METHODOLOGY FOR PRODUCING SECONDARY COPPER

By Max J. Spendlove
METHODS FOR PRODUCING SECONDARY COPPER

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* * * * * * * * * * information circular 8002

UNITED STATES DEPARTMENT OF THE INTERIOR
Stewart L. Udall, Secretary

BUREAU OF MINES
Marling J. Ankeny, Director
This publication has been cataloged as follows:

**Spendlove, Max Jones.**

Methods for producing secondary copper. [Washington]

ii, 41 p. illus. 26 cm. (U.S. Bureau of Mines. Information circular 8002)


1. Copper. I. Title. II. Title: Secondary copper. (Series)

TN23.U71 no. 8002 622.06173

U.S. Dept. of the Int. Library.
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INTRODUCTION AND SUMMARY

The secondary copper industry is comprised of numerous enterprises which collectively employ many of the recovery and refining processes now used in primary plants as well as many other processes that are unique to the industry. Effective methods are used for identifying and segregating all types of scrap according to widely accepted standard classifications. Segregated scrap and waste materials usually require some preliminary processing to remove both valuable and deleterious associated constituents.

Smelting, melting, alloying, and pyrorefining are common methods used for producing secondary metal, but operating techniques usually differ from primary metal operations because of the difference in physical and chemical properties of the respective raw materials. Some copper scrap is converted to copper chemicals rather than to refined metals or alloys. Recovery efficiencies of all processes are reasonably high, and the quality of products meets rigid specifications.

The number of publications on the technology of secondary copper does not measure the importance of the science. Papers that are found in the literature deal more with statistics than technology. The search for published information is further complicated because titles of many pertinent papers do not reveal the included discussions on secondary copper.

Metals statistics were published periodically by the Geological Survey as Annual Reports on Mineral Resources from 1883 to 1923, and by the Bureau of Mines from 1924 to 1931. They are now published in the Bureau of Mines Minerals Yearbook. Technological discussions usually accompany statistical discussions, but publications on processing and refining are fewer than would be expected from a new and expanding industry. The purpose of this report is to present a complete collection of information on secondary copper in a single

1/ Work on manuscript completed in March 1960.
2/ Supervising Metallurgist, Bureau of Mines, College Park Metallurgy Res. Center, Md. P. M. Sullivan, supervisory chemist, also stationed with the Bureau at College Park, Md., contributed data on the production of copper sulfate from scrap.
publication. Preparation of this report was done as part of the Bureau of Mines secondary metals research program.

About 60 years ago the secondary metal industry comprised a group of independent junk collectors and dealers who gathered, sorted, and sold scrap metals and waste materials to relatively uncertain markets. As more scrap metal sources were developed and collection became better organized, some of the collectors and dealers sought to increase their margin of profit by remelting their scrap and producing commercial ingots. These ingots were generally poor in grade, by present standards. Having no precedent on which to base expansion and improvements, this embryonic industry added technical personnel and began educating itself in the art and technology of reclaiming values from scrap and waste materials. The quality of products and the degree of specialization in all segments of the industry soon improved considerably. Collecting, marketing, melting, refining, and alloying became individual operations, each contributing to the general advance of the industry as a whole. As the number of enterprises increased, a need grew for organized technical and statistical information and for some effective means to disseminate it to the industry. This service has been supplied for many years by organizations and trade journals including the Waste Trade Journal, National Association of Waste Materials Dealers, American Metal Market, Brass and Bronze Ingot Institute, and Bureau of Mines Minerals Yearbook. These and other sources enlightened consumers on the merits of secondary metals in a period when they were thought to be inferior to primary metals, regardless of the degree of refinement. Shortages arising from World War I led to a general acceptance of secondary metals and stimulated a rapid growth of the industry. A similar situation arose during World War II. Simultaneously, the primary metal producers developed a greater capacity for scrap and waste materials, and the industry developed into the pattern that exists today.

The secondary copper industry is the largest of the nonferrous secondary metal industries. It produces about 30 percent of the total copper consumed in the United States. The industry consists of several thousand dealers and collectors, several hundred foundries, about seventy ingot makers, about a dozen primary smelters using scrap and mineral concentrates, and several secondary smelters using scrap only. These plants are found mostly in the Northeastern States, the Pacific Coast States, and in the East North Central States; a few plants are located in the Southern and West Central States.

ACKNOWLEDGMENT

The author acknowledges the valuable assistance given by the industry. Without such help, this publication would not have been possible. He is particularly indebted to the following for their review of the original manuscript and suggestions for its improvement: Harry E. Schwartz, plant superintendent, H. Kramer & Co., El Segundo, Calif.; A. E. St. John, technical manager, Barth Smelting Corp., Newark, N.J.; Henry C. Deterding, metallurgist, Sonken-Galamba Corp., Kansas City, Kans., who provided information on flux refining; and Meyer H. Marx, Meyer H. Marx & Associates, St. Louis, Mo., who provided operating data for the blast furnace. Figures 3, 4, 5, 7, and 10, courtesy H. Kramer & Co.; 6, 8, and 11, Stroman Furnace & Engineering Co.; 9, U.S. Smelting Furnace Co.; and 12, 13, and 14, Ajax Electro-Chemic Corp.
DEFINITIONS

The term "secondary metal" came into wide use before it had acquired a singular meaning and still carries some incorrect connotations. It is important to recognize that "secondary" pertains only to origin and not to quality. Secondary metal is produced from scrap metal or metallurgical wastes as contrasted with primary metal, which is produced from ores. In other words, secondary metal is re-refined metal returned to the industry after having been used and may be equal or even superior in quality to virgin metal.

There are several other terms used in the secondary metals industry that need to be defined. Among these are "new scrap," "old scrap," and "purchased scrap." New scrap refers to materials produced in manufacturing plants, such as punchings, turnings, defective or surplus goods, and drosses, resulting directly from the manufacture of goods. Old scrap comprises obsolete, wornout, or damaged articles such as used radiators, pipe, wire, and ship propellers. The terms, "alloyed copper" and "unalloyed copper" are self-explanatory. "Home scrap" refers to scrap that is produced and consumed at the same plant. "Purchased scrap" is a general term used extensively in secondary-metals statistics. It excludes home scrap but includes all new and old scrap which has been purchased, or has entailed the cost of transport from one plant to another by the same company. It also includes wornout equipment and metal articles such as those reclaimed in shipyard repair work and line operations at railroad foundries.

Secondary copper loses its identity, except statistically, as soon as it is produced. It is not possible, for example, to determine whether a copper wire bar was derived from the scrap charged to a converter, or whether a brass ingot is made from remelted brass scrap or primary alloys.

Most secondary copper is contained in brass and bronze ingot and other alloys. For example, secondary copper recovered in 1958 totaled 797,388 short tons, of which 517,680 tons was recovered in brass and bronze metals. About 250,000 tons was contained in brass and bronze ingot only. About one-third of the 1958 secondary production was recovered from unalloyed copper scrap.

Figure 1 is a graphic comparison of annual copper refinery production from domestic ores and copper recovered from new and old scrap. Approximately equal amounts of each have been produced annually during the last decade. The amount recovered from scrap first exceeded production from domestic ores in 1932 and again in the years 1946-50. The greatest consumers of copper scrap are secondary copper smelters, primary copper producers, and brass mills. Other scrap consumers include foundries, chemical plants, and manufacturers.

Secondary Copper Cycle

The role played by scrap and secondary copper in the copper industry is illustrated by the chart in figure 2. Each of the consuming segments of the

industry contribute copper products to a secondary-by-products channel, as well as the copper-consumer channel, thereby causing some recycling of copper before it reaches the main channels of consumption.

Copper sold to manufacturers returns to the producers either as new scrap or old scrap. New scrap returns directly from the manufacturers or via collectors and scrap brokers. Old scrap returns from consumers of copper in used products. Purchased scrap may move from one location to another within the same company, or from one company to another.

It is evident that copper flows back to the producers by several cyclic paths. Some involve only producers, while others involve consumers, some manufacturers and producers, and some involve producers, manufacturers, and consumers. The cyclic period may range from a few days to several decades. Copper consumed in dissipative uses such as paint bases and chemicals is permanently consumed and never returned for processing.

