THE MANUFACTURE OF BLACK LEAD CRUCIBLES.

By James C. Booth, Ph. D.

[Concluded from Page 288, Volume VI.]

Pulverizing the Graphite.—The manner of grinding graphite being of considerable influence on the quality of the crucible, some details upon the subject will be proper and useful. The larger masses of graphite being roughly broken by hammer into smaller pieces, the whole mixture of pieces and fine stuff is fed into an ordinary bark mill, consisting of two cast iron inverted cones, the smaller with iron teeth on its outer surface, revolving inside the larger one, similarly armed on its inner surface. The lumps are caught and crushed between these cones, and the resulting coarse and fine powder, being immediately passed between ordinary mill stones, is ground to a more uniform and finer powder, which is then assorted by sieves into finer and coarser portions, the latter being again ground and sifted until the requisite fineness is attained.

I have nothing to suggest to improve the machinery or mode of grinding, but as I differ from crucible makers in regard to the degree of fineness, I here present my views and the reasons for them. To economize time the ground graphite, passing through the sieves and consisting of fine flour on one extreme and on the other of flakes that can be split by a knife or ground to thinner scales, is usually mixed directly with the wet clay.

I regard the coarser flakes of insufficiently ground graphite as positively objectionable, because they lessen the strength of the crucible, which is the supreme consideration. I have often observed the surfaces of spalls split off from a crucible while in use, and noticed on them graphite scales of $\frac{1}{8}$ inch or more in diameter, with a part of the same scales remaining on the crucible and in the same spot. The knife easily separated more scales from the corresponding locality on spall and crucible. The facility of splitting offered by these graphite scales increases the facility with which pieces of the crucible itself split off through the feebly adhering laminations of the graphite. I have often seen a half dozen such scaling plates of graphite on a spalled surface of 2 to 4 square inches, and instantly read the cause of spalling and of serious injury to the crucible. If those coarser and thicker plates of graphite had been ground, spalling would not have occurred. Graphite is a very feeble substance in the plane of its lamination but strongly resists a force at right angles to this plane, and, what is more extraordinary, it will do so at a white heat.

Hence black lead should be ground exceedingly fine, not leaving a plate of it more than $\frac{1}{20}$ to $\frac{1}{50}$ inch in diameter. I have tried crucibles made of black lead powder that would not quite pass through a 126 wire sieve, and found them excellent. The dust that passed through such a sieve consisted of about 75 pr. ct. of good and tough flakes, and 25 pr. ct. of a fine black powder, without apparent structure, consisting of siliceous sand and iron pyrites. "If fine grinding were adopted," the economic manufacturer asks, "What should be done with the fine powder passing the 125 wire sieve?" About $\frac{1}{8}$ of it, being siliceous, is not hurtful, but the $\frac{1}{8}$ iron pyrites is injurious, and although it is so exceeding small in quantity, yet it would be preferable to utilize it in one of three ways: 1. To separate it by a still finer sieve into good flaky graphite, to be used with the rest, and dirt, to be thrown away; or, 2. To use it in mixture with good material for covers, etc.; or 3. To sell the whole mass passing the 125 sieve, with other inferior residues, for stove blacking, &c. In such case it may sell for half the value of good black lead.

Clay.—Admirable as are the qualities of black lead to resist the action of a white heat, burning off as it does only partially and with some difficulty, it must, in order to hold melted metal, be held firmly in the crucible shape given to it and this is effected by clay, that when wet mixes easily with the powdered black lead, and when dried and burned holds it rigidly and at the same time resists the highest white heat of the furnace. A clay from Klingenburg in the Palatinate seems to be pre-eminently adapted for such crucibles, and I strongly advise employing exclusively the best quality, every lump of which has the Government stamp impressed on it.

I have tried lower grades of the Klingenburg, and some of our own clays (Amboy, &c.), but I have found none to combine the excellent qualities of the best Klingenburg. In its wet state it is superlatively plastic, and free from every trace of grit, and in this state can be blended most intimately with the fine scales of black lead. It is barely fusible by itself at a white heat, and when mixed with an equal quantity of graphitic scales, as in the crucible mass, it only softens at a white heat so far as to surround each scale, which it holds immovably in its place, while the equal quantity of scales prevents any further fusion, even when kept for 10 or more hours at the highest white heat. Viewed chemically, Klingenburg clay is a hydrated silicate, containing in round numbers about ⅜ alumina, ⅛ water, and the balance silica, with minute quantities of oxide of iron, and alkaline fluxing matter. In its employment for fire crucibles, the only consideration is its content of ⅛ water when calculating the weights of the ingredients.

Sand.—The third constituent of the graphite crucible is a rather coarse siliceous sand that will stand a high heat without flying into fine powder. Its action is mechanical, causing a more uniform air drying, previous to burning, so as to avoid cracking, and playing the limited part of a skeleton to maintain the shape of the crucible. A good infusible clay, hardened by burning, and broken and sifted as a coarse sand, was found to act as well as sand, but not perceptibly better.

