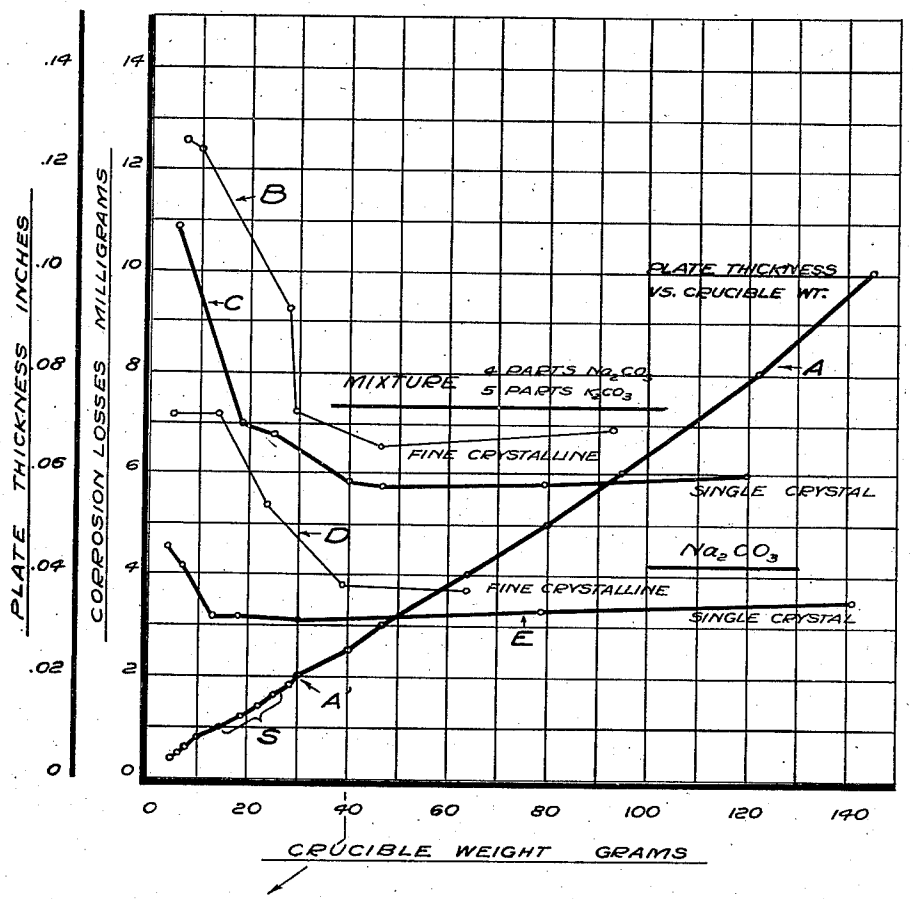
Jan. 3, 1939. J. S. STREICHER 2,142,660

PLATINUM CRUCIBLE

Filed Nov. 17, 1934 2 Sheets-Sheet 1

FIG. 1



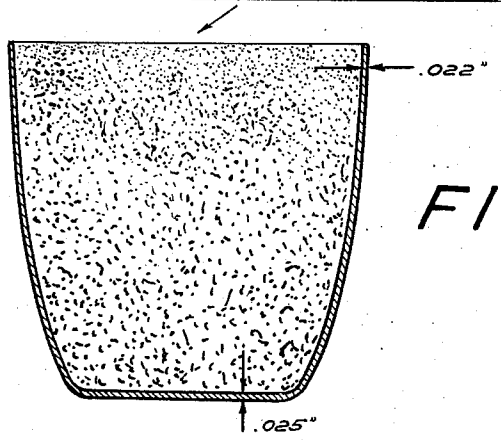


FIG. 3

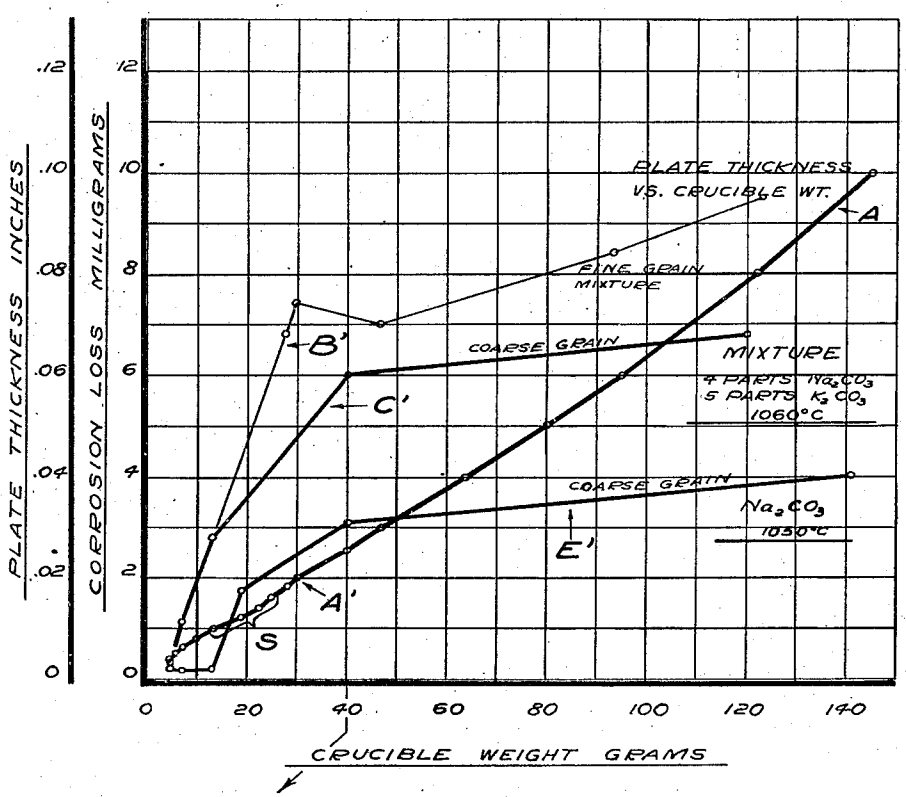
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Jan. 3, 1939. J. S. STREICHER 2,142,660

PLATINUM CRUCIBLE

Filed Nov. 17, 1934 2 Sheets-Sheet 2

FIG. 2



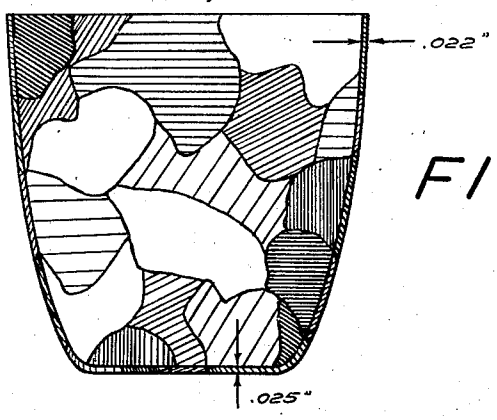


FIG. 4

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UNITED STATES PATENT OFFICE

2,142,660

PLATINUM CRUCIBLE

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Application November 17, 1934, Serial No. 753,478

5 Claims. (Cl. 263—48)

One of the most important tools of the analytical chemist is the platinum crucible. Since platinum belongs to the relatively rare metals it is a high priced metal. This fact always has been and still is today the reason for making the platinum crucibles with the least possible amount of platinum; the crucibles are made from such thin platinum sheets as to give the crucibles just the strength necessary for their use in the chemical laboratory. This rule has been applied to the manufacture of the platinum crucibles for generations; today a tacit agreement exists between the chemists and the manufacturers of the platinum crucibles which restricts the manufacture of the standard platinum crucible to this rule of thumb: "Platinum crucibles together with their covers shall weigh approximately as many grams as they hold cubic centimeters". Thus a platinum crucible with a capacity of 25 cubic centimeters weighs 25 grams (with a tolerance of .5 gram) including the cover. The weight of the platinum cover of this crucible is about 4½ grams; the weight of the crucible proper is, therefore, only about 20 grams. (Generally the platinum covers weigh from ¼th to ⅙th of the aggregate weight of the crucible and cover.)