**Scrap Classification**

Segregating scrap according to classification standards is one of the most important steps in the recovery of secondary metals. At one time, there were over 500 different commercial copper-base alloys made in the United States and the problem of sorting and grading mixed scrap with no uniform standards acquired major importance in the industry. Coordinated steps leading to present classification standards were not begun however until 1938. Classification standards now recommended by the National Association of Waste Materials Dealers, Inc., in their circular NF-58 include the following 54 types of copper-bearing scrap:

**Types of Copper-Bearing Scrap**

1. No. 1 Copper wire.
2. No. 2 Copper wire.
3. No. 1 Heavy copper.
4. Mixed heavy copper.
5. Light copper.
6. Composition or red brass.
9. High-grade, low lead bronze solids.
10. Bronze papermill wire cloth.
11. High-lead bronze solids and borings.
12. Machinery or hard red brass solids.
13. Unlined standard red car boxes (clean journals).
14. Lined standard red car boxes (lined journals).
15. Cocks and faucets.
17. Yellow brass scrap.
18. Yellow brass castings.
19. Old rolled brass.
20. New brass clippings.
21. Brass shell cases without primers.
22. Brass shell cases with primers.
23. Brass small arms and rifle shells, clean fired.
24. Brass small arms and rifle shells, clean muffled (popped).
25. Yellow brass primer.
27. Yellow brass rod turnings.
28. Yellow brass rod ends.
29. Yellow brass turnings.
30. Mixed unsweated auto radiators.
31. Admiralty brass condenser tubes.
32. Aluminum brass condenser tubes.
33. Muntz metal tubes.
34. Plated rolled brass.
35. Manganese bronze solids.
36. New cupro-nickel clippings and solids.
37. Old cupro-nickel solids.
38. Soldered cupro-nickel solids.
40. Miscellaneous nickel copper and nickel-copper-iron scrap.
41. New monel clippings and solids.
42. Monel rods and forgings.
43. Old monel sheet and solids.
44. Soldered monel sheet and solids.
45. Soldered monel wire, screen and cloth.
46. New monel wire, screen and cloth.
47. Monel castings.
48. Monel turnings and borings.
49. Mixed nickel silver clippings.
50. New nickel silver clippings and solids.
51. New segregated nickel silver clippings.
52. Old nickel silver.
53. Nickel silver castings.
54. Nickel silver turnings.
A few types of manufacturing waste materials are not considered as scrap, even though they contain copper. These materials are not included in the standard classification, and they are not usually handled by scrap dealers or brokers. An example is spent pickling solutions whose copper content is so low that the material must be discarded or processed near the point of origin. Some slags and drosses are in the same category.

SORTING SCRAP

Proper sorting of scrap requires quick and accurate methods of identification. Segregation practice varies with the amount and variety of materials involved. Small scrapyards usually segregate scrap to a few basic types, but larger yards find it practicable to segregate their scrap completely, according to all of the common grade specifications. Several methods have been developed for determining the approximate compositions of the thousands of items that pass through the scrapyards. The complexity of the tests ranges from simple recognition of known compositions to chemical analyses. Tradesmen usually acquire a great skill in applying the following simple tests to identify the common types of scrap.

Tests

Recognition

The simplest method of segregating scrap is by recognition of its source or previous use. For example, it is easy to classify copper wire, radiator fins, brass fittings, etc. by simple recognition. More nondescript items can often be identified by manufacturers trade mark or parts numbers.

Nicking, Filing, and Drilling

Alloys can often be identified by observing the physical characteristics of nick or file marks on the piece examined. Identification is based on hardness, color, and appearance of the freshly cut surface - a fairly accurate method of estimating the copper content of brass. The appearance of brass ranges from yellow to green or brown, aluminum bronze is a yellowish brown, and copper ranges from a smooth reddish brown when new, to green, when oxidized. A piece of brass chip is smooth, brittle, and relatively hard; a chip of copper is smooth, relatively soft, and heavy. Some sorters can identify as many as 20 varieties of scrap alloys in very rapid order by these characteristics, a skill which can be developed only by actual practice. It would be difficult, if not impossible, to teach this skill verbally.

Blowpipe

Blowpipe testing is less convenient than the file or nick tests, but it may be used when other tests are not applicable. The sample is heated with a blowpipe and identified by observing such properties as the color of the flame, melting speed, amount of fuming, and so forth. For example, tin bronze is fast melting, produces some fumes, and puddles like water; whereas brass
melts less rapidly, also fumes, and puddles like tin bronze. Pure copper is very slow melting, produces very little slag, and the molten pool bubbles.

Magnetic

Magnetic testing is used primarily to detect the presence of ferro-magnetic materials, such as iron, cobalt, or nickel. Only a few of the commercial copper-base alloys are magnetic, such as the cupro-nickel series, aluminum bronze, manganese bronze, and some copper-base alloys containing iron. Magnetic testing is also used for detecting copper-clad steel wire.

Spark

Spark tests are seldom used for identifying copper alloys but may be used to indicate the presence of ferrous components in the scrap. Tests are based on the fact that finely divided metal particles become heated and burn with characteristic incandescence when they are torn loose from the specimen by action of a grinding wheel. The test is used primarily for identifying various iron and nickel-base alloys.

Chemical Spot

There are numerous well-known spot tests for identifying alloys. They range from single drop tests, indicating only the rate of reaction, to relatively elaborate tests, determining the presence of specific alloying constituents as indicated by color, precipitate, gassing, or type of liquid mass formed. These tests are described in detail by Feigl.\(^4\) A less comprehensive but more convenient reference text was published by the Internation Nickel Co., Inc.\(^5\)

Chemical Analysis

Common analytical chemical methods are used extensively by scrap handlers and secondary metals producers. These methods are used primarily for process control, but can be used to identify scrap. Several standard texts are available for analysis of metals.\(^6\)

Spectrographic Analysis

Some of the major secondary metal producers have adopted spectrographic analysis. Its main advantage is that it gives a rapid quantitative analysis for most of the common metallic elements. Direct reading spectrographs are available equipped with multi-channel identification standards, which can


analyze a dozen or more elements. The entire analytical procedure takes about two minutes starting from the time the sample specimen is placed in the instrument. Although it is not practicable to use these instruments in the sorting area, they can be used to advantage to check identification by less accurate methods.

PREPARATION OF SCRAP

Mechanical

Equipment and refining procedures are essentially the same for primary and secondary copper. Both are often produced in the same operation. However, the methods used for pretreating scrap are quite different from those for preparing ore. Many types of scrap are prepared for smelting or melting by mechanical methods. Insulation and lead sheathing are removed from electrical conductors by special stripping machines or, occasionally, by hand stripping. Wire, thin plate, and wire-screen scraps are usually compressed into briquettes, bales, or bundles for convenient handling in subsequent processing operations. Losse materials are usually preferred for chemical recovery processes. Large solid items are reduced in size by pneumatic cutters, electric shearing machines, or manual sledgering. Brittle springy turnings, borings, and long chips are crushed in hammermills or ballmills to reduce bulk for easier handling in subsequent operations. Slags, drosses, skimmings, foundry ashes, spills, and sweepings are ground to liberate prills or other metallics from the gangue so that they can be recovered by gravity concentration or other physical means. Small-size materials such as drillings, clippings, and crushed turnings are often run over a magnetic separator to remove tramp iron.

Sampling

Scrap is usually received as a bulky heterogeneous collection of materials, which is very difficult to sample. The purpose of sampling is to select a small part of the scrap that will be representative of the whole lot. The probability of error in sampling increases with the degree of heterogeneity. It follows that sampling errors will be decreased by reducing the size of individual pieces to make the lot more homogeneous.

The criterion of sampling is expressed mathematically by the following equation:

\[ \frac{N(100)}{W} = \frac{n(100)}{w} = x. \]  

Where \( W \) is the total weight of the lot; \( N \) is the total weight of any one constituent in the lot; \( w \) is the total weight of the sample; and \( n \) is the weight of the corresponding constituent in the sample. \( X \) is the analysis expressed in percent.

In handling a large lot comprising several segregated parts, it is desirable to sample and analyze each part individually and make a weighted average according to the respective weights of the parts. A representative sample may
be obtained from fairly homogeneous lots by combining subsamples drawn from well-distributed points in the lot. Each subsample is representative only of that portion of the entire lot from which it is taken. The constituent \( n \), in equation (1) would, in this case, become the sum of the \( n \) constituents in all subsamples. An equation to represent this form of sampling is given by:

\[
\frac{N(100)}{W} = \frac{n_1 + n_2 + n_3 \ldots \ldots \ldots \ldots (100)}{w_1 + w_2 + w_3 \ldots \ldots \ldots} \tag{2}
\]

or

\[
\frac{N(100)}{W} = \frac{n(100)}{w} = X.
\]

This method of sampling is often condemned because the sampler may consciously or unconsciously influence the choice of samples. Thorough understanding of sampling theory and judicious sampling cannot be overemphasized when applied to bulky heterogeneous scrap. Sampling is satisfactory when several samples obtained by the adopted sampling procedure show agreement in analytical results.

Finely divided homogeneous materials present no problem, but special care must be exercised when sampling chips, borings, or materials that contain oil, water, or cutting compounds, which concentrate at the bottom of the pile or container. Loose materials are sampled by grab-sampling. A number of bales may be selected as representative samples, if the scrap is baled. Preliminary samples may be made into homogeneous alloys from which a final sample is taken.

Castings, mixed wire, tubes, rods, plate, foil, and utensils are particularly difficult to sample unless the scrap is segregated. Subsamples may be analyzed individually or melted together to form a composite sample.