Mixture for Crucibles.—The only point of agreement in the composition of the best crucibles seems to be that when burned they should contain over 50 pr. ct. carbon, as determined by analysis. The formula may vary within rather wide limits, but our experience with good crucibles of the best makers and with our own, indicated that the burned crucible should show 55 pr. ct. carbon on analysis. In calculating the quantity of black lead used in a crucible from the determination of carbon, it is only safe to add 6 pr. ct. for the siliceous sand and pyrites found in the black lead. Some of the choicest black lead grains now in commerce have scarcely 1 pr. ct. of foreign matter, so entirely has the earthly constituent of plants or the small percentage of ash in anthracite disappeared from the mineral graphite. The proportions of black lead, air dried clay and sand used by different makers, and by the same maker at different times, vary considerably, but the following is given as illustrating approximately the composition for making the convenient amount of about 2,000 lbs. of crucible mixture when burned. As a crucible maker, I prefer the weight of black lead to be 1,100 lbs., so that the burned clay and sand may together make up about 1,000 lbs., which will give the 55 pr. ct. carbon in the burned crucible:

Black Lead.	Air Dry Clay.	Sand.	Total, when burned.
1,100	1,000	120	2,100
1,100	950	160	1,996
1,100	900	200	1,990

Every manufacturer prefers such composition as, in his judgment, works best in practice.

Mixing.—The air-dried clay of a given weight for a batch of crucibles, after being roughly broken by hammer, is covered with water, and after being softened, by standing, to a thin paste, receives the due proportions of the finely ground and sifted black lead and sand, and is then partially incorporated by a shovel. Some makers, ambitious of reputation for quality, judiciously pass their hands through the incorporated mixture to detect and remove lumps of gravel of pyrites or quartz. No amount of hand or shovel mixing can equal or supersede the operations of the Mixer or Dolly-tub, a wooden vat of 4 or 5 ft. diameter and height, standing on end, to which the somewhat stiff pasty mass is transferred. Its simple machinery is a vertical shaft, revolving on the centre of the bottom, and provided with a series of horizontal knives, arranged at a distance from each other spirally, and extending nearly to the sides of the tub. The revolving knives, having their blades at a slight angle to the plane of revolution, cut the putty-like mass into separate strips which fall over the sloping blades, and again unite in neat layers, to be again cut, separated, reunited, in new positions. After thus working for some time a slide on the outside of the tub, near the bottom, is drawn and the blades slope in such a direction to their revolution that they pass the mass downwards and out of the sliding door. The protruding masses are cut off and thrown in above, and this operation, often repeated, has the effect of blending the whole into a stiff paste of absolutely uniform composition, so that each flake of the graphite, $\frac{1}{1000}$ inch diameter, is enveloped in plastic clay, and every particle of clay is in contact with a flake. The wonderful strength of the black lead crucible is due to this microscopic blending of graphite and clay.

To make the crucible still more perfect the finished mass of about a ton in weight should be laid by in a damp cellar, covered with cloths, and occasionally moistened. In spite of the restless and reckless driving of our American manufacturers, they recognize that in all clay wares, the ready-mixed masses kept for a long time make better wares than those freshly squeezed out of the Dolly tub. The experience of the Chinese for ages in making the finest clay wares, led a potter to accumulate a quantity of mixed batches, ready for use, during ten, twenty-five and more years, to be used by his son, grandson, etc., as in the case of some that had been similarly bequeathed to him.

Can this part of the manufacture of graphite crucibles, *i. e.*, making the dough, be improved? I offer the two following suggestions for consideration: 1. Scarcely any native clay is wholly free from occasional lumps of iron pyrites, which, happening to become located in the side of a crucible, might ruin the metal near it, or make a hole for the metal to escape, and in either case injure the reputation of the maker. Careful makers sometimes feel for such in the soft clay mass, but why may not this mass be passed between rollers of very small diameter, which would not pass the small gravel or lumps, but accumulate them on the feeding plate? If such arrangement will pay in brick or terra cotta ware, surely the costly crucible mass would bear it. 2. The roughly mixed crucible mass, made very liquid with water, might be passed through rotating sieves, of smaller meshes than the sand, and the slip evaporated to proper consistence on shallow furnaces similar to those employed in drying white lead. These suggestions can only be answered by the crucible maker on the gronnd of perfection of work, and economy.