This rule of thumb determines and limits absolutely the thickness of the crucible bottom and side wall. The crucibles are made from platinum sheets; these sheets are cut into circular discs or blanks and are spun over spinning forms made of steel or wood; during the spinning operation the circular blanks are pressed with a follower against the steel form; the follower has a face which nearly covers the bottom of the crucible-shaped steel form; in this manner a crucible bottom is obtained which always has the thickness of the original sheet; the crucible wall becomes slightly and gradually thinner from the bottom towards the rim of the crucible. Instead of spinning, other processes such as for instance pressing or deep drawing, are also used for forming the circular blanks into crucibles of the shape just referred to. Produced according to this rule of thumb platinum crucibles of the most widely used standard sizes have the following dimensions:

Capacity in cubic centimeters	Approximate weight in grams—		Thickness of bottom
	With cover	Without cover	
			Inches
10	10	7	.010
15	15	12	.0115
20	20	17	.013
25	25	21	.014
30	30	25	.0145
40	40	30	.015
50	50	36	.017
60	60	----------	.0175

When for instance .014" platinum sheet is spun into a 25 cubic centimeter crucible, the thickness of the sheet is reduced, from the bottom towards the rim, from .014" to .010" or .009"; sheets of a different gauge are reduced in thickness to an analogous extent, generally in about the proportion indicated.

