 1.035 1.025 1.000 0.969 0.968 0.966 0.965 0.963 0.963 | 1.375 1.382 1.387 1.390 1.394 1.397 1.400 1.402 1.4030 1.4031 1.4032 1.4033 1.4035 1.4035 | | | | |
| Dow Corning 550 Dow Corning 710 | High temperature baths; lubricants for instruments, clocks and other mechanical devices exposed to extreme temperatures, moisture, weathering. | 600 4000 | 150 480 | 24 33 | -50 -22 | - - | - - | 315 320 | 1.08 1.10 | 0.75 0.85 | 1.49-1.50 1.52 | | | | |
| DC 702 DC 703 | High Vacuum Diffusion Pump Oils. | 290 470 | 28 58 | 3.5 6.5 | - - | 400 450 | 10 ⁻² 10 ⁻² | 200 300 | 1.07 1.09 | 0.86 0.80 | 1.50 1.51 | | | | |
| DC Mold Release Fluid DC Mold Release Emulsions | Heat-stable lubricants used in molding rubber and plastic products. | | | | | | | | | | | | | | |
| COMPOUNDS and GREASES | | | | | | | | | | | | | | | |
| Dow Corning 4 Compound | Nonmelting, moisture-proof dielectric paste used to seal ignition systems and disconnectable junctions. Power factor, less than 0.001 at frequencies up to 10 megacycles; volume resistivity, more than 10 ¹² ohm centimeters at temperatures up to 200°C. | | | | | | | | | | | | | | |
| Dow Corning 7 Compound | Heat-stable, oxidation resistant release agent for porous or rubber molds, rubber bags or blankets used in low pressure laminating, heat-sealing equipment and patterns used in making Corning process molds. | | | | | | | | | | | | | | |
| Dow Corning 33 Grease | Heat-stable, oxidation resistant grease serviceable from -73 to 150°C. in ball bearings at a maximum speed factor in the range of 150,000 to 200,000. Used to lubricate gyro and small synchronous motor bearings, ball bearings exposed to severe outdoor weathering, cameras and optical instruments. | | | | | | | | | | | | | | |
| Dow Corning 41 Grease | Nonmelting and resistant to water, oxidation and chemical fumes; used to lubricate oven conveyor bearings, oven machinery, pumps handling hot liquids. Serviceable at temperatures from -25° to +210°C. and at a maximum speed factor in the range of 75,000. | | | | | | | | | | | | | | |
| Dow Corning 44 Grease | Nonvolatile, nongumming, nonsludging. Used as a permanent lubricant in sealed bearings at -40° to 175°C with a maximum speed factor in the range of 150,000 to 225,000. | | | | | | | | | | | | | | |
| Dow Corning Valve Seal A | Solidification point below -45°C. No true melting point. Serviceable in pump packings, flow meter bearings and valves operating at -54° to +260°C. and in contact with hot water, air and most gases, low pressure steam, aqueous solutions, dilute and some concentrated acids and alkalis, vegetable and mineral oils. | | | | | | | | | | | | | | |
| Dow Corning Stopcock Grease | Laboratory lubricant does not harden or melt at temperatures from -40° to over 200°C. Low vapor pressure; good resistance to many alkalis and acid reagents. | | | | | | | | | | | | | | |
| Dow Corning High Vacuum Grease | Heavier consistency lubricant for junctions in high vacuum systems operating at very low pressures. | | | | | | | | | | | | | | |
| RESINS and VARNISHES | | | | | | | | | | | | | | | |
| DC 801, DC 802, DC 803, DC 804 | Silicone resins for formulating heat and weather resistant coatings for ovens, heaters, stacks and other hot metal surfaces. Some silicone-aluminum paints withstand temperatures up to 500°C. | | | | | | | | | | | | | | |
| DC 935 | Flexible, fast drying silicone resin for coating glass cloth and sleeving and for bonding flexible mica-glass combinations. Dielectric strength of 10 mil, DC 935 coated glass cloth measured with 2 inch electrode is 1600 v/mil. | | | | | | | | | | | | | | |
| DC 993 | Very flexible silicone resin with excellent resistance to moisture and thermal aging for impregnating electrical equipment and for coating glass tapes and sleeving. Average dielectric strength (2 inch electrodes) 1500 v/mil; power factor, 100 cps at 25°C., 0.0077, at 150°C., 0.0050; craze life, over 1000 hours at 250°C. | | | | | | | | | | | | | | |
| DC 996 | Heat-stable, moisture resistant silicone varnish for impregnating and bonding electrical equipment. | | | | | | | | | | | | | | |
| DC 1097 | Modified silicone impregnating varnish with improved oil and solvent resistance. Average dielectric strength, 1500 v/mil; power factor, 100 cps at 25°C., 0.024; dielectric constant, 100 cps at 25°C., 3.2; heat endurance, over 1000 hours at 200°C. | | | | | | | | | | | | | | |
| DC 2103 | Thermosetting silicone resin for bonding inorganic fabrics in the production of rigid electrical laminates. Typical 1/8 inch silicone-glass laminates made with continuous filament cloth have a dielectric strength of more than 250 v/mil; power factor, 0.002 at 1 Mc/sec loss factor, 0.007 at 1 Mc/sec; wet insulation resistance more than 10 ¹² ohms; arc resistance, 300 seconds; heat distortion more than 250°C. | | | | | | | | | | | | | | |
| DC 2104 | Thermosetting silicone resin used in low pressure laminating and to bond heat formable thin laminates for use as slot liners and wedges, channel ground insulation, coil separators and forms. | | | | | | | | | | | | | | |
| DC PAN GLAZE | Silicone coating for pans used as a release agent in the commercial baking of bread and similar baking goods. One application remains effective for 100-200 bakings. | | | | | | | | | | | | | | |
| WATER REPELLENTS | | | | | | | | | | | | | | | |
| DC 1107 | Odorless, noncorrosive silicone product used as a coating to render paper water repellent and nonadhesive to rubber, asphalt, pressure sensitive tapes and to many sticky materials. | | | | | | | | | | | | | | |
| DeCetex 104 | Durable, water repellent treatment for nylon, Orlon, acetate rayon and other synthetic fabrics. | | | | | | | | | | | | | | |
| SILASTIC⁽¹⁾ RUBBERS | Williams Plasticity before curing | Hardness Shore A | Sp. Gravity | | Tens. Str. | | Elongation percent | | Brittle Point, °C. Minimum | Comp. Set Percent at 150°C. | Dielectric Str. ⁽²⁾ /v/mil | Dielectric Const. ⁽³⁾ at | | Power Factor ⁽³⁾ at | |
| | | | before curing | after curing | psi | psi | Avg. | Min. | | | | 10 ² c. p. s. | 10 ⁶ c. p. s. | 10 ² c. p. s. | 10 ⁶ c. p. s. |
| 6-125 | 15 to 35 | 40 to 55 | 1.5 | 1.6 | 460 | 375 | 160 | 125 | -79 | 35 to 55 | 500 | 6.2 | 6.4 | 0.003 | 0.002 |
| 6-150 | 110 to 160 | 45 to 55 | 1.9 | 1.9 | 300 | 250 | 210 | 175 | -79 | 30 to 50 | 500 | 5.6 | 4.4 | 0.005 | 0.025 |
| 6-161 | 120 to 180 | 55 to 65 | 2.1 | 2.3 | 400 | 350 | 160 | 130 | -79 | 30 to 50 | 200 | 9.2 | 8.1 | 0.015 | 0.040 |
| 6-167 | 130 to 180 | 60 to 70 | 2.0 | 2.0 | 520 | 400 | 100 | 80 | -79 | 50 to 70 | 500 | 9.0 | 8.4 | 0.003 | 0.002 |
| 6-181 | 130 to 200 | 75 to 85 | 1.5 | 1.5 | 680 | 500 | 65 | 50 | -79 | 40 to 60 | 800 | 3.6 | 3.2 | 0.020 | 0.006 |
| 7-170 | 110 to 180 | 65 to 75 | 1.4 | 1.4 | 610 | 500 | 90 | 60 | -57 | 15 to 25 | 700 | 3.3 | 3.0 | 0.015 | 0.005 |
| 250 | 150 to 220 | 40 to 55 | 1.2 | 1.2 | 650 | 550 | 300 | 250 | -79 | 40 to 60 | 800 | 3.2 | 3.0 | 0.002 | 0.009 |

(1) Physical properties given for Silastic 7-170 and Silastic 250 measured on 1/8 inch molded sections cured for 24 hours at 250°C; other properties measured on 1/8 inch sections cured for 4 hours at 250°C.

