ADVANCED MANUFACTURING PROCESSES FOR LOW COST GREENER LI-ION BATTERIES

NOVEL GRAPHITE MATERIALS FOR AQUEOUS COATING

Development and upscaling activities within the Greenlion Project

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Introduction

A lithium secondary cell consists of active components that allows the insertion and extraction of lithium-ions namely the positive (cathode) and the negative electrode (anode) providing the required energy density to the cell. The inactive components like the electrolyte, separators and the connectors do not actively contribute to the specific charge of the cell but lower the volumetric and gravimetric energy since they decrease the available volume for active materials. The electrolyte acts as an ion transfer medium through the electronically insulating separator and the connectors provide the contact to the external circuit.

The general process for the development and upscaling of a material typically goes via a few different steps which are schematically reported in Figure 1. A given material for a certain application is developed in the R&D stage in grams scale with laboratory equipment. At this stage, many experimental materials are produced and tested in the application conditions. The intention of this material screening is to identify the relevant material parameters that play a key role in the final application. The materials parameters, which in the case of an active material could be specific surface area, particle size distribution, density, etc..., are tentatively defined. Once the screening of the possible materials is completed the upscaling passes to the pilot stage. A few selected materials are produced in larger quantities (typically in the kg scale) on a pre-industrial plant. Minor tuning of the material parameters is still performed and a final selection should be done. The key target of the piloting stage is to correlate process parameters to the material parameters in order to maximize production capacity and keeping the desired material performance. During piloting several batches need to be produced in order to collect statistics on the material parameters and on their acceptable variation in order to define tentative material specifications. The last step in the upscaling process is the standardization of the material and the process. In this step the material is produced using industrial plants in industrial quantities (typically tons). The production plant is typically similar to the pilot plant however able to produce much larger quantities. The process parameters should be further optimized on the industrial plant in order to reproduce the material and to maximize production capacity. Statistics on production lots is collected and final specification for the material are defined.

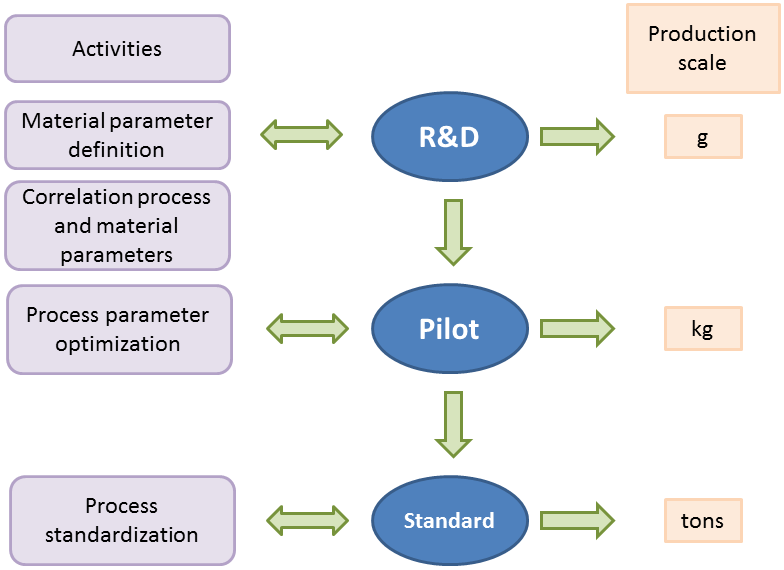

Figure 1 Scheme reporting the typical upscaling procedure

Development of novel graphite materials

Novel graphite materials were initially developed in the R&D stage on lab equipment; this stage was followed by two piloting phases performed on pre-industrial scale equipment. The first phase of piloting consisted in the qualification of the equipment and the target was reproducing and optimizing the material developed in the previous R&D stage. The second piloting phase was characterized by the optimization of the production capacity on the same equipment. Piloting phases are then followed by a production phase on industrial equipment. In this stage the material is standardized and the production capacity is at the moment one order of magnitude greater than the maximum capacity during piloting.

Key material parameters for the novel graphite materials have been identified during the first R&D phase as specific surface area and tap density, which evolution during the upscaling activities is reported in Figure 3. An additional parameter that is important for the application is the hydrophilicity of the graphite powder which allows the easy water processing. This one was taken as a go/no go criteria during the preliminary R&D activities. During the initial R&D activities the specific surface area was set to the range reported in Figure 3, however during the optimization in the piloting steps the acceptable range was changed since in the given working condition the new parameters were found to be more suitable for the application. Similar consideration for the tap density which initial acceptable range during the R&D phase was opened during piloting. The graphite powders were hydrophilic.