Pure platinum as well as the well-known platinum alloys ("crucible platinum" with 3% iridium or rhodium, or "platinum-rhodium" with 3.5 to 4% rhodium) are treated alike when shaped into crucibles; the platinum-rhodium crucibles sometimes are made from even thinner sheets than the pure platinum crucibles; these alloys are slightly harder than the "crucible platinum".

The metal of all the platinum crucibles has an extremely fine crystal-grain. This fine crystal-grain is attained by the most painstaking hammering of the metal after the spinning process. The metal of the commercial platinum crucibles has a grain varying in size from .25 to .66 millimeter, or about 18 to 2 grains per square millimeter (measured at the bottom of the crucibles). Chemists as well as manufacturers consider those crucibles as of the best qualities which have the smallest crystal grain.

There are, according to my experience and research, three main causes of the destruction of platinum crucibles during their use in chemical laboratories:

1. When the crucibles are used for holding molten masses of alkali carbonate or potassium bisulfate, the thin, hot and therefore extremely soft and pliable crucible metal sags under the weight of the melt. The crucibles are considerably distended along the bottom section and as far up as the melt is filling the crucible; the crystals of the crucible wall are stretched; the intercrystalline spaces are enlarged. When the melts cool slowly within the crucibles or when they are quenched, the melts expand slightly and cause a secondary stretching of the metal. Both of these actions change the shape of the crucibles very rapidly. Wooden or other forms are often used to iron out these "bulges"; this procedure very often causes cracks within the metal. The alkali carbonate melts also contribute towards the destruction of the platinum crucibles chemically. These melts corrode platinum slightly; but this corrosion takes place especially along the edge of the upper surface of the melt, where alkali carbonate, platinum and air act upon one another at the same time; in this way a very distinct corrosion ring is formed at the inner side of the crucible; the walls of the platinum crucibles become gradually thinner at this place. Along this corrosion ring the metal begins to bulge under the weight of the crucible. When the crucibles are ironed out with the wooden forms, cracks are therefore particularly liable to form along these corrosion rings.

2. Platinum crucibles are most frequently heated with gas burners (Meker burners, etc.); the crucibles are heated in such a way that they are completely enveloped by the blue cone of the gas flame. This blue cone of the gas flame contains hydrogen (the amount of hydrogen in this blue cone is regulated by the law of the water gas reaction). Wherever this blue cone of any gas flame touches the platinum crucible, hydrogen diffuses through the platinum and causes reducing processes within the melts; metal oxides are reduced to the metals; these metals alloy with the platinum very readily and destroy the crucibles rapidly.

3. Every platinum crucible is handed to the chemist in the hard (cold worked) and highly polished state. Heating the crucibles for the first time (in the gas burner or in an electrically heated muffle; the temperature attained is seldom higher than 1100° C.) causes instant recrystallization of the metal. The distorted grain structure, attained by forming and hammering the crucible, is replaced by new, small and normal grains (an equiaxed grain, whose diameter is approximately the same in all directions). The "strain hardening" effect is completely lost with this change of structure; the metal becomes very soft and ductile. During the use of these crucibles it very often happens that, at restricted areas or places, an exaggerated grain growth takes place and an abnormally large grain is produced. Such exaggerated grain growth is the cause of many cracks in platinum crucibles.

When, in the chemical laboratory, platinum ware is destroyed, these three causes of destruction are not always equally active; the conditions which cause such failures vary with each laboratory; they are very seldom recognized and identified individually. Therefore "failures" of platinum ware are very seldom traced to their true source. In chemical literature they are, mostly recorded as interesting phenomena, essentially connected with the nature of platinum metal and therefore unavoidable. But another notion obscures the underlying facts. The platinum crucible is made today, with only minor changes, according to a tradition nearly 75 years old. Neither the process of making the crucible, nor the kind of crucible produced by this process is considered to have any bearing upon the failures of the crucibles when in use. The platinum crucible of yesterday and of today is really taken from the hands of the manufacturer like a constant of nature and in case any faults are found, the blame is put primarily upon the chemical quality or purity of platinum. But since platinum is generally refined to a much higher degree than any other commercial metal ever was refined, the chemical quality of platinum is least to blame for any failures.

Through an extensive research I have found that when the platinum crucible is varied in its physical structure the metal shows widely varying behavior under the conditions under which platinum crucibles are generally used. In producing such variations in the structure of the metal it was necessary to stop giving primary importance to the purely commercial viewpoint of the price of the metal; it was necessary to follow a line of development based solely upon the properties of platinum metal itself. These tests have enabled me to devise my present invention, showing a way of making a platinum crucible which combines with a least amount of platinum the highest resistance to corrosion, which reduces the diffusion of the hydrogen of the flame through the crucible walls to a minimum and which overcomes the possibility of the changes in the shapes of the crucibles during the fusion of alkali carbonate.

