

Technical Note

Aspects of lead/acid battery technology

5. Dry charging of formed negative plates

L. Prout

Aydon Road, Corbridge, Northumberland NE45 5EN (UK)

(Received August 14, 1992; accepted February 18, 1993)

Abstract

The objective in the dry charging of formed negative plates in lead/acid batteries is to preserve the highly active sponge lead material from attack by atmospheric oxygen until the dry and unfilled charged battery is put into service. This review discusses the following methods that are commonly used for dry charging: (i) drying in a vacuum; (ii) drying by direct application of superheated steam; (iii) drying in an inert-gas atmosphere; (iv) removal of water by hot kerosene and subsequent drying in a closed kerosene vapour chamber, and (v) drying in the presence of anti-oxidants. The protection of dry-charge characteristics, rapid evaluation of dry-charge quality and testing for excess wax or oil inhibitors are also described.

Introduction

The dry charging of formed negative plates is aimed at keeping the highly active sponge lead material free from atmospheric oxidation until the dry and unfilled battery is filled and put into service, so as to behave as a fully-charged unit that is capable of giving its designed performance.

The oxidation of the formed negative material is rapid when it comes into contact with atmospheric oxygen. Thus, it is difficult to perform drying without losing some of the lead content of the plate to an oxidation reaction. For the most reliable results, the drying process has to be performed in an inert atmosphere, i.e., one free of oxygen. The basis of most dry-charging plants is the production of the required oxygen-free atmosphere concurrently with the application of heat to evaporate the absorbed moisture.

Many different dry-charging methods are employed in the industry; they can be classified as follows:

- (i) drying in a vacuum;
- (ii) drying by direct application of superheated steam;
- (iii) drying in an inert-gas atmosphere;

- (iv) removal of water by hot kerosene and subsequent drying in a closed kerosene vapour chamber, and
- (v) drying in the presence of anti-oxidants.

Drying in a vacuum

In this method, formed negative plates are taken out of the protective water baths and loaded in stacks on to the shelves of a pressure vessel, or placed in baskets hung within the pressure vessel. The method adopted is the one that exploits best the internal geometry of the pressure vessel.

The pressure vessel contains steam coils to raise the temperature of the plates above the boiling point of water and, thus, expedite evaporation. When the pressure vessel has been fully loaded, the door is closed and a vacuum pump is started. At the same time, steam is passed through the coils. Situated at the bottom of the vessel is a condenser unit through which the evaporated moisture passes and is condensed. At regular intervals, the condensate has to be drained so that the interior of the pressure vessel does not become saturated with water vapour. The degree of vacuum maintained is equivalent to ~28 inches of water gauge. Most vacuum dry-charge units require from 24 to 30 h at full vacuum to remove the absorbed moisture. The vacuum is maintained until no more condensate can be withdrawn. At this stage, the steam heating is turned off and the oven temperature is allowed to fall slowly. Throughout the cooling stage, the vacuum is maintained and is only released when the normal ambient temperature is recorded. At this point, the pressure vessel is opened and the contents removed to a dry storage area. The complete process takes about two days and, by comparison with other systems, is slow and expensive.

The system can give good results; the plates retain over 90% of their normal fully-charged capacity. The plates are vulnerable and will pick up atmospheric oxygen in store over a period of time. This causes the plates to partially discharge themselves unless a moisture inhibitor has been incorporated in the paste mix or added during the drying process.

Drying in superheated steam

Two basic types of superheated steam dryer have been used by the industry, namely, the 'Plews' or high-pressure dryer, and the 'Trouserpress' low-pressure dryer. The former consists of a pressure vessel connected to a supply of superheated steam. Wet, formed negative plates are put (spaced evenly apart) into wire baskets and the loaded baskets are then placed into the pressure vessel. The door is tightly shut and superheated steam is blown into the vessel for about 1 min with the outlet valve open. At the end of this minute, steam can be seen issuing out of the valve. If this is not readily visible, the initial period is extended until steam is detected. The outlet release valve is then closed and the plates kept under full boiler pressure for at least 2 min, or longer, if the steady pressure should happen to be below the stated value, for whatever reason. The pressure is then released by closing the steam inlet valve and opening the outlet release valve. The plates are now ready to be dried separately in an oven held at ~105 °C for 30 min to remove any traces of moisture that may have been present. At the end of the drying period, the plates are removed and cooled until they can be easily handled and stacked in a dry place until required for assembly into cells.