Moulding.—All graphite crucibles, between Nos. 30 and 80, embracing those for steel, brass, silver, etc., are formed on plaster molds, centered on the potter's table, or throwing lathe. The weighed mass for a crucible, being repeatedly cut and "slapped," is pressed into the bottom of the mold, which shapes the outside of the crucible, and while rapidly revolving, a steel or iron profile of the interior is gradually lowered into the mass, which is thus pressed against the sides and raised to the top or the mold by revolution, and gives the form of the interior of the crucible. This revolving method, like the ancient potter's throwing lathe, is far more important in this art, because it effectually disposes every scale of graphite, tangentially to the cylindrical or conical walls of the crucible, not merely on the inside and outside, but throughout the whole mass. Here lies the great strength of the graphite crucible. It is a mass of strong scales of carbon, that will not break at right angles to their lamination, being rigidly held in their sheet-like disposal by burnt clay, that will not yield to any heat. It is a graphite crucible, infusible and unalterable, where the black lead is very much, and for a long time, prevented from burning away by its envelope of clay. It is a clay crucible, which will not soften in the highest ten-hour, white heat employed in the arts, and will endure ten times the roughest usage of any other material, except bar iron. For many years I employed excellent wrought iron crucibles for melting silver, but find that black lead crucibles are better adapted to the purpose.

The proposition to make a cheaper graphite crucible by merely pressing out the soft crucible mass in molds, without the loss of time in revolving, would utterly fail for lack of the strength of structure given by revolution. It like manner the suggested substitution of anthracite, or any other form of carbon, for graphite, would signally fail to approximate to the strength and other qualities of a black lead crucible.

Drying is effected by keeping the formed crucibles in the plaster molds for a time, and then ranging them on shelves in warm, dry air, most economically in the spaces around the upper half of the burning kiln. If dried too rapidly, or if frost be allowed to enter the drying-room, fine cracks, like crooked hair lines, barely visible, around the interior, indicate that the crucible should be remade from the foundation.

Burning.—The crucibles must be thoroughly burned, and yet so as to avoid burning out black lead, their chief source of strength and value. To attain this object they are burned in seggars, which more or less exclude air, using two seggars for each crucible, one inverted over the other, and yet graphite is sometimes partly burned out by the air entering between them. This has been partially obviated, by some makers, by smearing the joint on the outside with clay slip. Another method which I invented, but never put into practice, as I soon after gave up the manufacture, consisted in replacing the oxygen of the air in the crucible and seggar with carbonic acid. I proposed to effect this by putting a small quantity of anthracite, or of rather dry bitumous coal, around the inner part of the lower seggar, and also, if practicable, a little of the same inside the crucible itself, avoiding defacing scratches or marks, which would be suspicious in the eyes of a buyer. I still suggest this idea to the manufacturer, for as long as there is present an easier burning carbon than graphite, the former will be attacked to the safety of the latter; and, moreover, the carbonic acid formed and forming from the coal, excludes the oxygen of the air, or the latter is consumed immediately on entering. A dry gaseous coal that neither puffs nor melts would be best for this purpose.

To secure equal and uniform distribution of heat, the furnace should have, and usually has, many openings as firing hearths around its base, and when the burning is nearly complete, these should be more or less closed, so as to prevent the entrance of more air than is barely necessary to maintain the diminishing combustion while the furnace is beginning to cool. This is another method of preventing the loss of black lead, and, with the same object, the entrance of air from above downwards should be prevented or counteracted.

The amount of carelessness or ignorance or lack of attention to prevent burning out black lead and possibly impairing the strength of a crucible, may be readily ascertained on the burned ware. When the outside of a crucible has the usual black lead color, it is either not burned enough, and then quite soft to the nail or knife, or it is extremely well burned, to be determined also by hardness under knife or nail, or it may have received the outside cleanly operation of a brush, which is, of course, only outside, and condemnatory. An outside drab color, which is the prevailing one, indicates burning off black lead. If the drab color be very shallow and show the subjacent black by friction of the finger, then it is well burned, like the greater part of the crucibles in commerce. Even if a slight scratch with a knife show the black immediately under the outside, it still shows excellent burning. But if it require a little digging into the drab before coming to the black lead, say $\frac{1}{4}$ inch, I should be disposed to throw off one-fourth, or more, of its value. If it go to a half inch, I should reject such a crucible as worthless, because a large proportion of its life, its strength, has been burned out.

Use and Life of Black Lead Crucibles.—The most extensive use of black lead crucibles is to melt blistered steel and cast it into any desired form, whence it is termed cast-steel. Tens of thousands of crucibles are weekly employed for this purpose, the amount of steel at each melt being 75 to 90 pounds, and a number of such melts constituting a day's work. Hence the great multiplication of furnaces, or of crucibles in a gas furnace, when 1,000 pounds or more are cast at a time. The life of a crucible for melting steel varies with the nature of the steel to be cast. If it be a highly carbonized (fine) steel, a moderate white heat will soon melt it, and the crucible may be repeatedly used. If a low (common) steel be employed, approximating to bar-iron, such as a locomotive driving wheel, the crucible has to endure five (5) and more hours of the most intense white heat for a single casting, and yet it can endure several such meltings. A crucible will bear repeated meltings of tool steel, and in order to extend its life, the outside is often coated with a good clay slip, with or without an admixture of black lead, after each melt. The wonderful protective power of the clay, embracing each particle of black lead, shows the advantage of grinding the black lead to great fineness, making clay and graphite mutually protective. In melting steel, the melter has to consider the length of the time of melting, and, therefore, the quantity of additional carbon the steel is assuming from the black lead pot, which excess he neutralizes by adding a certain quantitv of scraps of bar-iron to the melt