My researches showed that platinum can be best studied in its chemical and physical behavior when shaped into a large number of crucibles of the same capacity but with different bottom and wall thickness and when in such crucibles alkali carbonate melts are made under varying but always exactly determinable conditions. These tests and their results are of special advantage, as they correspond absolutely to the practical case in the chemical laboratory. In addition thereto, special tests have shown that alkali carbonate melts corrode platinum only insofar as they are dissociated to alkali hydroxide or alkali oxide and when oxygen, air or such gases as produce oxygen through dissociation (for instance carbon dioxide) are present. Soda ash and potash start to dissociate into the oxides at 300° C.; the higher the temperature, the more alkali oxide is produced within the melts (potassium carbonate is decomposed to a higher degree than sodium carbonate at the same temperatures), the greater is the corrosion, therefore, of the platinum crucibles by the alkali carbonate melts. The alkali carbonate melts act upon the platinum, under oxidizing conditions, not only in a purely chemical way; as tests have shown, the process of corrosion is also activated by the platinum itself and especially the platinum oxides which are formed during the initial corrosion and which are floating within the melt. Platinum is never attacked or corroded by alkali carbonate melts in a purely reducing atmosphere; on the contrary, in such systems hydrogen reduces to the alkali metals, the traces of alkali oxide formed above 300° C.; these metals are then deposited upon the inner surface of the platinum crucibles; these alkali metal deposits alloy with the platinum. Such melts, made under strictly reducing conditions, are always brilliantly white. If the alkali carbonate melts are made in a muffle which is electrically heated and where there is always an excess of air, fusion proceeds under purely oxidizing conditions. Tests have shown that this is the only way in which it is possible to obtain exact corrosion data for platinum at different temperatures. The melts resulting from such fusions are slightly colored (brown) by the platinum oxide which is formed by the corrosion of platinum; since the corrosion rate increases with the length of time and with higher temperatures the color of the melts is also intensified when the fusions are carried on for a longer time. When a gas burner is used for heating the platinum crucibles during these fusions, corrosion of the platinum also takes place, but the rate of corrosion is quite different from the above case; the melts corrode the platinum in a minor degree. Special tests have shown that the corrosion depends upon the rate with which the hydrogen within the flame diffuses through the platinum into the melt and upon the degree with which the flame enveloping the crucible blocks the air from entering the melt freely. These are the two factors which cause the reduced corrosion of the platinum when the crucibles are heated with a gas flame.

Applying the testing method explained above, to platinum crucibles with different bottom and wall thickness, there were ascertained a series of new and very important facts which had never been taken into account in the manufacture of

the standard platinum crucibles used in chemical laboratories:

1. Alkali carbonate melts made with a free excess of air (in an electrically heated muffle) corrode platinum most when the platinum sheets are very thin and very finely crystalline. The corrosion decreases with the increase in the thickness of the platinum sheet and with the increase in the size of the crystal grain.

2. The corrosion is least in crucibles made from platinum sheets of a thickness of .020" and over.

3. When the platinum crucibles are heated with a gas burner (Meker burner, etc.) the corrosion of platinum decreases with a decrease in the wall thickness of the crucible. The fusion proceeds under reducing conditions in crucibles made from sheets .018" thick and thinner. The reducing action within such thin-walled crucibles is caused by the diffusion of the hydrogen of the flame through the platinum into the melt.

4. Platinum sheets with a thickness of .020" and more act practically as an impervious shield against the hydrogen of the flame; within platinum crucibles made from sheets .020" and thicker, the fusion of alkali carbonate proceeds under oxidizing conditions similar to those obtaining with an electrically heated muffle.

5. During the fusion of alkali carbonate, platinum crucibles made from sheets .020" thick and more will sag but slightly or not at all under the weight of the melts. Thin walled platinum crucibles (thinner than .020") sag very quickly and easily under the weight of the melts.

These tests and their results show that the thin walled crucible which is made from sheets thinner than .020" (this includes all the commercial standard platinum crucibles, as they are made from sheets varying in thickness from .010 to .0175", according to the "volume by weight rule"), with the extremely fine crystal structure is in a physical state where it is most susceptible to destruction in the regular routine work of the chemical laboratory. These thin walled crucibles show the least resistance to the diffusion of the hydrogen of the flame. As they have an extremely fine crystal grain, they develop the highest catalytic activity when they are heated under oxidizing conditions; therefore, they show the highest corrosion rate with the alakali carbonate melts. The thin and finely crystalline platinum sheets are extremely soft when in the hot state; therefore, thin walled crucibles will sag and change their shape quickly under the weight of the melts.