The rate of drying is dependent on the pressure of the superheated steam and the mass of the plates in the chamber. The exact duration is a matter for experimentation since the actual steam pressure available will vary from plant to plant. The system is fast but tends to be less effective than the vacuum system. The percentage of charge retained after drying is around 90%.

The low-pressure (Trouserpress) system consists of a fixed and a moveable (hinged) platen between which several shallow piles of plates are placed. The platens are preheated and brought into contact with the plates. The contact is maintained by hand pressure. As the water in the plates is evaporated, it envelopes the plates in a blanket that excludes air from the drying plates. This is because the steam produced can only escape around the sides of the piles of plates. An indication that drying is virtually complete is the absence of steam or water vapour. When this occurs, the platens are kept in contact with the plates for a further 2 or 3 min in order to ensure that drying is truly complete.

This system has been popular with smaller manufacturers as each unit is relatively inexpensive, neither is it costly in power usage. The method is slow compared with other more sophisticated systems and its economics are difficult to justify where labour rates are high. Another disadvantage is that some moisture inhibitors, incorporated in the negative pastes to give surface protection in the dried state against the pick-up of moisture, break down at the high temperatures between the platens and produce unpleasant and noxious fumes that may induce sickness in some operators. It is important before purchasing this type of equipment to check the compatibility of the incorporated moisture inhibitor to the equipment.

Inert-gas drying

The basis of this system is to surround the wet plates with an inert gas whilst heat is applied to evaporate the absorbed water in the plates. It is common practice to use simple tunnel ovens into which is bled an inert gas of which the simplest to use is nitrogen. The inert gas is most economically supplied from an *in situ* gas generator although, for convenience, bottled gas can be used. A disadvantage of the gas generator is the ease with which the level of oxygen can rise unless the generator is well maintained or remedial action is taken as soon as the oxygen level reaches the upper permissible limit. This limit is generally taken as 2% of oxygen.

The tunnel oven is usually divided into a number of zones to maximize the rate of drying without running the risk of plates overheating and distorting. The negative plates, after being taken out of the protective water baths following formation, are loaded on to a conveyor where each plate (or double plate) is separated from its neighbour. The passage is such that hot convective air can pass between the individual plates as they pass through the oven. The conveyor takes the plates through a water seal at the entrance of the oven (to prevent loss of the inert gas) and into the first zone. This is a hot zone where the temperature is sufficiently high to evaporate the bulk of the moisture present in the plates. The value of the temperature is dependent both on the speed with which the plates pass through the zone and on the length of the zone, i.e., the time the plates are in the zone. A typical temperature range is 160 to 180 °C.

From the high-temperature zone, the plates pass into a lower-temperature zone where the remainder of the absorbed moisture is removed. It is normal for this zone

to be some 30 to 40 °C lower than the first zone. Some means to adjust the temperature settings is necessary to cater for situations where there is a preponderance of thick plates whose greater mass of absorbed water requires a greater heat input, particularly in the first zone.

The inert gas is fed into the first and second zones and led away for reuse as the plates enter the third or final zone. Here, the plates are cooled before being dispensed at the take-off end of the oven.

In passing through the first zone, the temperature of the plates attains a value some 20 to 30 °C below that of the oven. This is due to the evaporation of the water so that there is very little change in temperature in passing from the first to the second zones. During the drying process, a considerable amount of steam and water vapour is produced. Both have to be led away to a condenser to be converted to water and then disposed. The resulting condensate is produced continuously and it is convenient to pass it to a collection area where it can be used as wash water in the lead processing areas. The condensate exit needs to be examined at relatively frequent intervals to ensure that it is free as it is usual to fit a water trap between the condenser and the collection area and the trap is prone to blockage by foreign matter.