High-iron copper scrap is often received as mixed lots, containing items of copper and brass mechanically associated with iron and steel such as electric motors and transformers. The most accurate method for preparing a sample from this material is to melt a large sample with sulfur to form a copper matte. A careful accounting must be made of all constituents entering the smelting operation so that analysis of the resulting matte can be applied to the original scrap.

Further discussions on sampling scrap are presented by Wright\(^7\) and Anderson.\(^8\)

**Sweating**

Many types of scrap must be given a preliminary furnace treatment before actual melting and refining operations begin. Oil and other organic impurities


and moisture are removed by heating in muffle furnaces or kilns. Scrap such as journal bearings, lead-sheathed cable, and radiators can be sweated to remove babbitt, lead, and solder as valuable byproducts, which would otherwise contaminate a melt. However, if a melt is made requiring a substantial amount of the sweated constituents, the scrap may be added directly to the melt without sweating.

The simplest furnace for sweating is the conventional sloping-hearth, gas-fired furnace. Batches of charge materials are put into the furnace at the highest point on the hearth. Low-melting constituents liquify and flow to the low end of the hearth and out of the furnace into a collecting pot. Sweated scrap is raked over the hearth until it is free of all low-melting metals and removed from the furnace so that new charge can be added. The process can be a continuous or batch operation. The sweated babbitt, lead, or solder may be made into white-metal alloys, used for lead and tin addition to copper-base alloys, or sold as produced to the refiner. Heavy lead-covered cable, railroad journal bearings, and similar bulky scraps are most frequently sweated in stationary sloping-hearth-type furnaces.

Occasionally sweating is done in a pot by dumping the scrap into a pot of alloy which absorbs the low-melting constituents. The sweated scrap is raked from the pot when sweating is completed.

Small size scrap can be sweated efficiently in a rotary kiln. Scrap is charged continuously at the elevated end of the kiln. The burner is placed at the discharge end so that combustion gases flow counter-current to the scrap. The tumbling action is effective in removing liquified constituents which flow out of the furnace and collect in a holding pot. Solid scrap discharges through a screen section fastened directly to the discharge end. Heavy scrap is not sweated by this method because of excessive wear to the furnace.

Some types of soldered items are more difficult to sweat completely because much of the solder remains in folds and seams even when melted. Several types of furnaces have been developed to solve this problem. One is a reverberatory furnace with a shaking grate of steel rails about the size of the furnace floor. The grate is pivoted at one end and the other end is pushed up and down in a fast reciprocating motion by a motor drive connected to the grate through a crank and arm linkage. The reciprocating action moves the scrap over the grate in a series of rapid short jerks, which also shake the liquid solders from the scrap. The molten solder falls to the floor of the furnace, where it flows to a low corner and into a collecting sump.

Some melters prefer a tunnel furnace in which the scrap is placed in trays or racks and carried through a heated tunnel by an endless conveyor. Some of the solder melts and falls from the scrap while inside the furnace tunnel. The remaining liquid solder is knocked free when the scrap spills from the conveyor onto a tilted screen. Solder and the metal flowing from the tunnel floor collect in a sump. A typical tunnel furnace is shown in figure 3.
FIGURE 3. - Tunnel-Type Sweating Furnace.

SMELTING LOW-GRADE COPPER SCRAP AND WASTES

Blast Furnace, Cupola

The function of a primary copper blast furnace is reduction of copper compounds and formation of copper matte and slag. Although it has been virtually superseded by the reverberatory furnace in primary smelters, the blast furnace is still used extensively in secondary smelters for smelting low-grade copper and brass scraps, refinery slags, drosses, and skimmings. When used primarily for melting scrap, with little or no reduction of oxidized materials, it is called a cupola. Operations and equipment are similar to those used for smelting copper ores and concentrates. Differences arise mainly because most of the metals in scrap and wastes are already in metallic form.
The conventional secondary copper blast furnace is a top-charged, bot-tapped shaft furnace heated by coke burning in a blast of air introduced through tuyeres placed symmetrically around the bottom of the shaft. The upper section of the shaft is cylindrical, but the lower section (the bosh) is an inverted truncated cone tapering to two-thirds the diameter of the upper shaft. A crucible is located directly below the bosh to collect molten metal and slag produced in the smelting zone above. Refractories for the in-wall, or well, are usually fireclay brick from top to bottom. A layer of chilled slag takes the place of refractories in the water-jacketed steel bosh. The crucible is lined with magnesite or chrome brick.

The charge is normally made up from copper-bearing scrap, a slag, sinter, limestone flux, millscale, and coke. The scrap may contain iron-brass and copper, fine insulated wire, motor armatures, foundry sweepings, slags, drosses, and many other similar low-grade materials. With present market conditions, the minimum profitable copper content for the charge is about 30 percent. Fine materials are usually sintered to produce a strong sinter cake or densified by other means, such as briquetting. Coke is used as a fuel and reducing agent. Limestone and millscale are added as fluxes to produce an iron silicate slag. Sulfur in the coke or other charge materials reacts with copper to form copper matte. This is avoided as much as possible in the secondary blast furnace by using low-sulfur coke.

Charge materials are heated as they descend through rising hot gases, becoming semiplastic and then liquid when they reach the region in the furnace called the smelting zone. Metallic constituents, such as brass and copper, may actually melt above the normal smelting zone. Limestone and iron oxide fuse in the smelting zone and form a molten slag which mixes with the metals in the turbulence of the gases. Molten materials drip through the coke bed and into the crucible below. The coke remains virtually unchanged until it reaches the tuyere zone, where it burns to carbon dioxide. Part of the carbon dioxide is reduced to carbon monoxide by the white-hot coke near the tuyeres.

The gases rising through the shaft are composed of CO, CO₂, and N₂. The relative amount of CO₂ increases at higher elevation in the shaft; the coke and air ratio is adjusted to provide a reducing atmosphere. Oxides of the base metals either dissolve in the slag or fume off; many are reduced and dissolved in the copper. The black-copper product of the blast furnace may contain zinc, lead, tin, bismuth, antimony, iron, silver, nickel, or other metals contained in the scrap. Many of these are fumed off and recovered as baghouse dust.

Both slag and metal are usually tapped through a launder into a rever-beration where they are held in a quiescent state to allow more complete separation of metal and slag. Some operators tap metal intermittently and slag continuously.

Stewart² describes some of the difficulties attending the operation of blast furnaces:

secondary blast furnaces of conventional design. These difficulties are eliminated by inverting the bosh section of the furnace so that it flares out at its bottom rather than the top. The inverted-bosh design has been adopted by a number of secondary smelters. Following are performance data for a typical secondary blast furnace with an inverted bosh:

A blast furnace with a water jacket 10 feet high, measuring 50 inches diameter at the tuyere level and tapering to 40 inches at the top, has a nominal capacity of about 60 to 70 tons of pay material per day. Charge materials contain about 10 to 11 percent coke. The air requirement is approximately 125 to 150 cubic feet per minute per square foot of area at the tuyeres, delivered at a pressure of 25 to 30 ounces per square inch. Fluxing materials include limestone, millscale, and metallic iron. The resulting slag has the following approximate composition:

\[
\begin{align*}
\text{Pct.} & \\
\text{FeO} & \cdots \cdots \cdots 29 \\
\text{CaO} & \cdots \cdots \cdots 19 \\
\text{SiO}_2 & \cdots \cdots \cdots 39 \\
\text{Zn} & \cdots \cdots \cdots 10 \\
\text{Cu} & \cdots \cdots \cdots 0.8 \\
\text{Sn} & \cdots \cdots \cdots 0.7 \\
\end{align*}
\]

Collected dusts contain 58 to 61 percent zinc, 2 to 8 percent lead, 15 to 5 percent tin, 0.5 percent copper, 0.1 percent antimony, 0.1 to .5 percent chlorine, and some unburned carbon. The metal product will vary widely depending on the materials charged. The approximate range of composition is 75 to 88 percent copper, 1.5 percent tin, 1.5 percent lead, 0.1 to .7 percent antimony, 0.5 to 1.5 percent iron, 4 to 10 percent zinc, and 0.5 to 1.25 percent sulfur. The slag, which may contain up to 1.5 percent copper, is usually discarded.

Figure 4 illustrates an efficient secondary copper blast furnace. Figure 5 shows the dust collecting system for the blast furnace and other plant equipment.