The tests mentioned above indicate that the lack of stability of the standard platinum crucibles is overcome completely when the crucibles (of whatever capacity) are made from sheets of a thickness of at least .020" or more. My invention is based on these tests, and enables me to produce platinum crucibles far superior to the standard crucibles now employed, as to durability, permanence of shape, and resistance to deleterious influences such as corrosion by the melt contained in the crucible or impairment by the action of the hydrogen of a flame used for heating the crucible.

The superior results obtained by my invention appear very clearly from the data shown in the accompanying drawings, in which Figs. 1 and 2 are diagrams illustrating the corrosion conditions observed when crucibles of a predetermined capacity (here assumed as 25 cubic centimeters) are made from platinum sheets of different thicknesses and of different character and exposed to the action of two different melts; Fig. 1 represents the results obtained when an electrically heated muffle is employed, while Fig. 2 illustrates those observed when the crucibles are heated with a Meker gas burner; Fig. 3 is a vertical section of a crucible made according to my invention from finely-crystalline platinum; and Fig. 4 is a similar view of a platinum crucible the metal of which is of the so-called "single crystal" character.

According to extensive tests, the best results are obtained with my invention by using platinum sheets .025" thick. For the same volume or capacity, the new crucibles embodying my invention are heavier than standard crucibles such as now employed, as will be evident from the following table giving the approximate weight of platinum crucibles (crucible alone, without the cover) made from platinum sheet .025" thick:

Volume	New crucible, .025" wall	Standard crucibles	Increase in weight
Cubic centimeters	*Grams*	*Grams*	*Grams*
10	22	8	14
15	28	12	16
20	34	16	18
25	40	20	20
30	46	24	22
40	54	32	22
50	62	40	22
60	70	50	20

In the diagrams, Figs. 1 and 2, the line A indicates the relation between plate thickness (the thickness of the platinum sheet) and the weight of the crucible proper (without cover); that is, the ordinate of any point on line A indicates such thickness, according to the "plate thickness" scale at the left, while the abscissa indicates the weight of the crucible, it being understood (as explained above) that in each case the volume or capacity of such crucible has been assumed as 25 cubic centimeters. Thus, if the "plate thickness" is taken as .025", the line A indicates that the weight of the crucible will be 40 grams, which is also in accordance with the figure given in the above table with respect to the crucible having a capacity of 25 cubic centimeters. The portion S of the line A indicates the range of thickness (.010" to .0175") employed hitherto in making commercial standard platinum crucibles, whereas the portion of the line A from the point A' upward (thickness .020" or over) indicates the range of thickness to be employed according to my invention. As will be explained below, the point A' may be termed a critical point of the line A, in that conditions are materially different for points of the line A above A' from the conditions for points of said line below A'.

With an increase in weight from 14 to 22 grams of platinum for the standard sizes of the chemical laboratory crucible, the main and most powerful factors tending to destroy platinum crucibles are entirely eliminated and overcome, namely on the one hand, the diffusion of hydrogen into the melts when gas burners are used and the subsequent destruction of the crucible through chemical action; and on the other hand, the sagging of the crucibles and their subsequent destruction through mechanical causes. With the new "thick-wall" crucibles of my invention the fusion of alkali carbonates proceeds under oxidizing conditions even when gas burners are used for heating the crucibles. The modern textbooks of quantitative analytical chemistry advise the use of a small amount of niter together with the alkali carbonates in order to insure positively an

oxidizing melt when using a gas burner. The addition of niter to the melts, however, will never prevent the diffusion of the hydrogen into the melt; the fusion of such melts proceeds under oxidizing conditions for only a few seconds; platinum is corroded by such melts nearly in proportion to the niter content; subsequently the melts contain relatively large amounts of platinum oxide; the hydrogen diffusing through the thin walled crucibles reduces such platinum oxide to platinum and causes a platinum sponge deposit upon the inside surface of the thin walled crucibles. From such crucibles the cold melts can be separated only with difficulty; applying mechanical ways to separate the melts from such crucibles destroys the thin walled crucibles very quickly. The new "thick walled" crucible is least corroded by the alkali carbonate melts; the inner surface of the crucibles remains smooth for a very long time; the melts are very evenly heated within such crucibles; the cold melts are most easily separated from these crucibles. These improvements are attained with all the thick wall crucibles made from C. P. platinum, "Crucible platinum" as well as from the platinum-rhodium alloy containing up to 4% Rh.