To show other possible applications of the crucible mass I may mention that syphons have been constructed of it, and successfully employed for transferrino melted cast-iron from one vesssl to another.

Black lead crucibles are also extensively employed in melting brass, bronze, German silver, gold and silver. I here add a few data in relation to the last two metals. The crucibles employed at the U. S. Mint, at Philadelphia, are so called No. 70 pots, of three gallons capacity—being 9 inches in diameter at the top and 13 inches deep, both inside measurement. The walls are 1 inch thick at top, and $1\frac{1}{2}$—2 inches at bottom. In such a crucible we usually melt at once, every $1\frac{1}{2}$ hours, 5,400 ounces of standard gold ($\frac{9}{10}$ pure), or 370 pounds, avoirdupois; four melts, a moderate day's work, equal, therefore, 1,480 pounds, or 21,600 ounces. The value of such a single melt is over $100,000. One such crucible is run for four days with full confidence in its safety, and fearing no fracture or loss of gold; for although we have run them for six (6) days it is found the economy does not balance the possible danger of fracture and leaking. Taking the safe run of four days, we melt in a single crucible, with scarcely any loss, $1,600,000 worth of gold at the crucible cost of about $2.50.

We usually melt 3,500 ounces (=240 pounds, avoirdupois,) of standard silver in a crucible (No. 70) at one melt, and cast six such melts, without undue haste, in a day, making 1,440 pounds, or 21,000 ounces. Since it is safe to estimate its life at five days, we melt in one such crucible before it is laid aside 7,200 pounds (=105,000 ounces) of standard silver, or about the same amount in dollar value. This, however, is not all of its life, but only its manhood, for after doing duty for so long a time in perfect health, it is put aside to purify and toughen inferior silver and to gather into melts grains and residues, otherwise wandering off, for some time before it is thoroughly disabled and ground to powder, to recover the precious grains which it has concealed in its body.

To one who knows the ease with which alkalies attack clay at a high heat, and how readily fusing nitre will burn off carbon, even black lead, it is quite a surprise to witness the wonderful durability of the black lead crucible, when in its nearly worn-out condition it is used for fluxing residues, and we can only attribute its strength to the minute and intimate blending of clay and graphite, mutually resisting destruction.

No doubt the life of these crucibles is prolonged in melting bronze, brass, &c., because they are so tough through the blending of clay and graphite that they rarely break suddenly, but rather give notice of their intention to do so by gradually opening, and even then not until they have been worn thin by long use.

Some years ago I prepared some black lead crucibles with more than usual care in order to test their life. We made from 40 melts of silver, the lowest limit, to 60 melts, the higher limit, in a single crucible. I have no doubt that a careful study of the principles of the manufacture of black lead crucibles here developed, somewhat fully, and even perchance their further development, beyond my conception, can improve their quality so as to double the life and strength ascribed to them.

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NOTE ON CAMPHOR MOTIONS.

BY P. CASAMAJOR.

On the 4th of October, 1877, I read a paper "On the Motions of Camphor on the Surface of Water," before the American Chemical Society, in which I described experiments, which had led me to the conclusion that these motions were due to electricity.

The extraordinary motions which give an appearance of life to pieces of camphor, swimming on the surface of water, are not to be seen at all times. Very often camphor will remain motionless, while at other times the pieces gyrate with great animation. One of the earliest observers of these singular motions, Romien (1748), came to the conclusion that they were due to electricity, while subsequent investigators, among whom may be counted the great Volta, have generally decided that there is no connection between electricity and the motion of camphor on water.

I was led to believe that camphor motions were due to electricity by the results of experiments, of which I will give a brief account.

When pieces of camphor are thrown on water, they may remain torpid or they may gyrate with every appearance of life. In the latter case, the motions may be instantly arrested by dipping a finger in the water on which the camphor moves. If we have pieces of camphor lying quietly on water, they may be made to move by dipping into the water a rod of either glass, sealing wax or vulcanite, electrified by friction. After every immersion the glass is to be dried by wiping with a dry cloth or a piece of bibulous paper; and, before every immersion, the rod is electrified by rubbing with a piece of silk or flannel. After one or more immersions of the electrified rod, the camphor motions invariably start, and by a few additional immersions they increase in intensity. The fact that these