**Converter**

Converters are pear-shaped or cylindrical vessels used for converting copper matte, an impure mixture of iron and copper sulfides, into blister copper. They are made with steel shells lined with calcined magnesite, either in monolithic or brick form. Tuyeres are provided for blowing air into the molten charge when the converter is tilted to the "blow" position. Conversion of copper matte is performed in two stages of blowing. The FeS component of the matte is oxidized to FeO and SO\(_2\) in the first stage. The SO\(_2\) gas is expelled with other furnace gases, and the FeO combines with a siliceous flux to form an iron-silicate slag, which is poured off. It is possible to generate sufficient heat in the first stage of blowing to damage the refractory lining. Some smelters add heavy copper scrap to the converter as a convenient way to keep the melt from exceeding the proper temperature. Normally the scrap
FIGURE 4. - Blast Furnace for Smelting Copper-Base Scrap, Slag, and Residues.

should contain a minimum of oxidizable constituents in order to avoid an additional source of heat. The iron in high-iron scrap would be such a source. If high-iron scrap is used it can be compensated for by adding other materials such as refinery slags, reverts from furnaces and converters, and some clean copper scrap. Material and heat balances for a typical operation are virtually impossible because of the wide range of materials handled.

The second stage of blowing is accompanied by several reactions in which the net result is conversion of Cu$_2$S to Cu and SO$_2$. The heat generated is sufficient to maintain the converter temperature. Converter slag is a basic iron silicate which may contain up to 4 percent copper. It is returned to a reverberatory matte furnace where entrained copper collects in the matte.

The life of the converter lining is extended by the common practice of forming an infusible magnetite coating over the surface of the magnesite lining. This is done when necessary by blowing matte without adding silica flux. The product of the copper converter is blister copper, which contains 90 to
99 percent copper and some gold, silver, and other impurities. Blister copper is refined to high-grade copper in subsequent pyrometallurgical and electrolytic operations.

MELTING AND ALLOYING INTERMEDIATE-GRADE COPPER SCRAP

General

About two-thirds of the secondary copper production in the United States is used in ingot plants and foundries to make brass and bronze alloys by simple melting and refining methods. The amount of refining is usually small if the scrap is well sorted so that impurities or excess alloy constituents can be diluted to composition specifications with high-grade scrap or virgin metals. These conditions are not easily maintained, however, because of certain impurities such as aluminum and silicon, which have exceedingly low permissible limits in the product. In the red brass series, for example, maximum acceptable limits for aluminum and silicon are 0.005 and 0.003 percent, respectively. Both aluminum and silicon are difficult to remove by refining. Dilution to specifications is not practicable because of the relatively large proportions of high-grade scrap or virgin metals needed to dilute to these low limits. Impurities such as iron, sulfur, cadmium, bismuth, zinc, phosphorus, and manganese are not so difficult to remove by common refining techniques.
Melting, refining, and alloying procedures are essentially the same regardless of the type of furnace used. Operations are usually controlled by personnel who have acquired considerable skill through years of experience. Although indicating and controlling pyrometers are used extensively, a furnace operator may control the furnace temperature primarily by observing the color and consistency of the slag and metal when stirred with a rod. The degree of refining is indicated by the set of samples taken during various phases of the operation. This technique is common in copper refineries where it is used to indicate the various stages of oxygen and sulfur removal. Progress is also determined quite accurately by other physical changes, such as, the appearance of fractured surfaces (hardness, color, grain size, and texture). Experienced operators can estimate alloy compositions very closely and detect the presence of a number of impurities by these methods.

**Fluxes**

Fluxing is an essential part of both melting and refining. The basic functions of fluxes are essentially the same, used in reverberatory, rotary, or crucible furnaces.

Two general types of fluxes used for melting and refining scrap copper are: (1) Nonmetallic fluxes, and (2) fluxing alloys.

Nonmetallic fluxes may be solid, liquid, gaseous, or mixtures of these. Some are used for the sole purpose of protecting the surface of a melt from the prevailing atmosphere. Others refine by mechanical or chemical actions.

Fluxing alloys comprise one or more active agents, such as, phosphorus or lithium in a base like copper. This type is used either to refine the melt by deoxidation, add a definite amount of an alloy constituent, or both.

Sodium chloride, charcoal, borax, anhydrous rasorite, slacklime, glass, nitrogen, oxygen, and various combinations of these are common among the nonmetallic fluxes. Sodium chloride is used as a flux cover and as a fluid medium for separating metallic and nonmetallic materials in heterogeneous melts. Charcoal covers are used to add heat to the surface and provide a reducing atmosphere. Various combinations of charcoal-sodium chloride fluxes are used occasionally.

Borax, slacklime, and glass are added in various combinations to protect the metal surface and reduce volatilization of some of the constituents in the melt. Sodium borate flux is used extensively in the secondary copper industry, usually in the form of a concentrate known as anhydrous rasorite which contains about 90 percent sodium borate (Na₂B₄O₇). This is equivalent to about 62 percent boric oxide (B₂O₃) - the active ingredient in the borax fluxes. This flux has a great affinity for metal oxides and siliceous materials. It also has many of the most desirable characteristics of a good flux, which are: Quietness when melting, fairly low melting point (735° - 1367° F.), nonfuming, low moisture absorption, easily adjusted fluidity, and ease of handling. It is used primarily to scavange oxides and to provide a protective cover for molten scrap brass and bronze. The slag is rather fluid and not
excessively corrosive to refractories. The flux can be thickened for clean
easy skimming by adding dry sand or glass. Mixtures of lime and borax are
used frequently for fluxing alloys of the monel and nickel-silver types. Such
covers are normally very fluid but they can also be thickened with dry sand or
glass.

Some secondary smelters have used Gerstley borate fluxes satisfactorily.
The Gerstley borate ore contains the minerals colemanite and ulexite, which
are associated with some gangue. The major components of borate flux include
approximately 33 percent B₂O₃, 16 percent CaO, 5 percent Na₂O, 28 percent
water and organics, 10 percent SiO₂, and 4 percent MgO. It has about the same
fluxing properties as anhydrous rasorite.

Caustic soda has been used as a fluxing agent for removal of iron and
aluminum from some copper-base alloys. Iron is usually removed by blowing
with air under a cover of silicate slag. Blowing is not satisfactory, how-
ever, if the alloy contains more than 8 percent zinc, because loss of zinc is
high. Deterging claims satisfactory refining with a liquid flux of caustic
soda (2 percent of the charge) agitated gently with air. The procedure is
particularly effective for removal of iron and aluminum from high-zinc copper
alloys.

Gaseous fluxes may be inert or chemically active to the melt. Inert
gases are used to remove some of the high-melting oxides and some gaseous and
liquid impurities from the melt by simple entrainment during agitation. Dry
nitrogen is used extensively as an effective inert gaseous fluxing agent.
Water vapor and gases liberated during green poling act as inert fluxing
agents.

Gaseous fluxes are usually introduced into the melt through a pipe
inserted below the surface. Small bubbles of inert gas adhere to small
globules of molten slag, small solid oxide particles, and other impurities
providing buoyancy, which raises them to the surface where they may be
removed with the slag or flux cover. Some gaseous impurities may also be
removed. The free gaseous impurities such as hydrogen and oxygen tend to
interdiffuse with the fluxing gas, and the resulting gas mixture rises to the
surface.

Active gaseous fluxes are at least partially selective in removing impur-
ities by reacting chemically with specific constituents in the melt to form
new compounds, which may be solid, liquid, or gaseous. Materials such as
metal oxides, chlorides, or sulfides may be found and removed from the melt by
skimming or volatilizing. Oxygen in the air is not generally regarded as an
active gaseous flux, but it is active when blown into a melt to remove some of
the impurities by oxidation. It is probably the only active gaseous flux used in
the secondary-copper industry. Chlorine and boron trichloride are used
extensively to refine aluminum-base melts by chloridization of impurities.

Metallic fluxes are either pure metals or alloys which can be introduced
into a melt to produce a refining action which is somewhat similar to the
affects of nonmetallic fluxes. Best results are usually obtained when the
fluxing metal is alloyed with a base metal which is also the base metal of the melt to be fluxed. In other words, the metal fluxing agent used for copper-base alloys would also be alloyed with copper as a base metal. Fluxing alloys are usually made in several compositions as a means to control the rate at which the agent is released into the melt. These fluxing alloys are conveniently classified according to characteristic functions they perform. Accordingly, they are known as deoxidizers, degasifiers, densifiers, stabilizers, and fluidifiers. Many of the common flux alloys provide two or more of these functions simultaneously. Fluxing alloys are usually available in shot or cake. Some melters may use these as master alloys to produce others that are not available commercially. Phosphor-copper for example is produced as 10 to 15 percent phosphorus alloy for deoxidizing. The phosphorus reacts readily with oxygen in the melt, thus removing it as an oxide. The copper released from the flux alloy by the reaction simply becomes part of the melt. In some cases, the flux alloy is added so that the excess phosphorus will alloy with the melt as one of the desired alloy constituents. In this case, the flux alloy is used as a deoxidizer and a hardener. There are many other fluxing alloys available for making various copper-base alloys. Included are binaries of silicon, manganese, magnesium, lithium, and cadmium. Manufacturers of these fluxes are generous with complete instructions for their proper application. Departure from specific instructions may lead to abnormal behavior and serious contamination of a melt.