As an example, I may produce platinum crucibles embodying my invention, in the 25 cubic centimeter size, as follows: "Crucible platinum" is rolled to a thickness of 0.120" and annealed at 800° C. for three hours. This platinum sheet is then rolled down to sheets of different thicknesses, each sheet being of uniform thickness, and the several sheets varying in thickness from .100" to .004". From each sheet I cut two circular pieces or blanks, about 66 millimeters in diameter, and anneal these for 15 hours at 800° C. Thereupon all these blanks are spun to form the shells or bodies of crucibles of 25 cubic centimeter capacity, and such shells or bodies are finished by hammering or other suitable operation. One crucible of each pair is recrystallized by heating to 1000° C. and then kept in the resulting finely crystalline state. The other crucible of each pair is transformed into a single-crystal crucible according to a method described below. When I speak of the thickness of the crucible wall, I mean that of the crucible bottom; the side wall is usually of somewhat smaller thickness, as has been stated above.

The crucibles of both types or series are subjected to corrosion tests as follows: Each test is made for a period of one hour. In one series of tests, I produce within the crucible (of 25 cubic centimeter capacity) a melt of 20 grams of soda (Na_2CO_3), in another series of tests the melt consists of 20 grams of a mixture of 4 parts of soda ash (Na_2CO_3) and 5 parts of potash (K_2CO_3). The fusion of the substance from which the melt is formed may be accomplished in an electrically heated muffle at 1000° C. (Fig. 1 illustrates the results of this treatment), or with the aid of a Meker blast gas burner at 1050° C. (the results being shown in Fig. 2). In the drawings, the lines B, C, D, E, B', C', and E' indicate the corrosion losses (due to the action of the hot melt on the platinum) with different melts, different heating agents, and different weights and characters of the platinum crucible. For each of these lines, the abscissae indicate crucible weights in grams, and the ordinates, corrosion losses in milligrams. Lines B, C, D, E relate to fusion in an electrically-heated muffle, and lines B', C', E' to heating with the aid of a Meker blast gas burner. Lines B, C, B', C' relate to the corrosive action of a mixture of soda and potash of the composition mentioned above, while lines D, E, E' relate to the corrosive action of a soda melt. The light lines B, D, B' illustrate corrosion in the case of crucibles in the finely crystalline state, while the heavy lines C, E, C', E' indicate the degree of corrosion in the case of crucibles of the single-crystal type more fully described below.

Fig. 1 shows clearly that with a crucible made from a platinum sheet .020" thick (point A' of line A, indicating a crucible weight of 30 grams), the corrosion rate approaches its lowest and approximately constant value, in the case of an electrically heated muffle. The corrosion rate reaches its minimum when the sheet is .025" thick (crucible weight 40 grams). Thin-walled crucibles have the highest corrosion rates on account of the greater chemical activity of the small crystal grains. Under otherwise like conditions, the corrosion rate is generally smaller when the crucibles are of the single crystal type, as compared with crucibles of the finely crystalline type.

Fig. 2 shows that when the crucibles are heated with a Meker burner, the corrosion rate approaches its peak with the crucible made from a platinum sheet .020" thick. With this particular mode of heating, the thin walled crucibles (less than .020" thickness) show the lowest corrosion rates, on account of the high rate of diffusion of the hydrogen of the flame through the crucible walls. Comparison of the lines B' and C' shows that in this case also the single-crystal crucibles are corroded less than the finely crystalline crucibles.

The third main cause for the destruction of the standard platinum crucible is exaggerated grain growth of the metal, caused by secondary recrystallization of the pure metal or the crucible platinum (chemists call it mostly "recrystallization"). This harmful, locally restricted exaggerated grain growth of platinum is caused, as tests have shown, when the recrystallized metal is in some places slightly bent or hammered and after such locally restricted mechanical treatment again heated to 1000° C. and above. Such mechanical treatment causes critical and local strains or strain gradients within such metal which is already recrystallized and which has been transformed through such recrystallization into an extremely fine grain; these strains are suddenly released when the crucibles are heated again to 1000° C. and above; they cause the small grains to coalesce locally to extremely large grains. Platinum crucibles with nests of such large grains develop cracks within such nests or at the boundaries where large and small grains meet. The changes in shape, caused by the alkali carbonate melts when contained in the standard, thin-walled and finely crystalline crucibles always induce the chemist, after each fusion, to apply to the crucible the above characterized mechanical treatment, "to reshape" the same; but such "reshaping" operations result, sooner or later, in exaggerated grain growth and ultimately in the destruction of the crucible. As has been stated above, platinum crucibles with a bottom thickness of .020" and more, especially those from .025" upward, do not change their shape under the weight of the melts; therefore, they never need "reshaping" after the fusions. By the use of my improved thick-walled crucibles, therefore, the third cause of destruction of the platinum crucibles is also materially weakened.