The cooling zone is often cooled by water tubes and, in the process, the cooling water heats up. Where the cooling system is a recirculating one, there is a danger that the cooling water can become too hot for it to be effective. The temperature of the water should be checked regularly, and it may be necessary to insert a cooling unit into the water line.

The normal sequence of operations is first to start the gas generator. When the composition of the gas shows an oxygen level of less than 2%, it is passed into the oven for about 30 min to purge the oven atmosphere of air. It is essential that there is an oxygen recorder permanently connected into the gas system so that any increase in the oxygen level above the upper limit is quickly noted and rapid remedial action is taken.

Inert-gas drying ovens give good dry-charge retention; values in excess of 95% are not uncommon where good discipline is attached to the maintenance of gas inertness. The most common problems encountered with the ovens are a failure to keep the oxygen content of the inert gas below 2% and forgetting to increase the temperature of the first zone when dry charging thick plates so that the plates are not dispensed in a dry state.

If the emerging plates are visibly giving off water vapour, they will cause considerable trouble in service. Such plates should be rejected for immediate use and passed back for reforming and dry charging. Most problems are due to human fallibility, rather than to the limitations of the system. If the ovens are being used in a discontinuous manner with batches of plates and the temperature during the nonproductive periods is permitted to fall to ambient there will be physical movement in the framework of the equipment due to the sequence of expansion and contraction. As a consequence, the joints will be stressed and in time will leak. Regular checks of the gas consumption will indicate whether there are such leaks and whether there is a need for remedial action.

Attention should be given to the manner in which the plates are retained and taken through the oven. Simple hanging guides, using the plate lugs for location and retention on the guide, require care by the operators to hang the plates correctly and not in a misaligned condition. The latter will result in plates falling off within the oven where they may not be noticed and where they can build up and, in time, hinder

the free passage through the oven. The locations for the plates should be positive and easy to register.

Hot kerosene systems

The essential difference between this system and the inert-gas procedures is that an inert liquid is employed instead of an inert gas to prevent the ingress of atmospheric oxygen during the drying process.

Two variations of the hot kerosene drying have been adopted by the battery industry. Initially, a batch process was applied, but a subsequent procedure was to dry on a continuous basis. Both systems are intrinsically the same in principle and differ only in the method of transporting the plates.

As the boiling point of kerosene is well above that for water, the kerosene can be heated to 150 °C, or higher. At this temperature, the water in the negative material is rapidly converted to steam and taken up by the kerosene. Later, when all the moisture has been removed from the plates, the condensed steam is separated from the kerosene by simple stratification that relies upon the differing densities of water and kerosene, and their poor miscibility.

Figure 1 is a schematic of a typical batch system, as developed by the Tudor company in Sweden.

The sequence of operations commences with the removal of the formed negative plates from the protective water bath to a rinsing station where the plates are racked, preferably on stainless-steel frames, with a free space between adjacent plates of 0.5 to 1 cm. The loaded racks are placed into a rinsing tank where water is passed in