Reverberatory Furnace

Melting, alloying, and refining can be done in any one of several furnaces usually selected for a given application on the basis of quality and quantity of scrap and waste materials to be processed. The reverberatory furnace is a box-like, refractory-lined enclosure designed to heat the charge by both conduction and radiation. The furnace is usually made with magnesite brick walls, fused magnesite bottoms, and suspended magnesite brick roofs. Capacities of stationary reverberatories used in secondary smelters range from a few thousand pounds to 100 tons or more. The side or end-charged, arched-roof, tapping furnace is used most extensively. Figure 6 illustrates a typical reverberatory; figure 7 shows a reverberatory designed especially for convenient overhead charging; and figure 8 shows a tilting cylindrical reverberatory furnace.

Charge materials used in making brass or bronze ingot should contain a minimum of 40-percent copper in order to prevent excess slag accumulation, which reacts with the refractories and shortens the life of the furnace lining. Charges comprise batches or lots of scrap selected to produce a melt of the desired composition with a minimum of flux and as little dilution of metal constituents as possible. Scrap is charged at regular intervals until the furnace is filled to capacity. Melting is more efficient if light scrap is densified by bailing or briquetting. Oxidation and volatilization losses from copper-base alloys are usually kept to a minimum by rapid melting in slightly oxidizing atmosphere with a fairly fluid slag cover.

Reverberatory slags usually contain metal values that can be recovered in the blast-furnace. Slags produced by small secondary plants are frequently sold to primary smelters on the basis of copper content only. Some plants
grind the slag and recover metallic constituents in milling operations before
the slag is sold.

The following example illustrates the performance of a reverberatory fur-
nace (12 by 26-1/2 feet, with 36-inch depth for molten metals) operating in a
48-hour cycle:

Total weight of scrap charged: 190,000 pounds.

Charge, average composition, percent: 82 Cu, 4 Sn, 5.26 Pb, 0.6 Ni,
0.6 Fe, 0.18 Sb, 0.07 S, 0.0 to .05 Al, nil As, and 7.25 Zn.
FIGURE 7. - Reverberatory Furnace for Melting and Refining Copper and Brass Scrap. Designed for Charging Through Roof Doors.

Product, average composition, percent: 84.5 Cu, 4.4 Sn, 5.25 Pb, 5.4 Zn, 0.15 Fe, 0.22 Sb (from babbitt in tin scrap), trace Al, and Si.

Flow: Broken window glass, 500 pounds, removed after melting. Addition of 500 pounds glass and sand as long as it dissolves freely. Total flux 4000 pounds.

Slag weight: Approximately 10,000 pounds.

Slag composition, percent: 20 zinc oxide, 20 iron oxides, 35 silicon dioxide, 20 copper prills, 5 to 8 copper oxide, small amounts of cadmium oxide, magnesium oxide, and aluminum oxide.

Stack losses: Mostly oil, grease, and moisture. (Flue dust recovered.)
FIGURE 8 - Tilting Cylindrical Reverberatory Furnace for Melting Brass, Aluminum, and Gray Iron.

Weight balance:

<table>
<thead>
<tr>
<th>Charge</th>
<th>Pounds</th>
<th>Product</th>
<th>Pounds</th>
</tr>
</thead>
<tbody>
<tr>
<td>Scrap</td>
<td>190,000</td>
<td>Brass ingot</td>
<td>178,400</td>
</tr>
<tr>
<td>Zn, Sn, Pb (added)...</td>
<td>2,000</td>
<td>Slag</td>
<td>10,000</td>
</tr>
<tr>
<td>Flux</td>
<td>4,000</td>
<td>Volatilized material,</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>oil, grease, moisture.....</td>
<td>2,000</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Recovered flue dust........</td>
<td>4,000</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Estimated losses, gases,</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>dust, and others...........</td>
<td>1,600</td>
</tr>
<tr>
<td>Total</td>
<td>196,000</td>
<td>Total</td>
<td>196,000</td>
</tr>
</tbody>
</table>

Fuel consumption: 19,000 cubic feet of natural gas per hour for melting, slightly less for refining.
Time cycle:  
Charging and melting......30 hours  
Refining..................10 hours  
Pouring...................8 hours.

Metal recovery:  93 percent.

Rotary Furnace

The rotary furnace is designed to provide efficient melting and refining and convenient pouring of fairly large melts. The capacity of the rotary furnace ranges from several tons to 50 or more tons of nonferrous metals. Many melters believe that it has a particular advantage over stationary furnaces for melting loose or bailed light scrap, because the rotary mixing action promotes better heat transfer to the melt and causes a more rapid coalescence of melted globules.

The rotary furnace is a cylindrical steel shell with insulating material placed inside next to the shell. Magnesite or chrome-magnesite brick is used for lining. Frequently a monolithic lining of either refractory is used. Brick linings are usually backed with a cushion of grain magnesite.

Linings may last 100 or more heats, and the capacity of the furnace may increase many thousands of pounds, because the lining erodes from slag and by abrasion; heat losses also increase proportionately. The cylinder is mounted with its axis in a horizontal position and is supported by piers and trunnions at each end. It is fired by oil or gas burners inserted through either or both trunnions. The flame is directed lightly on the surface of the flux cover. One or more charging ports, large enough for admitting fairly bulky scrap, are located on the side of the cylinder and a pouring spout is attached to the furnace at a level slightly higher than the slag level when the furnace is fully charged. Figures 9 and 10 illustrate typical rotary furnaces. Charging, alloying, fluxing, and sampling techniques are essentially the same as for the reverberatory furnace.

Following is a typical charge makeup to produce 85-5-5-5 ingot:

<table>
<thead>
<tr>
<th>Charge</th>
<th>Percent</th>
<th>Products</th>
<th>Percent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Red brass solids...</td>
<td>50.3</td>
<td>Slag..................</td>
<td>7.2</td>
</tr>
<tr>
<td>Light copper........</td>
<td>7.6</td>
<td>Splatters.............</td>
<td>1.8</td>
</tr>
<tr>
<td>Red brass borings...</td>
<td>18.5</td>
<td>Loss..................</td>
<td>1.2</td>
</tr>
<tr>
<td>Splatters...........</td>
<td>3.7</td>
<td>Ingot (85-5-5-5).....</td>
<td>89.8</td>
</tr>
<tr>
<td>Hard brass borings..</td>
<td>3.9</td>
<td>Total................</td>
<td>100.0</td>
</tr>
<tr>
<td>Radiators...........</td>
<td>13.7</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Scrap lead..........</td>
<td>.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Phoscorper..........</td>
<td>.1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Non-metallics.......</td>
<td>1.7</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total...............</td>
<td>100.0</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Flux comprises equal amounts of anhydrous soda ash and anhydrous borax, about 1-1/2 percent of charge.

**Crucible Furnace**

A fairly large tonnage of secondary copper is produced in crucible furnaces. These may be heated by gas, oil, coke, or electricity. The once-popular, coke-fired, pit furnace is seldom used today, however.

Crucible furnaces are used in the secondary-copper industry for melting clean, well-segregated scrap - mostly in foundries. Very little fire refining is performed in crucibles. Nonmetallic fluxes are used for a protective covering, but alloy fluxes may be added as a refining agent and as a means of introducing some constituents into the melt.
Scrap is usually melted in crucibles by the puddling method; that is, melting enough scrap to make a liquid puddle, then forcing freshly added scrap below the surface of the melt until it becomes part of the molten body.

Crucible furnaces may be either stationary or tilting, but the latter is more convenient and much preferred. The gas or oil-fired tilting furnace comprises a refractory-lined, cylindrical steel furnace shell with a crucible mounted inside. It has two pivot shafts extending horizontally from opposite sides of the cylinder near the top so that the pouring distance, when tilting the furnace, will be as short as possible. The crucible is mounted in the center of the furnace shell and is small enough to provide an annular combustion space between the crucible wall and the refractory lining. Gas or oil
burners, with flexible fuel supply lines, are mounted in a position to direct the flame tangentially into the combustion space. This prevents excessive flame erosion of the crucible or furnace lining. Figure 11 illustrates a typical gas-fired crucible furnace.

FIGURE 11. - Gas-Fired Mechanical Tilting, Lip-Pour Crucible, Melting Furnace.