(2) ASTM D149-44, continuous voltage rise with 2 inch electrodes. Values measured on Silastic sections 1/32 inch thick.

(3) ASTM D150-47T, values measured on Silastic sections 1-1/2 inches in diameter and 1/8 inch in thickness, molded 5 minutes at 125°C. and cured for 4 hours at 250°C.

high-speed kinetic systems, but is not to be used where the device is to be sealed off from the pumps, and of course never where baking out is required. Soft solders containing antimony are not to be used, as the vapors are almost impossible to remove.

HARD SOLDERING in air, with a hand torch, generally requires the use of a soldering or brazing material containing silver, copper, cadmium and/or zinc, and sometimes other metals. Hard solders containing zinc and cadmium should be used in the construction of vacuum devices with caution, as both these metals have high vapor pressure. It is generally possible to remove the free zinc or cadmium by flushing the tube with a reducing or nonreacting gas while heating, so that the metallic vapors are carried off and do not deposit on any cool parts of the device.

FLUXES for hard and soft soldering should always be completely removed when the parts joined have cooled. This can usually best be done by boiling in plain or distilled water. Scrubbing may sometimes be necessary.

HYDROGEN FURNACE BRAZING provides the cleanest and most satisfactory method of making joints in vacuum devices, because solders can be used containing no volatile constituents and fluxes are not required. The alloys for use in hydrogen brazing are usually of a higher melting point than those used for torch brazing. (See special section on **HYDROGEN BRAZING**.)

STEEL: STAINLESS STEEL, etc. (See also Iron and Alloys, p. 63.)

Various types of steel are useful in tube construction (see Tables following). The low carbon steels are generally free machining, can be spot welded and brazed easily (copper or nickel plate for best results in hydrogen furnace brazing, as a clean iron surface is very susceptible to oxidation by the traces of water vapor present in tank hydrogen at the temperatures encountered), and can be outgassed by vacuum firing at 900 to 1000°C.

SOFT SOLDERS: TIN-LEAD ALLOYS

| Composition | | Description | M. P. °C | | Spec. Grav. | Tensile Str. tons/in ² | Hardness Brinell | Electrical Conductivity percent of copper |
|-------------|-----|--|----------|------|-------------|--------------------------------------|---------------------|--|
| Sn | Pb | | liq. | sol. | | | | |
| 100 | | Pure Tin | 232 | 232 | 7.31 | 0.94 | 4.6 | 13.9 |
| 63 | 37 | Eutectic | 183 | 183 | 8.42 | 3.35 | 13.9 | 11.9 |
| 60 | 40 | Best for general use. | 190 | 183 | 8.67 | 3.2 | 13.6 | 11.0 |
| 50 | 50 | | 212 | 183 | 8.91 | 2.75 | 12.0 | |
| 45 | 55 | Most ductile. | 225 | 183 | 9.13 | 2.75 | 11.7 | 10.6 |
| 40 | 60 | | 238 | 183 | 9.34 | 2.75 | 11.3 | 10.2 |
| 30 | 70 | Plumbers solder (best in tension and compression). | 257 | 183 | 9.48 | 2.66 | 8.7 | 9.5 |
| | 100 | Pure Lead | 327 | 327 | 11.34 | 0.89 | 4.1 | 7.91 |

SOLDERING AND BRAZING ALLOYS AND FLUXES

| MELT POINT °C | °F | FLOW POINT °C | °F | NAME OF SOLDER | COMPOSITION | FLUX | COMMENTS |
|---|------|------------------|------|-----------------------------------|---|-------------------|--|
| 60 | 140 | 65.5 | 150 | Woods Metal (see p. 90.) | Bi 50, Cd 12.5, Pb 25, Sn 12.5 | A | Weak, brittle |
| 145 | 293 | | | Low Temp. Solder | Pb 32, Sn 50, Cd 18 | | Used for pewter or fusible alloys |
| 181 | 358 | 181 | 358 | Eutectic Soft Solder | Pb 36, Sn 64 | A | |
| (See p. 78 for additional soft solders) | | | | | | | |
| 186 | 367 | 224 | 435 | Pipe and Cable Wiping | Pb 60, Sn 37.5, Sb 2.5 | A | |
| 188 | 370 | 225 | 437 | 50-50 Soft Solder | Pb 50, Sn 50 | A | Works easily, weak |
| 183 | 361 | 191 | 376 | "Fine" Solder | Pb 40, Sn 60 | A | Limited temperature range |
| 200 | 392 | 260 | 500 | 20-80 Soft Solder | Pb 80, Sn 20 | A, B | Weak |
| 204 | 399 | 240 | 464 | Sig. Cohn. Sn-Ag Solder | Sn Ag | A | |
| 231.9 | 449 | 231.9 | 449 | Tin | Sn 100 | A, B | High boiling point. Shrinks |
| 232 | 450 | 240 | 464 | Elec. work and Cu Tubing | Sn 95, Sb 5 | | |
| 288 | 550 | | | Alumaweld | Sn 40, Pb 39.9, Zn 20.1 | C, F | For castings and all metals except tantalum |
| 290 | 554 | 313 | 595 | Lead-Tin | Pb 95, Sn 5 | A | For joints up to 208°C |
| 304 | 579 | 304 | 579 | Lead-Silver | Pb 94-95, Ag 6-5 | A, B | |
| 304 | 579 | 380 | 715 | Lead-Silver | Pb 97.5, Ag 2.5 | A, B | |
| 327 | 621 | 327 | 621 | Lead | Pb 100 | B | |
| 337 | 639 | 399 | 750 | "TEC" Handy and Harman | Ag 5, Cd 95 | A | Note high Cadmium content |
| 400 | 752 | | | Intermediate Solder | Ag 20, Cu 3, Zn 2, Sn 75 | A | From John Strong "Procedures in Experimental Physics" 1942 |
| 550 | 1022 | 630 | 1166 | Gold-Indium | Au 80, In 20 | H ₂ | Hard, brittle |
| 600 | 1112 | 630 | 1166 | Low Melting Hard Solder | Ag 45, Cu 35, Sn 25 | H ₂ | From F. C. Hull, Westinghouse Mfg. Co. Very hard, brittle; low vapor pressure |
| 600 | 1112 | 640 | 1184 | Low Melting Hard Solder | Ag 46.5, Cu 32.5, Sn 21 | H ₂ | Very hard, brittle; low vapor pressure |
| 620 | 1148 | 636 | 1177 | "Easy Flo" Handy and Harman | Ag 50, Cu 15.5, Zn 16.5, Cd 18 | D, G | P. D. S. 7452 for ferrous and nonferrous use. Yellow, mechanically strong. |
| 630 | 1166 | 650 | 1202 | "RediFlo" American Platinum Co. | Ag 51, Cu 29, Zn 15, Sn 5 | D, H ₂ | White, mechanically strong |
| 641 | 1186 | 704 | 1300 | "Sil Fos" Handy and Harman | Ag 15, Cu 80, P 5 | None, G | For nonferrous work; self-fluxing, yellow, mechanically strong |
| 646 | 1195 | 688 | 1270 | "Easy Flo No. 3" Handy and Harman | Ag 50, Cu 15.5, Zn 15.5, Cd 16, Ni 3 | D, G | For ferrous and nonferrous use where fillets are required; yellow, strong |