It is, however, possible to overcome this third cause for the destruction of the platinum crucibles entirely, by transforming the fine crystalline crucible into a single-crystal crucible. Special tests concerning the crystal growth of platinum, especially with wires and sheets, have shown that it is possible to produce the secondary exaggerated crystal growth of platinum metal not only accidentally but intentionally; it has been found that exaggerated grain growth in platinum can be improved to such a degree that the crystals in sheets and wires will grow to sizes up to 1 centimeter and even more in length and width. When this method is applied to the finely crystalline platinum crucibles, the metal of the crucibles is transformed in its entirety into an aggregate of such single crystals which have a thickness equal to that of the crucible wall and a length and width many times the thickness of the crucible wall; the whole crucible consists of a few tightly packed, large "single crystals". These large crystals are all of the same order of magnitude, that is to say, the crucible contains no small crystals. The term "uniform large crystals" as used in the appended claims, is to be understood as stating that the crucible consists exclusively of such large crystals. These single crystal crucibles have features and characteristics analogous to those of the well-known single crystal tungsten wires. In particular, the metal of these new platinum crucibles is in the completely unstrained condition, and free of local strains and strain gradients.

The finely crystalline platinum crucible is transformed into a single-crystal crucible according to the following method:

1. The crucible is painstakingly hammered to transform the metal through and through into the hard-worked and finely crystalline state.

2. This hard-worked crucible is quickly heated throughout to 1200° C. and above, preferably to 1600° C. and above. The crucible is kept at these temperatures from ½ to 2 hours. The metal recrystallizes instantly to a fine and normal grain. The metal is then in the dead soft state.

3. This platinum crucible (recrystallized completely and transformed into the dead soft state) is now hammered lightly with a highly polished steel-hammer all over its surface. The reduction in thickness resulting from this hammering treatment should preferably not exceed 10%; I have found the best results are obtained when such reduction in thickness amounts to about 3%. The best way to attain such a mechanical treatment is to take care never to apply the hammer twice to the same spot. Through such mechanical treatment critical strain gradients are created all over and within the dead soft metal.

4. This still finely crystalline, but dead soft and very lightly hammered crucible is quickly heated to 1200° C. and more, preferably to 1600° C. and more and is kept at these high temperatures for ½ to 7 hours. The higher the temperature, the shorter may be such annealing. Through this annealing the fine crystal grain of the crucible is completely transformed into such coarse grain that most of the crystals have a thickness equal to that of the wall, while their length and width may be .5 to 1 centimeter and even more.

5. Operations 3 and 4 are repeated at least once, preferably twice to four times. This causes an absolutely tight packing of the large single crystals.

Special tests have shown that with the same metal and the same wall thickness the single-crystal crucibles are the better, the finer the crystal grain of the original crucible is. According to these tests the finest grain of the metal is obtained when the platinum sheet used for the shaping of the crucible shells is reduced in thickness at least 75%, when rolled to the special size (best results are obtained with a reduction of 85% and even more). This reduction in thickness is generally performed in two or more stages with intermediate annealing, and these intermediate annealing operations are each conducted at a temperature preferably not higher than 800° C. and for a time of from 1 to 15 hours. These working conditions should also be observed in the method of producing the crucible shells by the spinning process and to the necessary intermediate and final annealings before the shells are hammered. The method of producing the single crystal crucibles can be applied to the commercial crucible platinum (platinum with .3% iridium or rhodium), and especially to the different kinds of the C. P. (chemically pure) platinum. The size of the single crystals increases with the purity of the metal. This method of producing single-crystal crucibles is not applicable, however, to an alloy containing platinum and more than 2% of rhodium. The method of producing single-crystal crucibles can be applied to all the crucible sizes, to the thin-walled as well as to the thick-walled crucibles. The best results are obtained with the thick-walled crucibles, which are made from sheets .020" to .060" thick, especially those .025" thick. Tests have shown that the critical strain gradients are most easily attained with these thick-walled crucibles through the hammering process explained above. With a sheet .025" thick, single crystals with an average length and width of more than six times the wall thickness are easily attained when "crucible platinum" is used; when C. P. platinum is used, "single crystals" measuring in length and width up to 20 times the diameter of the crucible wall (made from the .025" sheet) are obtained and even larger crystals. In any event, I prefer that both the length and the width of these "single crystals" should be at least three times the thickness of the crucible wall. With the thin walled crucibles the slightest strokes with the lightest hammer cause reductions and strains within the dead soft metal which exceed the critical range. When the sheets are thicker than .060" they are difficult to work; very strong strokes have to be applied to them; these strong strokes cause at the surface of the metal a high reduction whereas the core of the metal is hardly influenced by the mechanical treatment.