Electric crucible furnaces (including high- and low-frequency induction and resistance types) may be either tilting or stationary, but the tilting type is used now almost exclusively. Electrical resistance furnaces are very seldom used for melting and refining scrap outside of the laboratory. Induction furnaces are particularly well-suited for melting relatively small batches rapidly. Some of the larger low-frequency types are now being made with capacities equal to the larger rotary furnaces. The crucible for a high-frequency induction furnace is placed symmetrically in the center of a hollow helical, water-cooled copper induction coil, as shown in figure 12. The crucible is thermally and electrically insulated from the coil, but the metal charge in the crucible is heated by electrical-eddy currents which are induced into the metal by a high-frequency magnetic field generated in the induction coil. The eddy currents are of such a magnitude that the charge metal actually melts because of its electrical resistance to the heavy current.
Low-frequency or line-frequency induction furnaces generate heat by the same basic principles but in a slightly different way. Referring to figure 13, heat is generated in the melt by very high currents induced into the charge metal by the primary winding of a transformer that is coupled magnetically to the melt through an insulated, water-cooled iron core. The furnace crucible is fashioned in such a way that the melt forms in a channel or heating duct which, when filled with molten metal, comprises a short-circuited secondary turn of the transformer. The induced voltage causes very high currents to circulate in the metal. The metal is heated because of its resistance to the flow of electrical currents. Primary and secondary magnetic fields react with each other to produce mechanical forces which may cause considerable turbulence in the melt. The turbulence has the desirable effect of causing rapid mixing and heat transfer to all portions of the melt. Some furnaces are equipped with electro-magnetic pumps which can tap metal from the crucible by the same forces which cause the stirring. A line of foundry induction furnaces is shown in figure 14.

Summary of Furnace Applications

The stationary reverberatory is the most practicable furnace for making very large tonnages of standard alloys from scrap. The rotary furnace is more flexible than the reverberatory, but the capacity is limited to moderate tonnages. Tilting and stationary crucible furnaces, either gas or electric, are used to advantage for making small melts of special alloys. Electric induction furnaces are increasing in popularity at ingot plants and foundries where special high-grade alloys are made. Advantages of electric furnaces include higher melting speed and precise temperature control. These help to defray the relatively higher cost of electrical equipment.

Open-flame stationary or rotary reverberatory furnaces give greater fuel efficiency than furnaces using indirect heating, but oxidation and volatilization losses may be higher if the melt is not protected by a slag or flux cover.
Mold Line Equipment

Melting furnaces are always associated with other equipment designed to receive the melt. Melts are usually tapped from reverberatories and rotaries into feeder ladles which transport the metal to a mold line for making conventional ingots. The mold line is a series of ingot molds placed on a rack which may be stationary or movable. If stationary, the molds are filled with metal poured from a portable ladle.

An automatic mold line is an endless mold-conveying system in line with, or on the periphery of, a large circular rack known as a casting wheel. The casting wheel may carry either ingot molds for alloy melts or anode molds, provided that the furnace operation is a step in the production of electrolytic copper.

Melting and refining furnaces are operated frequently in conjunction with a plant or mill to produce items such as rods, tubes, sheet, and similar products. When they are, the furnaces are tapped into special billet molds to make shapes for subsequent milling operations.

Automatic mold lines convey each mold to a position where it is filled from a header or feeder ladle. Some ingot makers use special auxiliary mold-conditioning devices in which the molds are sprayed with a mold wash and then dried thoroughly before the ingot is cast. Automatic devices are often used to sprinkle ground charcoal in the molds to provide a special smooth top on the ingots. Some ingot makers question the necessity or even the advisability of this practice. In the Kaufman Controlled Process, metal is melted and ingots are cast in an atmosphere of nitrogen to eliminate the need for charcoal topping.

Cast ingots are usually cooled by water spray or other means and dumped from molds and racked for shipping.

REFINING HIGH-GRADE COPPER SCRAP

Copper products smelted from low-grade scrap, slags, drosses, and sludges are eventually brought together with other impure copper products for fire

refining. Although some degree of refining is done in smelting and melting furnaces, the final pure copper is made by fire refining.

The refining furnace is either a stationary reverberatory or cylindrical tilting type with a capacity of 20 to 300 tons. The most satisfactory refractory materials are magnesite brick walls, fused magnesite bottoms, and suspended magnesite roofs for the stationary furnace; magnesite brick is used throughout for rotary furnaces. Super-duty firebrick is used extensively in furnace areas that are not in contact with molten metal. Full fire refining is often required to produce billets, slabs, cakes and bars for manufacturing plates, sheets, rods, and so forth. Copper ingots are produced for making copper-base alloys.

Fire refining is only partly completed when the metal is to be cast as anodes for further electrolytic refining; that is, when the copper contains other valuable metals which can be recovered from the cell sludge.

The first step in the refining operation is to melt pigs of black copper, blister copper, and high-grade scrap rapidly in an oxidizing atmosphere, until the melt begins to "work." "Working" is a bubbling action accompanying liberation of sulfur dioxide in the reaction between cuprous oxide (Cu$_2$O), dissolved in the melt, and cuprous sulfide (Cu$_2$S), contained in the melt. Copper and sulfur dioxide are produced according to the reaction:

$$\text{Cu}_2\text{S} + 2\text{Cu}_2\text{O} \rightarrow \text{SO}_2 + 6\text{Cu}.$$
The melt is skimmed after working has ceased, and a sample is obtained for observing the "set", which is a characteristic shape and appearance of the solidified sample, indicating relative amounts of some constituents within very narrow limits. It may be necessary to saturate the melt with Cu₂O in order to reduce the amount of other impurities by oxidation. Ordinarily, the melt will contain considerable Cu₂O after working stops. The Cu₂O is reduced by skimming the melt; covering the surface with a reducing agent, such as anthracite, charcoal, or coke; and then "poling" the metal. "Poling" is used to reduce the Cu₂O to copper and further remove sulfur. The ends of green wooden poles are inserted below the surface of the melt, where they decompose and expel gases and carbon and produce much turbulence in the melt. The gases act as a flux to purge some impurities from the melt, and the carbon reduces Cu₂O to copper. The "pitch" is indicated by the appearance of a fracture surface of a sample. The surface exhibits a texture of coarse brownish-red crystals, if the metal still contains large amounts of Cu₂O. As reduction continues, the fracture surface changes to a fine crystalline texture then to a fiberous appearance and finally when poling is finished, it acquires a satiny orange-red sheen. The copper is then "tough pitch" and is ready for casting ingots, slabs, wire bars, and billets.

Some silver and gold may still remain in the copper after fire refining. If these metals are present in substantial amounts, the copper will not be completely refined by oxidizing and poling, but will be made into anodes suitable for electrolytic refining. The anodes will contain about 99 percent copper and small amounts of silver, gold, lead, selenium, tellurium, and other metals. During electrolytic refining, the copper from the anodes is deposited as copper cathodes. These are melted and fire refined to produce oxygen-free, high-conductivity copper (99.9 percent Cu) which is also used to make billets, wire bars, ingots, or cakes. Considerable No. 1 copper scrap (99 percent copper) can be melted and refined with the cathodes in the cathode-refining furnace.

FIRE REFINING SPECIFIC TYPES OF COPPER SCRAP

Removal of Lead and Tin from Copper-Base Metals

Lead and tin are difficult to remove from melted copper scrap, particularly less than 0.005 percent. Several investigators have proposed methods to do this refining more simply and efficiently than is possible by techniques in present use. Stolarczyk and Ruddle\(^{11}\) have reported some success with a combination of oxidation and reflux refining. Their studies revealed that it is impossible to reduce the tin content of a melt below 0.01 percent by oxidation alone. However, both lead and tin can be removed when either of two slags are added to the melt prior to air oxidation. One slag is 56 percent silica, 25 percent phosphorus pentoxide, and 19 percent iron oxide. The other slag is formed by adding an alloy of 60 percent copper, 26 percent silicon, and 14 percent iron. Either flux is followed by air oxidation which reportedly removes

lead and tin at concentrations below 0.01 percent. The rate of removal is slow, more than one treatment is usually necessary, but impurities can be reduced below 0.005 percent. A copper loss of 6 percent is removed with the slag.

A slag of 50 percent iron oxide and 50 percent silica removes the tin more rapidly and the lead moderately fast. Copper loss in this case is about 3 percent. Best results are obtained with the addition of sodium carbonate for tin removal and phosphor-copper for lead removal. Both impurities are reduced from 0.1 to 0.002 percent. The slag is corrosive however, and requires extra protection for refractories at the slag line. Water cooling this zone appears to be effective.

Distillation of Zinc from Nickel Silver Scrap and Other Alloys

Several distillation methods have been used for refining zinc-containing, copper-base scrap alloys. Poland12 developed a method which is claimed to be particularly suitable for treating scrap brass or nickel silver. The unique piece of equipment in this process is a specially-designed, high-temperature, electric-resistance distillation furnace. The following observations were made while processing 6,000 tons of scrap: The scrap varied in composition between the limits: 5 to 20 percent nickel, 15 to 30 percent zinc, 0.01 to 2.5 percent lead, 0.05 to 0.5 percent iron, 0.15 to 0.5 percent manganese; the balance was copper. The zinc averaged about 20 percent and nickel 14 percent. About 225 tons of leaded nickel silver (keys), 250 tons of cartridge cases, and some scrap monel were also treated.