SOLDERING AND BRAZING ALLOYS AND FLUXES (continued)

| MELT POINT °C | °F | FLOW POINT °C | °F | NAME OF SOLDER | COMPOSITION | FLUX | COMMENTS |
|------------------|------|------------------|------|---|-----------------------------------|----------------------|---|
| 670 | 1238 | 700 | 1292 | Low Melting Hard Solder | Ag 53, Cu 32, Sn 15 | H ₂ | From F. C. Hull, Westinghouse Mfg. Co.; very hard, brittle; low vapor pressure |
| 671 | 1240 | 779 | 1435 | "SS" Handy and Harman | Ag 40, Cu 30, Zn 28, Ni 2 | D, G | Pale yellow color |
| 677 | 1250 | 727 | 1340 | "ET" Handy and Harman | Ag 50, Cu 28, Zn 22 | D, G | Yellow-white color |
| 677 | 1250 | 743 | 1370 | "DE" Handy and Harman | Ag 45, Cu 30, Zn 25 | D, G | Yellow-white; ASTM Spec. B-73-29 4 |
| 682 | 1260 | 718 | 1325 | "RT" Handy and Harman (SC-1) | Ag 60, Cu 25, Zn 15 | D, G | Silver-white color |
| 693 | 1280 | 718 | 1325 | "Easy" Handy and Harman | Ag 65, Cu 20, Zn 15 | D, G | ASTM Spec. B-73-29 6; silver-white, for sterling silver |
| 693 | 1280 | 774 | 1425 | "ETX" Handy and Harman | Ag 50, Cu 34, Zn 16 | D, G | |
| 707 | 1304 | 750 | 1382 | "Phos-Copper" Westinghouse | Cu 93, P 7 | None, G | PDS 6713; for nonferrous work; self-fluxing; hard stock in CM 30. Does not come in small wire or thin sheet |
| 720 | 1328 | 740 | 1360 | Low Melting Hd. Solder | Ag 59, Cu 31, Sn 10 | H ₂ | From F. C. Hull, Westinghouse Mfg. Co. Very hard but can be swaged; Low vapor pressure. |
| 721 | 1330 | 779 | 1435 | "DT" Handy and Harman | Ag 40, Cu 30, Zn 28, Ni 2 | D, G | Pale yellow color |
| 724 | 1335 | 754 | 1390 | "Medium" Handy and Harman | Ag 70, Cu 20, Zn 10 | D, G | ASTM Spec. B-73-29 7. For sterling silver; silver-white |
| 732 | 1350 | 774 | 1425 | "Hard No. 1" Handy and Harman | Ag 75, Cu 20, Zn 5 | D, G | |
| 738 | 1360 | 793 | 1460 | "IT" Handy and Harman | Ag 80, Cu 16, Zn 4 | D, G | ASTM Spec. B-73-29 8 |
| 741 | 1365 | 788 | 1450 | "Hard" Handy and Harman | Ag 75, Cu 22, Zn 3 | D, G | Silver-white for Sterling |
| 743 | 1370 | 766 | 1410 | "NT" Handy and Harman | Ag 30, Cu 38, Zn 32 | D, G | Pale yellow color |
| 752 | 1385 | 785 | 1445 | "RE-MN" Handy and Harman | Ag 65, Cu 28, Mn 5, Ni 2 | D, G | |
| 777 | 1430 | 816 | 1500 | "AT" Handy and Harman | Ag 20, Cu 45, Zn 35, Cd 5 | D, G | Brass-yellow ASTM Spec. B-73-29 No. 2 |
| 777 | 1430 | 816 | 1500 | "ATT" Handy and Harman | Ag 20, Cu 45, Zn 30, Cd 5 | D, G | Brass-yellow ASTM Spec. B-73-29 No. 3 |
| 779 | 1435 | 779 | 1435 | "BT" Handy and Harman Silver-copper Eutectic | Ag 72, Cu 28 | D, G, H ₂ | PDS 6062-7; silver-copper eutectic white; excellent for copper. High electrical conductivity 77.1 percent of copper |
| 816 | 1500 | 857 | 1575 | "NE" Handy and Harman | Ag 25, Cu 52.5, Zn 22.5 | D, G | |
| 820 | 1508 | 950 | 1742 | No. 22 Tobin Brazing Bronze | Cu 59, Zn 38.7, P 0.3, Sn 1, Mn 1 | MARVEL | Good wear resistance and high tensile strength for cast iron and steel |
| 821 | 1510 | 871 | 1600 | "TL" Handy and Harman | Ag 10, Cu 52, Zn 38, Cd 0.5 max | D, G | Brass-yellow, ASTM Spec. B-73-29 |

SOLDERING AND BRAZING ALLOYS AND FLUXES (continued)

| MELT POINT °C | MELT POINT °F | FLOW POINT °C | FLOW POINT °F | NAME OF SOLDER | COMPOSITION | FLUX | COMMENTS |
|------------------|------------------|------------------|------------------|---|---|-------------------|--|
| 857 | 1575 | 871 | 1600 | "TE" Handy and Harman Brazing Compound | Ag 5, Cu 58, Zn 37 Cu 54, Zn 46 | D, G E | From John Strong, "Procedures in Experi- mental Physics" 1942 Lowest melting Gold-Copper |
| 890 | 1634 | 890 | 1634 | Gold-Copper Eutectic | Au 80, Cu 20 | E, H ₂ | Good wetting on tungsten |
| 950 | 1742 | 950 | 1742 | Gold-Nickel Eutectic | Au 82.5, Ni 17.5 | E, H ₂ | |
| 950 | 1742 | 980 | 1796 | Gold-Copper | Au 94, Cu 6 | E, H ₂ | |
| 960 | 1760 | 960 | 1760 | Silver | Ag 100 | E, H ₂ | |
| 1063 | 1945 | 1063 | 1945 | Gold | Au 100 | E, H ₂ | |
| 1083 | 1981 | 1083 | 1981 | Copper | Cu 100 | H ₂ | Wets tungsten |
| 1160 | 2120 | | | Platinum Solder | Ag 73, Pt 27 | H ₂ | Wets tungsten |
| 1205 | 2201 | | | Nickel Coinage (Prewar) | Cu 75, Ni 25 | H ₂ | Wets tungsten and molybdenum |
| 1450 | 2642 | 1450 | 2642 | Nickel | Ni plus Co 99-99.5; traces C, Mn, Si | H ₂ | |

FLUXES A - (a) Liquid: 40 ZnCl₂, 20 NH₄Cl, 40 H₂O
 (b) Paste: 90 Petrolatum, 10 NH₄Cl
 (c) Solution of Resin in Alcohol