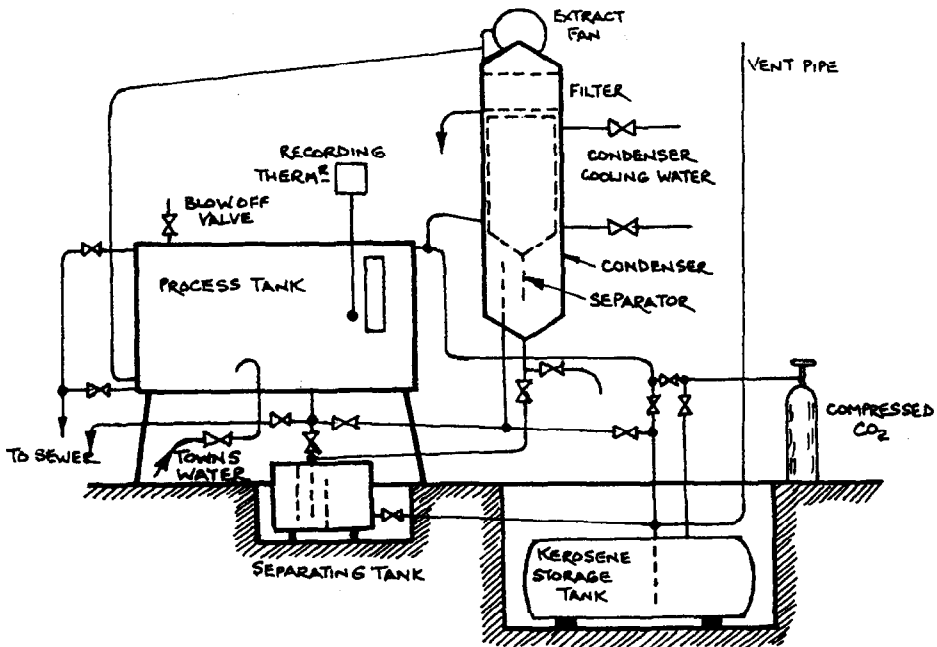


Fig. 1. Hot kerosene dry-charge system (Swedish Tudor).

and weired over the edges until the water in the tank contains less than 10 g/l H_2SO_4 . When this occurs, the loaded rack of plates is transferred to the process tank of the dry-charge plant. The tank will have been filled previously with town's water to the level marked on the sight glass. The loaded racks are lifted out of the rinsing tank and passed straight into the process tank so that the period, during which the plates are exposed to the atmosphere, is very short. In a typical plant, the full tank load would be about 2000 double automotive plates.

The cover of the process tank is then lowered and firmly closed with edge clamps. The water-release valves at the bottom of the process tank are opened and this allows the water in the tank to pass to discard. At the same time, the kerosene tank outlet valve is opened and compressed carbon dioxide is used to blow kerosene from the storage tank into the process tank. As a safety measure, a blowcock in the top of the process tank is opened and left open until all the water in the process tank has been replaced by kerosene. By adjusting the water release valves, the input of kerosene is balanced by the output of water and the plates are kept out of contact with atmospheric oxygen. Just before all the water has been removed, the compressed carbon dioxide is shut off and the remaining water is bypassed to a separating tank and the water release valve to discard (or sewer) is closed. This action is taken to prevent the release of kerosene to the sewer. The valve controlling the water inlet to the separating tank is closed when no more water is left in the process tank. This will be confirmed by the ingress of kerosene into the separating tank. Later, the kerosene in the separating tank, floating on top of any water, will be reclaimed and returned to the kerosene storage tank.

The level of kerosene in the process tank is then set to that in the separator (see Fig. 1) and the equalized level is adjusted to ensure that all the plates are fully covered with ~ 10 cm of kerosene. At this stage, cooling water is supplied to the condenser and continues until the end of the complete cycle. The safety cock and the kerosene outlet valve in the process and kerosene tanks, respectively, are closed so that the process tank is converted into a pressure vessel.

The steam supply valves are opened and superheated steam is passed through the heating coils. With steam at 70 psi and a full loading of 2000 double plates, the kerosene will reach boiling point in about 30 min. When this occurs, bubbling will be seen through the sight glass and this indicates that water is being driven out of the plates. Subsequently, the temperature of the kerosene rises to ~ 150 °C and then stabilizes. This usually takes a further 1 to 1.5 h. On stabilization, the heat is maintained for another 30 min or until there is no further bubbling, whichever is the longer.

When all the water has been evaporated, the process tank is drained of all kerosene and the safety blowcock and all storage tank valves are opened to ensure that all vaporized kerosene can be cleared from the process tank. After this is achieved, compressed carbon dioxide is blown through the system to purge both the tanks and the pipework of vaporized kerosene. This only takes a few minutes, after which the extraction fan in the condenser system is switched on and the vaporized kerosene is drawn into the condenser for reclamation. Here, it condenses into the separator as a mixture of kerosene and water. The kerosene will float on top of the water so that the water can be drawn off by opening an outlet valve at the lowest extremity of the separator, and thus leave the kerosene in the separator.