Platinum metal when transformed into the single crystal structure changes its qualities considerably. The finely crystalline crucible always has rather a dull tone in its color when in the highly polished state; the metal of the single-crystal crucible has an extremely high brilliancy; the metal attains this state of high brilliancy with the first secondary recrystallization, when it is annealed at 1600° C. This difference in color is most easily observed with artificial light. Since the brilliancy of the single-crystal crucible is its original and natural condition, it is never necessary to polish the surface of such a crucible. The standard platinum crucibles such as made prior to my invention, are polished with iron oxide or chromium oxide; small amounts of these substances always stick to the surface of the metal:

the single-crystal crucible has an absolutely clean surface. The natural brilliancy of the single-crystal crucible is very stable; only the most severe corrosive action can destroy this brilliancy. The forces which cause recrystallization and exaggerated grain growth in the fine crystalline crucible are completely deadened within the single-crystal crucible; the single-crystal crucible is therefore absolutely stable in this respect. Therefore repeated rehammering and subsequent reannealing is never harmful to the single-crystal crucibles (as it is to the thin-walled, standard and also to the thick-walled finely-crystalline platinum crucibles); such repeated rehammering and reannealing always improves the thick-walled single-crystal crucibles. Since the metal of such single-crystal crucibles is extremely ductile, slight changes in the shape of such crucibles are corrected very easily. The metal of the single-crystal crucible is transformed into the highest state of passivity; therefore such crucibles are chemically less corroded than the finely crystalline crucibles. At extremely high temperatures (1200 to 1700° C.) the metal of the single-crystal crucibles is slightly less volatile in atmospheres which contain oxygen, than the finely crystalline crucibles.

Taking into consideration all the facts mentioned before concerning the advantages of the heavy-walled platinum crucibles (.020 to .060" wall thickness, especially those which have a bottom thickness of .025") and the changes resulting from the transformation of such finely crystalline crucibles into single-crystal crucibles, it is most evident that the single-crystal crucible with a heavy wall constitutes the most perfect type of platinum crucible. In this type of crucible, with the use of the least possible amount of platinum, the following advantages are combined:

1. It is the most perfect kind of a single-crystal crucible. All the forces which could cause defects through recrystallization are absolutely killed.

2. Greatest mechanical stability; the crucibles do not sag under the influences of alkali carbonate melts; there is never any necessity for reinforcing the crucibles at the rim or for reshaping them.

3. Maximum resistance to corrosion. The crucibles have a smooth inner surface; the melts are always easily separated from the crucibles.

4. The greatest possible shielding action against the hydrogen of the flame; no reducing actions within the crucibles.

5. The most even heat transfer to the melts because of the greater thickness of the metal.

6. Smallest heating losses above 1100° C. in atmospheres containing oxygen.

7. No surface contamination of the metal by polishing powders. Natural, clean surface with high brilliancy.

The principles outlined above for making an improved platinum crucible can be applied to any shape of the platinum crucible used in the chemical laboratory, to the standard shape of the common platinum crucible (these crucibles have the shape of a truncated cone, the smaller end of which is used as the bottom), to the many variations between this shape and the simple, cylindrically shaped crucible, as well as to the recently introduced shapes which are known as "Interchangeable and combination crucibles".

Fig. 3 of the accompanying drawings illustrates a crucible made from finely crystalline platinum and embodying my invention, while Fig. 4 shows a similar crucible made from single-crystal material.

The term "platinum" as used in the appended claims is to be interpreted as including the various kinds of platinum which I have stated to be suitable for use in connection with my present invention, that is to say, so-called crucible platinum, as well as C. P. (chemically pure) platinum and platinum-rhodium alloys of the character explained above.

Various modifications may be made without departing from the nature of my invention as defined in the appended claims.

I claim:

1. A platinum crucible made of platinum in the completely unstrained condition and having a wall composed of uniform large crystals at least a majority of which are of a thickness substantially equal to that of the crucible wall and of a length and width at least three times the thickness of the crucible wall.

2. A platinum crucible made of platinum in the completely unrestrained condition and having a wall composed of uniform large crystals at least a majority of which are of a thickness substantially equal to that of the crucible wall, such wall thickness being at least .020" at the bottom.

3. A platinum crucible made of platinum in the completely unrestrained condition and having a wall composed of uniform large crystals at least a majority of which are of a thickness substantially equal to that of the crucible wall, such wall thickness being .025" at the bottom.

4. A platinum crucible made from C. P. platinum in the completely unrestrained condition and having a wall composed of uniform large crystals at least a majority of which are of a thickness substantially equal to that of the crucible wall, such wall thickness being .025" at the bottom of the crucible.

5. A platinum crucible made of platinum in the completely unrestrained condition and having a wall composed of uniform large crystals at least a majority of which are of a thickness substantially equal to that of the crucible wall, such wall thickness being within the range of from .020" to .060" at the bottom.

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