B - Lloyd's No. 6

C - Lloyd's Alumaweld All-Metal Flux (thin paste)

D - Handy Flux, Lloyd's No. 7

E - (a) Thin paste (10 pts. powd. Borax, 1 pt. Boric Acid)
 (b) Borax applied dry

F - Lloyd's Stainless Steel Liquid flux

G - "Fluxine" PDS 8355-1

H₂ - Hydrogen

Marvel - 1450° - 1900°F

SUPPLIERS

Lloyd's - Lloyd S. Johnson Co., 2241 Indiana Avenue, Chicago, Illinois

"Handy" - Handy and Harman Co., 82 Fulton Street, New York, New York

Lead and Tin - Kester Solder Co., 4201 Wrightwood Avenue, Chicago, Illinois

Lead and Silver - Belmont Smelting and Refining Works, Inc., 330 Belmont Avenue, Brooklyn, New York

Sigmund Cohn, Gold Street, New York, New York

So-called "cold-rolled" steel is not any particular type, although generally the low carbon steels are received from the mill in a cold drawn or cold finished condition (see Table). For tube parts, types 1010, 1019, 1020 and 1022 are among the steels of choice, since the low sulphur content reduces gassing. In addition to the steels mentioned in the Table p. 84, there are DRILL ROD and GROUND FLAT STOCK (tool steels), both of which can be hardened and tempered, for making tools, dies, punches, etc. Broadly speaking, the low carbon (cold-rolled) steels cannot be hardened except by case-hardening or carburizing.

STAINLESS STEELS: (See Table, p. 83) The stainless steels are useful in tube work, not only as component parts, but also for making brazing jigs, where the protective layer of chromium oxide which is formed in the hydrogen furnace prevents the work from being brazed to the jig. In choosing a stainless steel for the making of brazing jigs, the "18-8" composition must be specified (types 302, 302B, 303 or 304; type 303 is preferred because of its good machinability), as a lower chromium content will not form an adequate oxide layer in the furnace. Although the formation of chromium oxide is a useful property in making brazing jigs, it is sometimes required to braze the stainless steels. The Westinghouse Laboratories have developed a nickel plating technique (described in detail elsewhere in this manual, see p. 15) in which the plate has excellent adherence and thus prevents oxide formation in the hydrogen furnace, allowing brazing with the usual silver or other solders.

STRONTIUM: An alkali earth metal of the barium type (See Barium, p. 54). The oxide is a normal constituent of the so-called oxide-coated cathode material.

TANTALUM: (See Table, p. 66.) A high melting point metal much used in tube making. It is very inert to chemical attack, ductile, malleable and machinable, much like mild steel in these respects. It spot welds easily. Tantalum parts for tubes must be thoroughly outgassed in a vacuum, because at elevated temperatures it absorbs gases, especially hydrogen, nitrogen and oxygen, to such a marked degree as to destroy the structure of the metal, making it extremely brittle. Tantalum parts must therefore not be passed through the hydrogen oven. For the same reason tantalum acts as an efficient getter (about 600°C; re-evolves H₂, N₂ and O₂ at about 800°C). In addition to its general use for tube electrodes and parts, tantalum can be used as a cathode material; it has the following emission properties:

| Temperature °C | Electron emission ma/cm ² |
|----------------|--------------------------------------|
| 1000 | 1 × 10 ⁻⁵ |
| 1250 | 4.7 × 10 ⁻³ |
| 1750 | 19.5 |

PROPERTIES OF SOME TYPES OF STAINLESS STEELS

| Type No. | 301 | | 302 302B | 303FM | 304 | 305 | 306 | 309 | 316 | 410 | 431 | 446 | 502 |
|--|-----------------|-----------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|-----------|--------------|--------------|
| Composition (approx.) percent | Cr | 16-18 | 17-19 | 17-19 | 18-20 | 17-19 | 19 | 22-24 | 16-18 | 11.5-13.5 | 15-17 | 23-27 | 5 |
| | Ni | 6-8 | 8-10 | 8-10 | 8-11 | 10-13 | 9 | 12-15 | 10-14 | 0.15 max | 1.25-2.50 | 0.35 max | 0.1 max |
| | C | 0.08-0.15 | 0.08-0.15 | 0.15 max | 0.08 max | 0.12 max | | 0.2 max | 0.1 max | | 0.2 max | | |
| | Mn | 2. | 2. | 2. | 2. | 2. | | 2. | 2. | 1. | 1. | 1.5 | |
| P | 0.04 | 0.04 | 0.04 | 0.04 | 0.04 | 0.04 | | 0.04 | 0.04 | 0.04 | 0.04 | 0.04 | |
| S | 0.03 | 0.03 | 0.03 | 0.03 | 0.03 | 0.03 | | 0.03 | 0.03 | 0.03 | 0.03 | 0.03 | |
| Si | 1. | 1. | 1. | 1. | 1. | 1. | | 1. | 1. | 1. | 1. | 1. | 0.5 |
| Mo | | | | | | | | | 2-3 | | | bal. | |
| Fe | bal. | bal. | bal. | bal. | bal. | bal. | | bal. | bal. | bal. | bal. | bal. | |
| Melting point °C | 1427 | | 1421 | 1421 | 1454 | | | 1454 | 1399 | 1510 | | 1510 | 1427 |
| Anneal temperature °C(1) | 1093 | | 1038 | 1038 | 1038 | | | 1038 | 1038 | 843 | 816 | 843 | 1093 |
| Hardening temperature °C(2) | | | | | | | | | | 954 | | | |
| Tensile strength 1000 psi | 90 | | 80-95 | 80-90 | 80-90 | 90 | 85 | 90-110 | 85-95 | 64-85 | 105 | 75-95 | 65 |
| Brinell hardness | 140-170 | | 130-175 | 130-150 | 125-150 | 130-162 | 125-150 | 150-180 | 150-200 | 140-180 | 250 | 160-195 | 150 |
| Specific gravity | 7.9 | | 7.91 | 7.91 | 7.91 | 7.91 | 7.91 | 7.88 | 8.02 | 7.7 | 7.86 | 7.57 | 7.75 |
| Coeff. of linear expansion × 10 ⁶ per °C | 0-100°C 18.2 | | 17.1 18.3 | 17.1 18.0 | 17.1 18.3 | 17.1 18.3 | 17.1 18.3 | 14.9 17.2 | 16.0 17.5 | 10.4 11.7 | 10.6 | 10.1 11.1 | 11.0 12.8 |
| Electrical resistivity microhm/cm | 73 | | 71-73 | 72 | 71-73 | 71-73 | 71-73 | 78 | 74 | 60-70 | 60 | 67 | 73 |
| Magnetic(3) | No | | | No | No | No | No | No | Slightly | Yes | Yes | Yes | No |
| Machinability | Tough | | | Good | Tough | Tough | Tough | Tough | Tough | Fair | Fair | Fair | Tough |
| Structure | Aust. | | Aust. | Aust. | Aust. | Aust. | Aust. | Aust. | Aust. | Mart. | Ferr. | Ferr. | Aust. |

NOTES: (1) The austenitic steels are quenched for annealing. The Fe-Cr types (410) are furnace cooled to 590°C and air cooled. Small parts can be air cooled from 760°C. Type 446 is air cooled or water quenched when over 3/16" thick.

(2) Cutlery type steels (410, 431) are oil quenched at 1010°C and drawn at 230°.