The extraction fan is kept running for about 2 h when the plates are purged of kerosene and any kerosene smell. During the whole of the extraction or fuming period, steam continues to pass through the heating coils of the process tank.

At the end of the fuming period, the steam supply is turned off and cooling water is passed through the coils until the temperature in the process tank falls to about 40 °C. When this temperature is reached, the cooling water in both the tank and the condenser is turned off, the tank is opened, and the plates are extracted with the extraction fan running continuously until the tank is empty. The full batch process takes about 4.5 h.

The Boliden continuous hot kerosene process is shown schematically in Fig. 2. It uses two hot kerosene baths. The first eliminates most of the absorbed water and is called the dewatering bath. The second completes the elimination of water and impregnates the active material with a heavy-fraction oil. Some operators of the Tudor batch system include a heavy-fraction oil in the kerosene for the same purpose, the creation of a moisture barrier between particles of the material. After passing through the dewatering and impregnation baths, the plates are force-dried to remove any kerosene, and then cooled. They then pass through an inert gas lock to be dispensed as dry-charged negatives.

Incorporated in the equipment are condensers and water/kerosene separators that follow much the same sequence of events as in the Tudor plant.

The plates are accommodated in small open crates that hold about 75 single automotive plates, or a smaller number of traction plates. The factor controlling the number of plates in a crate or carriage is the weight and thickness of the plates.

Thin automotive negatives of weight 130 to 150 g per plate will pass through the plant in 6 min. Thicker automotive plates of about 200 and 315 g take 7 and 9 min, respectively, to pass through the equipment. This gives standard equipment throughputs of 1500, 1285 and 1000 plates per hour for each of the weights quoted. Over a full working week, the outputs of both Tudor and Boliden plants are similar.

The removal of absorbed water from thick industrial plates is a slower process and this is reflected in a reduction in the maximum weight of plates processed per hour of about 35%.

The plant should be worked on a continuous 7 days-per-week basis for maximum efficiency. The dewatering and impregnation baths take about 8 h to heat up from cold. Where a continuous 7 days-per-week working is not acceptable, then arrangements need to be made to maintain the baths warm during idle periods so that the time spent in raising the kerosene to the working temperature does not unduly affect the attainment of the expected plant output. Considerable heat insulation is normally

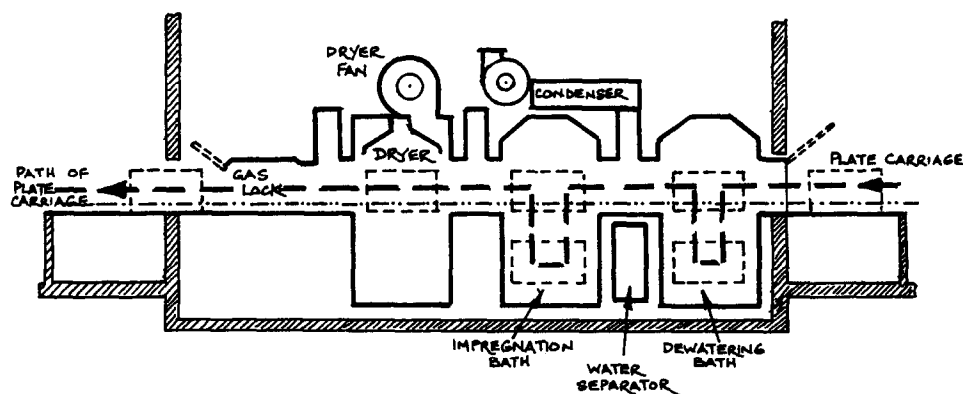


Fig. 2. Boliden hot kerosene continuous dry-charging plant.

provided around the baths so that 12 h after the heating has been cut off, there is sufficient retained heat to require only 30 min of heating to bring the baths into full working order.