Nickel silver scrap was melted down in a resistor-type electric furnace and transferred in a continuous molten stream to the distillation furnace. Volatilization of zinc during meltdown was eliminated by use of 5 to 10 percent rasorite flux. Volatilization during transfer of metal to the distillation furnace was prevented by confining the molten stream to a closed system. The zinc evaporated immediately when the stream entered the distillation furnace. The furnace temperature was raised from 2,200°F to 2,330°F while zinc was distilled from nickel silver scrap. It was raised from 1,850°F to 2,050°F while distilling zinc from brass. Distillation was continued until nearly all of the zinc was evaporated. This situation was indicated by a rapid drop of temperature in the zinc condenser. About 99.5 percent of the zinc was condensed as liquid metal, but some blue powder was formed. Residual metal in the distillation furnace was transferred to the converter, either in a molten state, or as shot metal. Distillation furnace residues from low-lead nickel silver were refined during 5 hours of blowing and skimming. Residues from lead-containing charges required about 4 hours longer. No flux was used with nonleaded charges, but a flux of sand and sodium carbonate was used on leaded alloys. Leaded material was blown until a sample showed an insoluble residue of nickel oxide when dissolved in nitric acid. Scrap iron was then added to reduce nickel and copper oxides in the slag. The metal was then skimmed and green poled as in standard fire refining of copper.

Brass scrap was treated in the same way as the copper-nickel scrap, except that the degree of blowing in the latter case was determined by microscopic examination for oxygen.

Cupronickel ingots obtained from the nickel-silver scrap contained 15 to 20 percent nickel, 0.0 to 0.05 percent lead, 0.2 percent iron. Manganese, zinc, tin, silicon, and aluminum were each present at less than 0.002 percent; the balance was copper. A saleable slag was produced in the converter.

Copper ingots obtained from treatment of refinery-grade scrap brass contained 0.25 percent nickel, 0.002 percent iron, 0.01 to 0.05 percent lead, 0.002 percent tin. Manganese, silicon, aluminum, and zinc were nil. The amounts of arsenic, antimony, selenium, tellurium, and sulfur were about the same as contained in the electrolytic copper.

**Volatilizing Zinc from Brass Scrap**

Kohlmeyer\(^13\) was able to recover copper from scrap brass and bronze by a similar but somewhat simpler method. The scrap was heated in a neutral atmosphere for long periods in a rotary furnace. Zinc vapor was burned to zinc oxide at the mouth of the furnace; tin and lead were not volatilized. Zinc oxide was recovered in a dust collecting system, while lead and tin were removed from the copper residual metal by blowing with air to form copper stannate and lead oxide. Tin could also be volatilized by blowing the copper with air in the presence of coke.

**Recovery of Tin and Copper from Bell Metal**

Kroll\(^14\ \text{and}\ \text{Kroll}\)\(^15\) describes a method used in 1915 in Germany to recover pure tin and copper cathodes from bell-bronze scrap containing 20 percent tin, some silver, and other noble metals. The scrap was melted in a cupola and charged as liquid to a magnesite-lined drum converter with lateral tuyeres. Solid scrap could also be charged with coke to the converter and melted by blowing with air. The residual copper was blown to about 97 percent purity and poured into anode molds after skimming. These were electrorefined to produce copper cathodes. Gold and silver were recovered from the cell sludge. Oxides of tin, zinc, and silver were produced in the converter and collected as baghouse dust. It is believed that SnO\(_2\) was formed in the melt by the air blast and then reduced to SnO by coke and excess copper; the SnO probably burned to SnO\(_2\) at the mouth of the converter. The baghouse dust was leached with sulfuric acid to eliminate ZnO. At times, lead was partially removed with caustic soda. Tin oxide was reduced to metal in a reverberatory to make tin anodes containing about 80 percent tin. Pure tin was produced by sulfostannate electrolysis.


Melting Copper-Clad Steel Scrap

Craig\textsuperscript{16} describes a wartime proposal for processing copper-clad steel scrap in a cupola. The process is based on an observation that the iron-copper system is the only metallic binary in which all possible compositions form a single molten liquid, but in which combinations within a specific composition range, separate into two conjugate liquids at elevated temperatures. This suggested the possibility of melting copper-clad steel scrap in a cupola to form two separable products, one, a relatively pure copper with little iron, the other, an iron-base alloy with little copper.

Recovery of Copper and Zinc from Gilding-Clad Steel Scrap

Craig\textsuperscript{17} also describes a wartime suggestion for leaching gilding-clad steel with molten lead. This proposal is based on the composition diagram for the copper-lead system which shows formation of two liquid layers in the composition range between 40 and 90 percent lead at temperatures of 950° to 1,000° C. The two metals are completely miscible at higher temperatures. Zinc and lead are known to be miscible at 790° C. and higher. It is presumed that no undue immiscibility occurs when copper, lead, and zinc are mixed; iron and lead are known to be completely immiscible at temperatures considered. It is likely that gilding cladding could be dissolved from steel in a lead bath at 1,075° C. The lead bath should continue to dissolve copper and zinc without any effect of the steel, within reasonable time limits.

Lead solvent, with considerable dissolved copper and zinc, would be allowed to cool slowly from a copper-zinc liquid phase and then quickly freeze to a crust which could be removed from the melt. An alternative method for separating the two phases would be to chill cast the homogeneous mixture, then separate the lead phase by sweating or centrifuging at 400° to 600° C.

CHEMICAL RECOVERY PROCESSES

A small quantity of scrap material is used for making metallic copper, brass and bronze powders, and copper salts and oxides by chemical processes. Chemical processes, like pyrometallurgical processes, usually require precise segregation of scrap and waste materials and some form of preliminary preparation. Chemical methods, like other recovery and refining methods, are most efficient when compositions of source materials are known accurately, and maximum process control can be maintained at every stage.

Although only small amounts of scrap copper are presently consumed in the United States by chemical processes, interest in these processes appears to be increasing. This is true even though a number of the methods are not considered practicable, except in times of short copper supply, as during war.


\textsuperscript{17} Work cited in footnote 16, p. 32.
Methods described here are but a few of many chemical processes developed specifically to recover copper and copper compounds from scrap and waste materials. Readers are referred to the list of references in the appendix for additional information on this subject.

**Copper and Clad Steel Leaching Processes**

Craig\(^{18}\) described a number of war-time processes for recovering copper and zinc from gilded and copper-clad steel scrap. Some of the methods described would be impractical at present, but the technology is sound and of academic interest.

**Ammonium Sulfate**

Copper cladding was leached from scrap iron with ammonium sulfate and later precipitated with sulfuric acid to produce copper sulfate and to regenerate ammonium sulfate for recycling.

**Cupric Ammonium Carbonate**

Cupric ammonium carbonate, in an excess of ammonium carbonate, reacts with copper cladding to produce cuprous ammonium carbonate on a weight for weight basis. The resulting copper ammonium carbonate solution is regenerated to new leach solution by oxidizing with air to produce the cupric condition. In other words, cuprous carbonate and oxygen react in the presence of ammonium carbonate to form cupric ammonium carbonate which is used as leach liquor in the next cycle. An equivalent of the leached copper is removed from the saturated leaching solution by distillation. Copper oxide is formed and ammonia and carbon dioxide are recovered as ammonium carbonate. Solvent losses in the leaching cycle are theoretically zero, and only oxygen from the air is consumed, appearing in the copper oxide product. Copper-free steel scrap is shipped to steelmills, and the copper oxide is reduced and fire-refined to produce an electrolytic-grade copper.

Ammonia leaching was used successfully during World War II to recover large tonnages of copper from scrap, specifically to produce copper-oxide for ship-bottom paint.\(^{19}\) The process was used for treating scrap brass and bronze turnings and shavings from munition plants, electric motors and generators, transformers, busbars, and cables. Metal armor and vinylite plastic were salvaged from Navy scrap cable in the process.

\(^{18}\) Craig, C. L., Recovery of Copper from Clad Steel Scrap: AIME, Metals Technol., October 1943, pp. 46-50.

\(^{19}\) Engineering and Mining Journal, Calumet and Hecla Recovers Scrap Copper: Vol. 146, No. 10, October 1945, p. 89.
Ammonia-Carbon Dioxide-Copper

A process\textsuperscript{20, 21} for producing powdered copper from copper scrap formerly called the "Chemico Technique" has been investigated rather thoroughly by two firms licensed by Chemetals Corp. of New York. The process which is now beyond the pilot-plant stage, includes the following five steps: (1) Leaching, (2) purification, (3) reduction and powder washing, (4) drying, crushing, and classifying, and (5) standards and controls.