(3) The ferritic and martensitic structures are magnetic. The austenitic types are nonmagnetic with a permeability of 1.003 μ/μ₀ (air = 1) which cold work raises to 7.0. Cold worked type 316 goes up to 10. In cold worked condition the austenitic types are magnetic.

STEELS: AVERAGE DATA ON SOME REPRESENTATIVE TYPES

| Type No. | ARMCO INGOT | 1010 | 1019 | 1020 | 1022 | 1035 | 1040 | 1042 | 1045 | 1095 | 1112 | 1113 | 1115 | 1117 | 1120 | 1137 | 1141 | 4130 | 8630 | MILD STEEL |
|--|----------------|---------------|----------------|------------|-------|-------|--------------------|--------------------|--------------------|--------------------|--------------------|----------------|----------------|----------------|-------|-------|--------------------|--------------------|--------------------|---------------|
| Condition | | HR | HR CF | F HR CD | TGP | HR CD | G and P A CF CD | CF | CD HR CF F | OH HR Annealed | CF A B | CF B | CF CD | HR CF CD | HR | A TGP | F HR | OH HR Strip | OH HR Strip | |
| Application | | Soft steel | Screw stock | | | | | | Screw stock | Spring stock | Screw stock | Screw stock | Screw stock | Screw stock | | | | | | |
| Melting point °C | 1521 | | | | | | | | | | | | | | | | | | | |
| Specific Gravity | 7.87 | 7.85 | | | | | 7.84 | | | 7.83 | | | | | | | | | | |
| Composition, percent; other than Fe | C | 0.013 | 0.10 | 0.175 | 0.20 | 0.20 | 0.35 | 0.40 | 0.435 | 0.455 | 0.975 | 0.13 | 0.13 | 0.155 | 0.17 | 0.205 | 0.355 | 0.41 | 0.305 | 0.305 |
| | Mn | 0.018 | 0.40 | 0.85 | 0.40 | 0.85 | 0.75 | 0.75 | 0.75 | 0.75 | 0.4 | 1.15 | 1.15 | 0.85 | 1.15 | 0.75 | 1.5 | 1.5 | 0.5 | 0.8 |
| | Pmax | 0.005 | 0.04 | 0.04 | 0.04 | 0.04 | 0.04 | 0.04 | 0.04 | 0.04 | 0.04 | 0.045 | 0.045 | 0.045 | 0.045 | 0.045 | 0.045 | 0.045 | 0.045 | 0.04 |
| | Smax | 0.024 | 0.05 | 0.05 | 0.05 | 0.05 | 0.05 | 0.05 | 0.05 | 0.05 | 0.05 | 0.105 | 0.285 | 0.125 | 0.105 | 0.105 | 0.105 | 0.105 | 0.105 | 0.04 |
| | Si | trace | | | | | | | | | | | | | | | | | 0.3 | 0.3 |
| | Ni | | | | | | | | | | | | | | | | | | 0.55 | 0.55 |
| | Cr | | | | | | | | | | | | | | | | | 0.95 | 0.5 | |
| | Mo | | | | | | | | | | | | | | | | | 0.20 | 0.20 | |
| Tensile strength 1000 psi | HR | 46 | | 69 | 65 | | 85 | 91 | 93 | 99 | 142 | 67 | | 69 | 71 | | 92 | 97 | 90 | 90 |
| | CD | 49.5 | | 78 | 78 | | 103 ⁽¹⁾ | 113 ⁽¹⁾ | 116 ⁽¹⁾ | 120 ⁽¹⁾ | 200 ⁽²⁾ | 80 | 83 | 80 | 82 | | 112 ⁽¹⁾ | 126 ⁽¹⁾ | 146 ⁽³⁾ | 130 |
| Hardness Brinell | HR | 69 | | 143 | 143 | | 183 | 190 | 61 | 200 | 293 | 140 | | 117 | 135 | | 192 | 201 | 183 | 207 |
| | CD | | | 163 | 156 | | 201 ⁽¹⁾ | 235 ⁽¹⁾ | 63 | 235 | 388 ⁽²⁾ | 170 | 180 | 170 | 162 | | 255 ⁽¹⁾ | 269 ⁽¹⁾ | 293 ⁽³⁾ | 285 |
| Machinability | HR | | | 62 | 50 | | 65 | 63 | | 55 | 45 | | | | | | 70 | 67 | 65 | 58 |
| | CD | | 53 | 70 | 60 | 63 | 67 | 65 | 60 | 60 | | 100 | 120 140 | 80 | 94 | 78 | 75 | 74 | 70 | 42 |
| Ryerson color code | | | | Green | Green | | Blue | Yellow | Yellow | Yellow | Yellow and red | White | Orange | Black | Gold | | Red and Gold | Brown and Gold | | 50 |

LEGEND: A-Accuracy stock; B-Bessemer process; CD-Cold drawn; CF-Cold finished; F-Forged; G and P-Ground and polished; HR-Hot rolled; OH-Open hearth process; TGP-Turned, ground and polished.

NOTES: (1) Water quenched at 850°C, tempered at 540°C.
(2) Water quenched at 790°C, tempered at 430°C.
(3) Water quenched at 860°C, tempered at 540°C.

HIGH SPEED STEELS (Average Data)

| Type | | REGULAR | REGULAR | REGULAR | REGULAR | MOLY | MOLY | COBALT | COBALT |
|--|----|---|--|---|--|-------------------|-----------------------------|--|---------------------------------|
| Composition percent; other than Fe | C | 0.55-0.75 | 0.55-0.75 | 0.75-0.85 | 0.55-0.75 | 0.72-0.82 | 1.10-1.20 | 0.65-0.80 | 0.65-0.80 |
| | W | 17-19 | 19-20 | 17-19 | 13-15 | 1.3-2.0 | 5.5-6.0 | 17-19 | 18-21 |
| | Cr | 3.5-4.5 | 3.75-4.5 | 3.5-4.5 | 3.5-4.5 | 3.75-4.25 | 3.75-4.25 | 3.5-4.5 | 3.5-4.5 |
| | Va | 0.75-1.25 | 0.75-1.25 | 1.75-2.25 | 1.75-2.25 | 0.9-1.3 | 2.8-3.2 | 0.75-1.25 | 1.75-2.25 |
| | Mo | | | 0.4-0.9 | | 7.75-8.75 | 5.0-5.5 | | 0.5-1.0 |
| | Co | | | | | | 3.5-5.50 | | 10-13 |
| Specific Gravity | | 8.6 | | | | | | | |
| Comments | | Most used; so-called "18-4-1". Brittleness and cutting properties vary directly with C content. | Better cutting ability but more brittle. | Best cutting; excellent for finishing cuts. | Roughing tools; somewhat erratic in hardening. | Twist drills etc. | For resistance to abrasion. | For cutting hard, gritty or tough materials. | Maximum cobalt to be forgeable. |

Advantage may be taken of the refractory nature of the metal (mp 2900°C) to obtain part of the energy for cathode purposes by means of electron bombardment. Tantalum foil (about 0.001") can be used to advantage as a flux in spot welding tungsten and molybdenum.

TEFLON: (duPont) A trade name applied to tetrafluoroethylene resin. It is a plastic with good electrical, heat and chemical resistant properties. Teflon has a high coefficient of thermal expansion, with fair recovery. It can be used over the temperature range -320°F to 550°F, and is chemically resistant to many solvents, including a variety of halogenated hydrocarbons, ketones, esters and alcohols. Teflon can be boiled in aqua regia, hydrofluoric acid, sulphuric acid or nitric acid, with no change of weight or properties. It is equally resistant to the attack of strong alkalies. Chlorine, bromine or iodine do not react with Teflon, but fluorine and chlorine trifluoride will react under special conditions.