The Tudor batch process, operating within a pressure vessel, has a lower usage of kerosene per ton of plate weight. Data quoted indicate a usage factor of as low as 3 l of kerosene per 2000 double plates, compared with 4 to 4.5 l on the Boliden plant for an equivalent loading. This factor has to be weighted against the heating-load costs, compressed carbon dioxide costs, etc., as well as manning and convenience.

Although hot kerosene alone will produce an excellent dry charge with freshly dried plates, i.e., as good as the best vacuum dried plates, it has limitations in extended storage under average conditions. To confer good stocking properties, it is customary to add to the kerosene a quantity of a mineral oil, usually to SAE standard. When the kerosene has been fumed off, the heavy-fraction oil remains as an extended film that covers the developed surface area of the material. The Tudor batch process uses ~0.3 l of SAE 90 oil with each batch of 2000 double automotive plates, i.e., about 75 mg per plate. Whilst this heavy oil acts as an effective moisture barrier to water vapour, it readily breaks down in the presence of sulfuric acid. Kerosenes vary in composition and there are some that contain a proportion of a heavy oil as a contaminant. If the amount of the contaminant oil is adequate and of the right fraction, a fully-acceptable storage life can be obtained without any further addition. This possibility has to be checked with the supplier before a decision to add heavy oil is made.

Too much heavy oil addition leads to a plate that can be stored for a greatly extended time under adverse conditions, but will not readily activate when the battery in which the plates are used is filled with electrolyte.

The main problem with hot kerosene dry-charging is the ease with which indiscipline can enter the operation. If the fuming time, after all the water has been expelled from the active material, is skimped there will be some kerosene left in the material. The plates will feel oily to the touch, will smell of kerosene, and a film of kerosene will float on the surface of the electrolyte after the battery has been filled. Whilst the presence of kerosene on the electrolyte does not adversely affect the battery performance, after a while, bituminous sealing compounds, if used to seal lids to cell compartments, are softened and become sticky. In contact with polystyrene vent plugs, kerosene acts as a strain-release agent and causes the moulding to stress craze and, in extreme cases, to crack.

The Boliden system originally used white spirit, but later changed to kerosene. No additive was used in the dewatering bath, but paraffin wax was added to the impregnation bath to provide a moisture inhibiting film over the surface of the particles of the active material.

Both systems used for hot-kerosene drying impart to the plates excellent long-term stocking qualities. Experience has shown that storage lives in excess of four years have regularly been achieved without a significant loss in low-rate capacity, and with automotive batteries, only a small loss in the cold-start performance.

There is evidence to indicate that, in the case of thick traction plates, there is good penetration of the oil into the active material but not always fully into the mass of the material. As a result, the low-rate capacity tends to fall below the rated values. Where traction cells are assembled with hot kerosene, dry-charged negatives, it is prudent to give the cells, after filling with electrolyte, a short initial charge of the order of 50% of rated capacity before they are put into service use. This input usually produces excellent results.

Drying in the presence of anti-oxidants

The process of dry charging seeks to set up conditions whereby atmospheric oxygen is denied access to the natural active material. About 40 years ago, it was thought that if the formed plates could be impregnated with a material which preferentially seized the available oxygen, then the plates could be dried in the open air. Impregnants, such as hydroquinone, cresol and phenol (organic inhibitors) and boric acid (inorganic inhibitor), have been successful. The former have not achieved, to any appreciable extent, commercial acceptance; on the other hand, boric acid has found application on the basis of good economic operation.