Copper scrap such as radiators, radiator leaves, or No. 2 scrap is leached at atmospheric pressure and 100° to 140° F. with a solution of ammonia-carbon dioxide, water, and dissolved copper. Solutions maintained at atmospheric pressure and normal leaching conditions present no corrosion problems in carbon-steel equipment. The rate of leaching depends upon the temperature, physical character of the scrap, and velocity of leaching solution. The dissolved cupric copper in the leach solution combines with metallic copper in the scrap to form cuprous copper in the leach solution. Some of the leach solution is oxidized with air to convert cuprous copper back to the cupric state so that the solution will again be effective for leaching. Zinc in the scrap dissolves in the solution at about the same rate as copper, so that it becomes necessary to maintain the zinc content at a fixed amount by adding zinc, if necessary, or extracting zinc in an extraction unit. Other metals such as lead and tin are much less soluble in the liquors and are recovered as oxides or carbonates in the leach tank residues. The small amounts of lead and tin dissolved in the leach solution are removed by high-speed centrifuging to purify the liquor before reduction of dissolved copper.

Reduction is accomplished in an autoclave at a temperature of 400° F., with an over-pressure of hydrogen gas produced in a standard gas reform unit. Reduction is not carried to completion, because some dissolved copper must be recycled for subsequent leaching, and copper powder of highest purity is produced by this method. The slurry of partially-reduced liquor and copper powder is discharged through heat exchangers into a decanter. A portion of the decanted liquor is returned for leaching and some is passed through a zinc-boil unit. The copper powder is centrifuged to 10 percent moisture and dried completely in a hydrogen atmosphere. Dried copper is crushed and classified in a closed system. Classified powders are blended to maintain uniformity of product. Two types of powders are produced: (1) lower-density powders for friction materials and copper brushes, and (2) higher-density powders for sintered bronze bearings and similar items. Considerable progress has been made recently in the use of copper powders for producing commercial products.\textsuperscript{22}

Copper and Zinc Salts from Brass

Kazarnovskii and Zvenigorodskaya have developed a process for recovering copper and zinc salts from brass. Scrap brass is oxidized by heating in a current of air; resulting oxides are leached with a solution of sulfuric acid. Then dry brass fines or turnings are added to the pregnant solution as a source of metallic zinc, which precipitates an equivalent amount of copper. The precipitated copper can be converted to copper sulfate, and the zinc can be recovered as zinc sulfate.

Copper Sulfate from Scrap

Copper sulfate is produced commercially by digesting No. 2 scrap wire, or its equivalent of mixed heavy and light scrap, in hot aerated sulfuric acid solution.

Careful sorting of the scrap is required to minimize chances for introducing zinc, iron, and other soluble materials in the copper feed. Scrap is prepared for feed to the digester by melting in a reverberatory furnace and tapping a molten stream into a water pit to prepare shot. Shot is removed from the pit by lifting a wire basket located at the bottom of the pit. The most desirable forms of shot are those having large surface areas, controllable to some extent by the temperature of the molten copper and use of water jets where the molten stream strikes the water in the pit.

Digestion of the shot is accomplished in batches in lead-lined wooden or steel towers and requires 5 to 15 hours for completion. Bureau of Mines studies suggest the following procedures for optimum results.

Mother liquor of specific gravity 27° B. is pumped from crystallizers to the digesting tank which is completely filled with shot. Water is added to adjust the gravity to 24° to 25° B. This solution would have a pH of 1 to 2 and would contain about 2-1/2 pounds of copper sulfate crystals per gallon. Approximately 1-1/4 pounds of 60° B. sulfuric acid per gallon is added, sufficient to produce a solution of copper sulfate having specific gravity of 40° B. The solution temperature is raised to 200° F. and maintained during the course of the reaction. Air is added through spargers at the bottom of the digester in an amount equivalent to a cubic foot of air per minute for each cubic foot of material in the digester. In this type of operation the reaction rate is controlled primarily by the quantity and the effectiveness of air distribution. Acid content of the solution decreases linearly with time, and the course of the reaction is most easily followed by periodic checks for solution acidity. A batch is considered complete when enough acid has been consumed to bring the pH to approximately 1 or 2 and the specific gravity to 40° B., or slightly higher. Pregnant solution is pumped hot from the digester to filters and then to the crystallizer.

Impurities in the copper shot will contaminate the product depending on their solubilities in the solution under conditions at the end of the reaction. Iron and zinc, for instance, will go into solution and contaminate the crystals. Lead precipitates and is removed by filtering. Tin oxidizes and precipitates as a fine suspension that cannot be removed completely by filtration and will contaminate the product slightly. Except for lead and tin, most metallic impurities can be controlled best by carefully sorting the scrap before melting.

The crystallization step must be controlled, because too rapid cooling produces copper sulfate "snow." Too slow cooling produces excessively large crystals. The most desirable crystal is about the size of rice. Under proper conditions of cooling in the crystallizer and solution pH, uniform crystals of 100 percent pentahydrate can be produced. Excessive amounts of acid in solution cause formation of partially dehydrated crystals containing white streaks or white centers. After leaving the crystallizer the crystals are given a clean-water rinse, dried, and bagged. Mother liquor at approximately 90° F., containing 3 pounds of dissolved crystals per gallon, is returned to the digester for the next batch.

Small-scale experiments in Bureau of Mines laboratories have shown that this reaction can be accomplished in one-half hour by using fine copper powder violently agitated in solution with extremely fine air dispersion. Acid consumption decreased linearly with time, and only a slight excess of copper powder was required; this and a short reaction time suggest a continuous process. It was apparent in all tests that close control of reacting temperature, volume of air, and most important, proper dispersion of air were essential.

Copper Cementation

Cementation is a well known process in which the metal in a metallic salt solution corrodes and replaces a less noble metal. A typical example is the copper sulfate solution which dissolves iron and leaves a deposit of cement copper adhering loosely at the iron surface. This process has been used extensively to recover copper from dump rock, mine water, and low-grade ore bodies. An impressive installation at Copperton, Utah, is described by Martin²⁴/.

Pregnant solution of copper sulfate in slightly acid leach water is obtained by leaching waste rock from mining operations dumped in nearby gulches. The solution is conducted in a wooden pipe to the precipitation plant and there distributed through a system of wooden launders filled with loose, detinned, light scrap-iron. The iron in the scrap replaces the copper in the solution, yielding an accumulation of copper mud (cement) in the launder. Copper recoveries are 95 percent, and the cement averages 90 percent copper. Iron consumption was not reported, but the theoretical requirement is about 1 pound of iron for 1.14 pounds of copper. Actual iron consumption is estimated between 1 and 2 pounds per pound of copper cement.

Ion Exchange

The ion exchange process is particularly suitable for recovering metal in very low concentrations from waste liquids, such as spent plating and pickling solutions. Although the process is not, for copper, generally considered highly profitable, it gives a relatively simple method for realizing some return from operations such as those designed to prevent stream pollution in areas where it is prohibited by law.

There are several fairly complicated theories on the mechanism of ion exchange, but no particular one is now generally accepted. Fadgen describes the ion exchange process applied to metal recovery from waste solutions in the following rather simple terms: A liquid containing metal ions is passed through beds of granulated plastics, or resins, each resin granule having mobile ions attached. Trading of the mobile ions on the plastics with metal ions in the solution is called ion exchange. The process is different from ordinary chemical reactions because the resins are not soluble and only the mobile ions take part in the exchange.

There are many different types of resins developed for extracting many different materials. However, this complete cycle must occur for any successful application: (1) The resins must be exposed to the waste solutions so that the ion exchange can take place. (2) Exhausted resins must be reactivated before each successive exposure to waste solutions. (3) Metal is recovered from the process usually as a salt or an acid.

Fadgen presents examples for appraising the economy of ion exchange methods applied to the recovery of copper from acid rinse waters. In his example the rinse solutions were generated at a rate of 6,000 gallons per day and contained 0.0375 ounces of copper per gallon.

He determined that 9 cubic feet of cationic and anionic resins would handle the solution at a rate of 45 gallons per minute. At this rate, 45 ounces of copper could be removed before the cation exchanger became spent. The cation exchanger was used to remove copper and the anionic exchanger to remove phosphates, sulfates, chlorides, and so forth. About 11.7 gallons of sulfuric acid would be required to regenerate the cation exchanger and 108 pounds of sodium hydroxide to regenerate the anion exchanger. A total of 585 gallons of plating solution would be returned to the plating tanks from the cationic resins. Daily water consumption would be reduced 45 percent, with only 3,300 gallons discharged to the sewer.

Materials costs are not so impressive, however, if considered without regard to overall accomplishments. Estimated daily costs for sulfuric acid and sodium hydroxide are $23.06 and $29.66, respectively. The estimated value of copper sulfate recovered daily is $6.21.

27/ Work cited in footnote 26, p. 37.
Cuprous Cyanide from Scrap

An appreciable quantity of high-grade copper scrap is consumed in the production of copper cyanide for the copper plating industry. The copper scrap is used to reduce the cupric chloride in a heated sodium chloride solution. Sulfur dioxide is used to complete the reduction. A sodium cyanide solution is added to the resulting cuprous solution to precipitate cuprous cyanide, which is washed and dried.

SELECTED LITERATURE REFERENCES

Smelting


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**Milling**


**Electrolytic Processing**


**Ion Exchange Recovery**


Chemical Extraction From Various Waste Solutions