Teflon can be machined easily with standard wood or metal cutting tools if they are kept sharp. Chips must be removed from the machining areas rapidly. High cutting speeds with light cuts are recommended. For heavier cuts, soap solution or water-soluble oils in water are to be used. Teflon tape and sheet stock can be formed into parts with simple curvatures by drawing in male and female dies, to a maximum depth equivalent to one-half the original diameter of the flat sheet. If more extensive drawing is required the Teflon can be heated up to 450°F. Thin walled tubing can be flared with the same flaring tools used for copper tubing. Heavy tubing can be threaded with standard pipe threading equipment,

Typical properties of Teflon are given on p. 86.

THERMOCOUPLES: It is sometimes required to measure the temperature of heated bodies in tubes directly, and this can be done in some cases by means of thermocouples. The so-called thermocouple vacuum gauge utilizes this principle to measure the temperature of a filament which is directly affected by the heat conductivity of the residual gas in the tube. Various combinations of metals can be used for the couple; the choice is chiefly determined by the range of temperatures to be measured. The table below summarizes the ranges of some of the couples used:

| Type of Thermocouple | Range for Continuous Service °C | Range for Short Service °C |
|---|---------------------------------|----------------------------|
| Copper to Constantan (or Advance) | -190 to 350 | to 600 |
| Chromel to Constantan (or Advance) | 0 to 900 | to 1100 |
| Iron to Constantan (or Advance) | 0 to 900 | to 1100 |
| Chromel to Alumel (Hoskins) | 0 to 1100 | to 1350 |
| Platinum to platinum-13 percent rhodium | 0 to 1450 | to 1700 |
| Platinum to platinum-10 percent rhodium | 0 to 1450 | to 1700 |
| Tungsten to molybdenum* | 1000 to 2500 | to 2500 |

*Tungsten-molybdenum couples behave as follows: from 0 to 500°C the emf increases; above 500° it drops back to zero at 1000°, then reverses polarity: at 1400° the emf is -1.65 mv and at 1750° it is about -2.7 mv.

Properties of duPont Teflon

| Property | Value | Remarks |
|---|----------------------------------|---|
| Specific gravity | 2.1-2.3 | |
| Tensile strength at 77°F psi | 1500-2500 | Tensile str. in oriented film may be as high as 15,000 psi. |
| Elongation at 77°F percent | 100-200 | |
| Flexural strength at 77°F psi | 2000 | Specimens do not break. |
| Stiffness at 77°F psi (0.125") | 60,000 | |
| Impact strength, Izod-70, 77, 170°F ft-lb/in. | 2.0, 4.0, 6.0 | |
| Hardness "D" Durometer | 55-70 | |
| Compressive strength, psi, at 0.1 percent deformation | 1700 | |
| Deformation under load at 50°C under 1200 psi, percent in 85 hr. | 4-8 | |
| Heat-distortion temperature, low load °F | 270 | |
| Specific heat, Btu per hr. per ft ² | 0.25 | Method of mixtures. |
| Coeff. of linear expansion/°F (77-140°F) | 5.5×10^{-5} | |
| Thermal Conductivity Btu per hr. per ft ² for 1°F/in (for 0.18 section) | 1.7 | Cenco-Fitch apparatus. |
| Dielectric strength, short time v/mil (0.080") | 400-500 | 1000-2000 volts per mil in thicknesses 5 to 12 mils. |
| Power factor at 60, 10 ³ , 10 ⁶ , 10 ⁸ cycles | 0.0005 | |
| Dielectric const. at 60, 10 ³ , 10 ⁶ , 10 ⁸ cycles | 2.0-2.05 | |
| Water absorption percent | 0.00 | Not wet by water unless a surface-active agent is added. |
| Static coeff. of friction against polished steel | 0.09-0.12 | |
| Vapor pressure, mm Hg at 25°C | 10 ⁻⁶ | |
| Outdoor weathering (Florida) | No detectable change in 5 years. | |

Thermocouples for use in air must be protected against oxidation and mechanical injury. When thermocouples are subjected to excessive temperatures or oxidizing or corrosive atmospheres they cannot be depended upon to hold their calibration. For heavy duty it is desirable to use heavy gauge wire in making the couples. However, the thinner the wire the more responsive to small changes the couple will be. External connections from the couple may be made with copper or nickel wires provided that both conductors are the same, and that the joints are produced in the same manner, i. e. by soldering, brazing, welding or clamping. For highest accuracy a cold junction should be used in series with the heated junction, i. e. an

identical thermocouple immersed in melting ice. Thermocouple calibration tables will be found in the Handbook of Physics and Chemistry (Chemical Rubber Publishing Company).

THORIUM and THORIUM OXIDE: Thoriated tungsten, in which metallic thorium is combined in minor amounts with the tungsten, is used as an emitter of electrons, generally in the form of filaments. A detailed discussion of this type of cathode will be found in the section "Processing of Tubes on Exhaust", p. 40. Considerable success has been had in this Laboratory and elsewhere (S. T. Martin and co-workers) in the use of pressed and sintered mixtures of thorium oxide and tungsten or molybdenum powder, for use as a rugged, directly heated cathode. The mixture is formed into a hollow tube, current being applied from the ends through platinum brazed expansion type ferrules of molybdenum. This cathode gives good emission at fairly low temperatures (1650°C), does not have to be activated, and can be repeatedly exposed (cold) to the atmosphere without damage.

TIN: Although the mechanical strength of pure tin is low, it is sometimes used as a soft solder for tube parts made of copper or other metals because of its low melting point (232°C) and low vapor pressure:

| Temp. °C | Vapor pressure mm Hg |
|----------|----------------------|
| 823 | 10^{-5} |
| 922 | 10^{-4} |
| 1042 | 10^{-3} |

At temperatures below the melting point, the vapor pressure is not much above that of copper. Joints soldered with pure tin should be designed so that there are no stresses on the solder.

TRANSITE: A Johns-Manville Company proprietary product consisting of various proportions of asbestos fiber and portland cement. It can be obtained in various forms including sheets and tubes, and is useful in the tube laboratory for building ovens, heat-resistant table tops, etc. Can be drilled, sawed and machined.

TRICHLORETHYLENE: A noninflammable solvent useful as a degreasing agent. The vapors should not be inhaled. Parts to be cleaned should be handled in a tray or basket, and the hands should be protected with synthetic rubber gloves because the degreasing action is sufficiently strong to cause painful drying of the skin.

TUNGSTEN: This element has the highest melting point of any metal (3382°C). It

cannot be machined, but can be formed and cut by grinding with carborundum or other abrasives. Small wires of tungsten can be formed readily into various shapes provided there are no sharp bends. Tungsten is almost universally used as a filament or heater material, generally in the form of wire. It is also much used for support and press wires because of its rigidity and glass sealing properties; it seals readily to Pyrex. For further discussion of the fabrication of tungsten heaters see the section "Heater Design and Preparation", p. 33. Tungsten heaters and filaments should always be annealed and should be mounted strain-free, i. e. the ends should be welded to their supports or lead wires in the position they take naturally, if possible; otherwise only a slight amount of bending is allowable. Tungsten spot welds to itself and to molybdenum only with difficulty, but this type of weld can be made easily to nickel, tantalum and other metals. (See FLUXES, p. 59.) Tungsten and molybdenum can be brazed in the hydrogen furnace if first nickel plated, or if certain high temperature solders are used (gold 82.5 percent, nickel 17.5 percent; pure platinum; copper 75 percent, nickel 25 percent; and pure nickel). Tungsten oxidizes easily even at room temperature. Methods of cleaning are given in the section on chemical procedures, p. 2.