In the boric acid system, formed negative plates are taken from their protective water bath and loaded on to a carrier jig where, with a riffling action, the plates are spread on to notches that separate adjacent plates in a uniform manner. Where false lugs for formation connections are used, the operator knocks these off before allowing the carrier jigs to start the sequence of drying operations. The carrier jigs are then lifted on to a moving conveyor that allows the jigs to swing freely in a vertical plane. The jigs are first conveyed through a washing area where fine pressurized sprays of water impinge on the plates and remove the bulk of the formation electrolyte by continuous dilution. Following the washing operation, the carrier jig and plates dip into an impregnating tank that contains a solution of boric acid. Emerging from the impregnating tank, the plates then pass into an oven where the absorbed water of solution is evaporated to leave the inner structure of the material coated with boric acid. There is a risk that the boric acid may not have penetrated fully into the mass of the material. To remove this unwanted situation, the plates are passed into a second impregnating tank and then into a second drying oven from which they emerge as fully-protected and dry-charged plates.

The purity of the wash waters need not be too high, but highly chlorinated water should be avoided. The degree of dilution of the formation electrolyte after the washing stage should bring the residual H_2SO_4 to less than 35 ml per 100 kg of active-material weight. The temperature of the drying ovens is $\sim 120^\circ\text{C}$. This is sufficient to raise the temperature of the active material to around 75°C when evaporation of the absorbed water is relatively rapid.

The solubility of boric acid in water at normal temperatures is about 6.35 g/100 ml of cold water and the rate of solution is slow. The impregnating solution is best prepared by using hot water.

Good dry-charge results can be obtained with boric acid concentrations in the range 20 to 60 g/l. Long-term stocking characteristics are good and little degradation occurs in batteries where the only protection given to the cell elements has been that of normal vent plugs that have been screwed in place. Some criticism has been made that plates treated with boric acid require more initial soaking than plates that have been processed differently. Because of delayed wetting, Bode [1] suggests that soaking times of one to several hours should be employed.

Protection of dry-charge characteristics

Plates that have been dry charged by the superheated steam, vacuum and inert gas methods will deteriorate during storage unless a moisture inhibitor is incorporated within the active material. This inhibitor acts in the same way as the heavy oil film in the hot kerosene drying system. There are a number of hydrocarbons that can be

used, e.g., stearic and lanolinic acids. The former acid is the more common and has displaced to a great extent the earlier additions of paraffin wax that were used in these systems. Stearic acid is readily available in the form of a fatty material. This material can be produced in the form of granules that are incorporated easily into the dry ingredients of the paste mix before the water and sulfuric acid additions.

Stearic acid blends readily with the mill oxide and expanders during the paste mixing, but there is some difficulty in dispersing the relatively small amount of material evenly throughout the paste. As a result, it is necessary to rely on the heat of reaction during the paste mixing, the heat from the flash-drying oven immediately after pasting and the sustained vat temperature during formation to convert the stearic acid into a mobile liquid that will readily spread throughout the active material and uniformly coat the individual particles of the active material. Often there are areas within the plate material that are relatively rich in the inhibitor and others that are poor in inhibitor content. There is often a small loss of the inhibitor during formation, possibly from inhibitor-rich areas. This loss shows up as a scum that deposits on the sides of the formation vats.

A balance has to be achieved between the amount of the inhibitor required to give the order of long-term storage to suit the market, and the amount that does not affect the high-rate discharge performance unduly. This amount has to be determined by trials with the specified paste mix and the equipment available for dry charging. Nevertheless, a rough guide would be to limit the maximum addition of stearic acid to 0.05% by weight of mill oxide in the paste mixing.

Where paraffin wax is used in white spirit or kerosene, an acceptable level would be 6 g of paraffin wax to each litre of spirit. Where SAE 90 oil is used, the quantity required is ~0.3% by weight.

Rapid evaluation of dry-charge quality

There are a number of simple tests that can be applied on the shop floor to give an indication that the dry-charge process is broadly under control.

Acid spotting test

Sample dry-charged plates are spotted in a number of places with dilute sulfuric acid (1.28 sp. gr.). Initially, the drops remain as discrete globules on the surface of the plate and then slowly penetrate the active material. The protective film should be broken down by the electrolyte and a minimum and a maximum time to penetrate of 10 s and 10 min, respectively, are acceptable. This is a wide range and individual manufacturers may wish for a more precise procedure. This would involve tightening up the tolerances throughout the full process and may increase the cost of the process. The economics of any such move should be examined and weighted against the market benefits before a decision is taken.