The electron emission of pure tungsten is as follows:

| Temp. °C | Emission ma/cm ² |
|----------|-----------------------------|
| 830 | 1.5×10^{-10} |
| 1630 | 2.3×10^{-1} |
| 2230 | 298 |

VAPOR PRESSURE: See Table, p. 89.

WATER: Water, in the form of WATER VAPOR is probably responsible for many virtual leaks in vacuum systems. Glass tubes should be baked during evacuation at 450° to 550°C, the temperature depending on what the glass will stand. Metal parts should be outgassed previous to assembly and, when feasible, in the finished tube by induction heating while being pumped. Water vapor is a product of the reaction occurring during the activation of barium-strontium "oxide" coated cathodes. For this reason the cold trap is to be in operation with liquid nitrogen during activation. Some workers prefer, in addition, to bake the tube again after activation, to insure removal of evolved water vapor from the walls and parts of the tube.

WILLEMITE: Zinc orthosilicate; used as a fluorescent coating material for cathode ray screens, electron gun targets, etc. See PHOSPHORS, pp. 74, 75.

VAPOR PRESSURES OF SOME OF THE ELEMENTS*
at various temperatures °C

| Element Symbol | Melting Point °C | Pressure mm of Hg | | | | | |
|-------------------|---------------------|-------------------|-----------|-----------|-----------|-----------|------|
| | | 10^{-5} | 10^{-4} | 10^{-3} | 10^{-2} | 10^{-1} | 1 |
| Ag | 961 | 767 | 848 | 936 | 1047 | 1184 | 1353 |
| Al | 660 | 724 | 808 | 889 | 996 | 1123 | 1279 |
| Au | 1063 | 1083 | 1190 | 1316 | 1465 | 1646 | 1867 |
| Ba | 717 | 418 | 476 | 546 | 629 | 730 | 858 |
| Be | 1284 | 942 | 1029 | 1130 | 1246 | 1395 | 1582 |
| Bi | 271 | 474 | 536 | 609 | 698 | 802 | 934 |
| C | --- | 2129 | 2288 | 2471 | 2681 | 2926 | 3214 |
| Cd | 321 | 148 | 180 | 220 | 264 | 321 | --- |
| Co | 1478 | 1249 | 1362 | 1494 | 1649 | 1833 | 2056 |
| Cr | 1900 | 907 | 992 | 1090 | 1205 | 1342 | 1504 |
| Cu | 1083 | 946 | 1035 | 1141 | 1273 | 1432 | 1628 |
| Fe | 1535 | 1094 | 1195 | 1310 | 1447 | 1602 | 1783 |
| Hg | -38.9 | -23.9 | -5.5 | 18.0 | 48.0 | 82.0 | 126 |
| In | 157 | 667 | 746 | 840 | 952 | 1088 | 1260 |
| Ir | 2454 | 1993 | 2154 | 2340 | 2556 | 2811 | 3118 |
| Mg | 651 | 287 | 331 | 383 | 443 | 515 | 605 |
| Mn | 1244 | 717 | 791 | 878 | 980 | 1103 | 1251 |
| Mo | 2622 | 1923 | 2095 | 2295 | 2533 | --- | --- |
| Ni | 1455 | 1157 | 1257 | 1371 | 1510 | 1679 | 1884 |
| Os | 2697 | 2101 | 2264 | 2451 | 2667 | 2920 | 3221 |
| Pb | 328 | 483 | 548 | 625 | 718 | 832 | 975 |
| Pd | 1555 | 1156 | 1271 | 1405 | 1566 | 1759 | 2000 |
| Pt | 1774 | 1606 | 1744 | 1904 | 2090 | 2313 | 2582 |
| Sb | 630 | 466 | 525 | 595 | 678 | 779 | 904 |
| Si | 1410 | 1024 | 1116 | 1223 | 1343 | 1485 | 1670 |
| Sn | 232 | 823 | 922 | 1042 | 1189 | 1373 | 1609 |
| Ta | 2996 | 2407 | 2599 | 2820 | --- | --- | --- |
| W | 3382 | 2554 | 2767 | 3016 | 3309 | --- | --- |
| Zn | 419 | 211 | 248 | 292 | 343 | 405 | --- |
| Zr | 2127 | 1527 | 1660 | 1816 | 2001 | 2212 | 2459 |

From "Scientific Foundations of Vacuum Technique"
by Saul Dushman; John Wiley, New York, 1949

*The values given in the above table are from a variety of sources, not all of which are in agreement. The table is to be used, therefore, only as a general guide. For a detailed discussion of vapor pressure data, see Dr. Dushman's book, pp. 752-754.

WOOD'S METAL: A fusible alloy having the composition: bismuth 50, lead 25, tin 12.5, cadmium 12.5; melting point 65.5°C, which can be used as a soft solder where temperatures must be kept low. There are several other alloys which have melting points in the neighborhood of 100°C. See also next page.

| Name | Composition | | | | | MP °C |
|------------------------|-------------|------|------|----|------|-------|
| | Bi | Pb | Sn | Cd | Hg | |
| Eutectic fusible alloy | 53 | 32 | 15 | - | - | 96 |
| Eutectic fusible alloy | 52 | 40 | - | 8 | - | 91.5 |
| Rose metal | 50 | 27.1 | 22.9 | - | - | 100 |
| Lipowitz alloy | 50 | 27 | 13 | 10 | - | 70-74 |
| Bismuth solder | 40 | 40 | 20 | - | - | 111 |
| Eutectic fusible alloy | 54 | - | 26 | 20 | - | 103 |
| Mercury alloy | 45 | 30 | 17 | - | 5-10 | 100 |

ZINC: A soft metal (mp 419°C) commonly a constituent of brasses and of many hard solders for use in torch brazing. It is easily vaporizable so that brass and those hard solders containing zinc should not be used in vacuum devices that are to be heat treated. See BRASS, p. 54.

ZIRCONIUM: A semi-refractory, ductile and malleable metal with the following physical properties: (Other refractory metals given for comparison.)

| | Zr | Ta | W | Mo |
|--|--------|---------|-------|-------|
| Melting point °C (approximate) | 1860 | 2850 | 3380 | 2620 |
| Specific gravity | 6.44 | 16.6 | 19.32 | 10.4 |
| Resistivity microhm/cm at 20°C | 41.0 | 15.5 | 5.3 | 5.7 |
| Temperature coeff. of resistivity, thousandths 0-100°C | 4.4 | 3.1 | 4.8 | 3.3 |
| Linear coeff. of expansion $\times 10^{-6}$ (0-100°C) | 6.3 | 6.5 | 0.39 | 0.346 |
| Specific heat cal/g °C | 0.068 | 0.036 | 4.45 | 4.9 |
| Ultimate tensile strength ₂ annealed, tons/in ² | 20-30 | 20 | 75 | 50 |
| hard, tons/in ² | 50-60 | 70-112 | 300 | 100 |
| Hardness, annealed (Brinell) | 50-60 | 70-120 | 290 | 250 |
| hard (Brinell) | 80-100 | 150-200 | 488 | 147 |

From Metals Reference Book, Smithells, 1949.

One of the uses of zirconium is as a high-temperature getter (above 400°C). See Espe, Knoll and Wilder, Electronics, October 1950, p. 80.