Some firms consider the above test to be too coarse and run parallel with it a similar test using 0.1 M H₂SO₄. With this reduced concentration, the times to penetrate the plate surface are extended and typical minimum and maximum acceptance times are 30 min and 2 h, respectively.

Freedom from excess oil or wax inhibitors

There is always the risk that operators will not wait in the batch process for the plates to fume away all the excess kerosene or wax. The following examinations should be conducted:

(i) Look at the sample plates and compare with a standard plate that is known to be thoroughly free from oil itself and the smell of oil. If the colour of the plate is darker than the standard and there is a kerosene or white spirit smell, the batch of plates from which the sample was taken should be quarantined until further tests are made.

(ii) Place a sample of the suspect plates on a clean sheet of absorbent paper. Apply a hot iron (either a domestic iron or a large bit soldering iron) to the upper surface of the sample plate for not less than 1 min. At the end of this time, remove the plate and view the paper against the light. If there is a grease spot, it will indicate the presence of excess kerosene or white spirit in the sample plate. If the presence of free oil or spirit is confirmed, the plates should be reprocessed. Investigations should be made to ensure that the process procedures have been fully understood by the operators and that the correct practice will be followed in the batch process, and, in the continuous process, that the dryer temperatures and the fan speeds comply with the process specifications.

Effectiveness of dry charge

There are two recognized methods of assessing the effectiveness of the dry charge, namely: (i) by the amount of chemical reaction after immersion first in water and then in battery electrolyte, and (ii) by the engine-starting performance after filling with electrolyte.

In the former, one test is to immerse sample dry-charged plates in distilled water or demineralized water for exactly 30 min, after which the plates are maintained at 60 °C for not less than 16 h nor more than 24 h. The dried plates are then cut into small pieces of about 4 cm × 2 cm. 200 g of the cut pieces are placed in a Dewar flask in which, previously, 250 ml of dilute sulfuric acid (1.280 sp. gr.) had been placed. The temperature of the electrolyte was measured immediately before receiving the plate pieces and then after 10 min. The temperature rise is a measure of the chemical reaction that has occurred. If the rise exceeds 5 °C, the plates should be reformed and again dry-charged.

In the engine-starting test, an element is made as for a standard battery and uses separate take-off pillars. This element is assembled in an appropriate compartment of a container and the resulting test cell is filled with electrolyte. The cell is then stood for a specified time in order to allow the electrolyte to be absorbed into the active materials. This standing time, in the case where the selling policy is a 'fill and fit' one, needs to be relatively short. It is usual to adopt a 20-min soaking time. Where the sales outlet prepare batteries in advance, in anticipation of a demand, a longer soaking time can be accepted; this is usually 2 h. It is important to recognize which of these selling policies is relevant. In the case of the shorter time, the drying of the positives is critical otherwise the condition of 'blanking off' can adversely affect the results.

At the end of the soaking period, the battery or cell element is discharged at its declared high-rate discharge current at normal ambient temperature. The duration should not be less than that required for low-temperature engine starting, as laid down in the appropriate national or international standards.

Effectiveness of plate washing prior to dry charging

Where simple overflow tanks are used for plate washing, it is necessary to select sample plates from at least two levels in the tank for estimation of residual sulfuric acid. In systems where the plates pass through a water washing stage of an integrated

plate preparation and dry-charging plant, it is sufficient to remove a single plate from successive carriages.

The water from the wet plates is allowed to drip from the plates into a clean beaker. A quantity of 20 ml of the collected water is titrated with 0.01 M NaOH solution using phenolphthalein as an indicator. Not more than 1 ml of NaOH solution should be required for neutralization. If a greater volume is required, then the washing period should be extended.

Reference

- 1 H. Bode (ed.), *Lead-Acid Batteries*, Wiley, New York, 1977, p. 278.