LOW MELTING POINT ("CERRO") ALLOYS

From Cerro de Pasco Copper Corporation, 1950

| Trade Name | Composition | MP °C | Wt. lb/in ³ | Brinell Hard | Characteristics* | Uses |
|--------------|-------------------------|------------------------------|------------------------|--------------|---|--|
| CERROBASE | Bi-Pb Eutectic | 125 | 0.371 | 10.2 | Shrinks slightly during solidification. After 1 hr. grows to max. of 0.0022" in. per 500 hrs. | Proof casting for forging dies; core dryer spotting slugs; filler for bending large tubing. |
| CERROBEND | Bi-Pb-Sn-Cd Eutectic | 70 | 0.339 | 9.2 | Expands approx. 0.001" during solidification, grows rapidly for 1 hr. and more slowly for 1000 hrs. to max. of 0.0057". | Filler in bending tubing. |
| CERROLOW-117 | Bi-Pb-Sn-Cd-In Eutectic | 48 | 0.32 | 12.0 | Expands slightly during solidification; shrinks to 0 in 30 min. and stabilizes in 2 hrs. at 0.0002". | Fusible cores in foundry work and in electric fire alarms etc. |
| CERROMATRIX | Bi-Pb-Sn-Sb Noneutectic | 102-227 Pour temp. 149 | 0.343 | 19.0 | Expands during solidification and grows rapidly for 15 hrs. with max. growth of 0.0061" during 1000 hrs. | Anchoring punches in stamping dies, etc. |
| CERROSAFE | Bi-Pb-Sn-Cd Noneutectic | 70-88 | 0.341 | 9.0 | Shrinks during solidification but grows to 0 in 1 hr., and in 96 hrs. reaches max. of 0.0025" oversize. | Proof-casting bullet molds and gun chambers, etc. Spotting and assy. fixtures. |
| CERROTRU | Bi-Sn Eutectic | 139 | 0.315 | 22.0 | Expands 0.0007" during solidification, shrinking only 0.0002" in 5 hrs. then stabilized. | Fusible mandrels in electroforming; fusible patterns in precision casting, and for anchoring rotors in Alnico magnets. |
| CERROSEAL-35 | In-Sn Noneutectic | 116-127 | 0.264 | -- | Adheres to glass, mica, glazed ceramics; solders metals. | Joins glass to metal for hermetic or vacuum systems, etc. |

*Growth figures are compared to cold mold dimensions. Measurements made on a 1/2" x 1/2" x 10" test bar, weighing approximately one pound.

**Inch per inch.

APPENDIX

Note A. VACUUM FIRING (from p. 33)

Metals such as nickel, Kovar, steels, copper, monels, etc. will begin to evaporate in vacuum at red heat, and the evaporated material will condense on the glass, gradually building up toward complete opacity. Thus the actual color of the work will be brighter than apparent through this obscuring film so that caution is to be exercised in applying r-f power beyond a given point, since sudden melting may occur before the operator is fully aware of what is happening. Consideration is also to be given to work which has several diameters, where attempts to heat the smaller diameter may result in overheating the larger in view of the closer coupling of the latter to the coil.

Note B. STRAIGHTENING TUNGSTEN WIRE (from pp. 31, 39)

The smaller sizes of tungsten wire (up to 0.025") should for many purposes be straightened. This is conveniently done by holding one end of a length in a vise and grasping the other end with pliers. Clip leads from a variac are placed close to each end of the wire and, while pulling with the pliers, the voltage is brought up slowly until the wire glows red. The tension is maintained until after the current is shut off. The wire may alternatively be straightened by suspending appropriate weights at the end of the length and applying current as mentioned. Keep feet away from under the weight. The oxidation resulting from this operation can be removed by the method given on p. 1. Tungsten wire for use as formed heaters or filaments (see Note C below) generally requires no straightening, but if conditions call for it, the process should be carried out in hydrogen, forming gas, or in vacuum, so as to prevent oxidation.

Note C. MINIMIZING SAG IN TUNGSTEN FILAMENTS (from pp. 31, 36)

A tungsten filament which is to be operated in such a position as to sag undesirably can be treated so that this effect is reduced. The filament is first formed in hydrogen on a stainless steel jig or mandrel as described on p. 36. It is then removed from the jig or mandrel and mounted on a press, or otherwise in a position similar to that which it will have in use, under a hydrogen bell jar. The hydrogen flow should be shielded or baffled so as not to impinge directly on the heated wire. The temperature should be raised to something over 1400°C (white heat) and observed through a welder's head mask, and the filament is then poked or pushed with a piece of heavy copper wire, formed into a small hook at the end, wherever sagging appears. The copper wire should be withdrawn frequently so as not to overheat and cause sticking. Considerably more current will be required to heat the wire in hydrogen than in vacuum because of the thermal conductivity of the gas.

Note D. GRADE A LAVA (from bulletin of the American Lava Company, see p. 56)

This material, a hydrous aluminum silicate, can be obtained in prisms up to a cubic

foot in volume. It should not, however, have wall sections greater than one-half inch to avoid cracking on firing. It is a good insulator after firing but has a high frequency loss factor considerably higher than the steatites. Grade A Lava can be easily machined like the steatite Lava, Grade 1137. The firing treatment is the same as for the latter, as outlined on p. 56. Grade A Lava expands rather than shrinks on heating, growing larger in size after a temperature of 650°C is reached. The expansion attains 1.9 percent at 980°C and 2 percent at 1040°C, over which there is a negligible change in dimension. A piece of Grade A Lava 0.980" will be 1.000" after firing.

Other properties of the fired material are given in the table, p. 56A.

Note E. TITANIUM (from "DuPont Titanium Metal", E.I. duPont de Nemours and Co., Inc. 1950.)

This metal may have some application in electronic tube construction especially in view of its lightness, resistance to corrosion, fairly high melting point, nonmagnetic properties, strength, workability and increasing availability. Some typical properties of titanium are as follows:

| | |
|---|----------------|
| Density at 20° C | 4.54 |
| Melting point °C | 1725 |
| Specific heat (cgs) | 0.142 |
| Coefficient of linear thermal expansion/°C × 10 ⁻⁶ | 8.5 |
| Electrical conductivity I. A. C. S. percent Cu | 3.1 |
| Electrical resistivity microhms/cc | 61 |
| Thermal conductivity Btu/ft ² /inch/ °F/hr | 105 |
| Magnetic | Para |
| Ultimate tensile strength psi annealed | 80,000 |
| one-half cold work | 110,000 |
| full hard cold work | 125,000 |
| Annealing temperature (in air) °C | 700 for 1 hour |
| Stress relief °C | 300 for 1 hour |

(thin sections, wire, foil, etc. should be annealed in vacuum)

Beginning at temperatures in the neighborhood of 700°C, titanium reacts appreciably with such atmospheric gases as O₂, N₂, water vapor, CO₂ and CO. As little as 1 percent O₂ or N₂ causes serious embrittlement. It may be handled at these temperatures in vacuum, or in argon or helium. It also reacts with other metals and refractories with the possible exception of graphite.

Descaling is accomplished in a sodium hydride bath for 20-30 minutes. This may be followed by a brightening dip for 10-15 seconds in a 2 percent HF – 10 percent HNO₃ mixture.

Titanium can be spot-, seam- and inert-arc-welded to itself.

The machinability of titanium is similar to that of austenitic stainless steels. Slower speeds and heavier feeds than those used for 18-8 stainless steel are recommended.

The metal exhibits corrosion resistance similar to that of stainless steel, and is especially resistant to salt water.

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