In addition to typical material parameters also application related parameters such as specific charge and irreversible charge have been optimized during upscaling. The specific charge for a graphite used in lithium ion batteries is the maximum charge can be reversible stored into the graphite via lithium intercalation. The irreversible capacity is defined as the amount of charge (indicated as percentage of the reversible capacity) is lost during the first lithiation/delithiation cycle of the graphite. Such charge is irreversibly lost during some irreversible reaction occurring in the first cycle that leads to the formation of the so called SEI (Solid Electrolyte Interface). The reversible capacity should be as high as possible and approaching the theoretical value of 372 mAh/g, and the irreversible capacity should be as low as possible. The measure of reversible and irreversible capacities needs the definition of a test procedure that should rigorously applied since the values are strongly linked to the test procedure. Bound electrodes with 6% PVDF, with a density of 1.2-1.3 g/cm^3 and a loading of 12-13mg/cm^2 were used. The first cycle is performed at 10mA/g, while subsequent cycles at 3C. These conditions are known to be not optimal and should be considered as a “worst case scenario”.

The evolution of the desired reversible capacity and irreversible capacity during the different phases of the material development are reported in figure 4. It can be seen that during the development phases the requirements became more stringent.

The aim of the upscaling process is, beside the optimization of the material parameters and the process parameter, the increasing production capacity which is reported in figure 5.

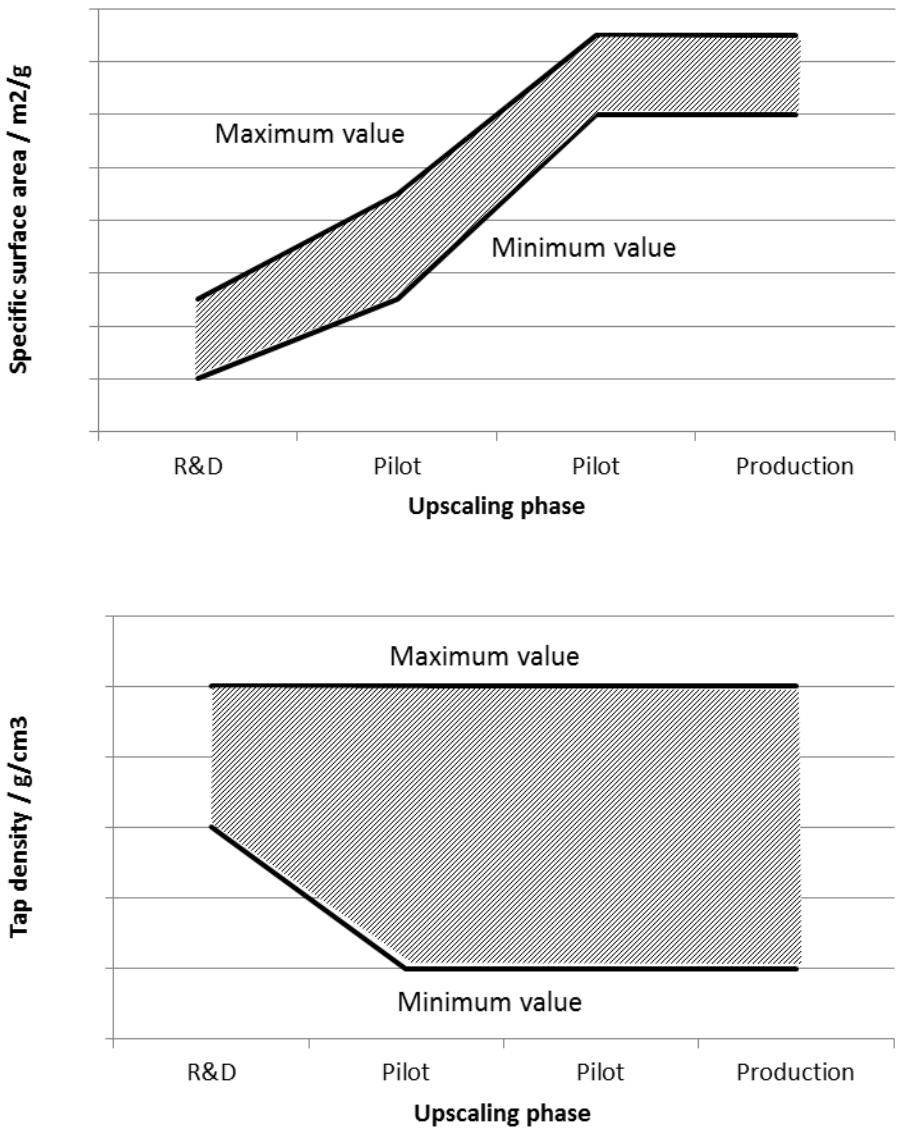

Figure 3 Evolution of Specific surface area and Tap density during upscaling

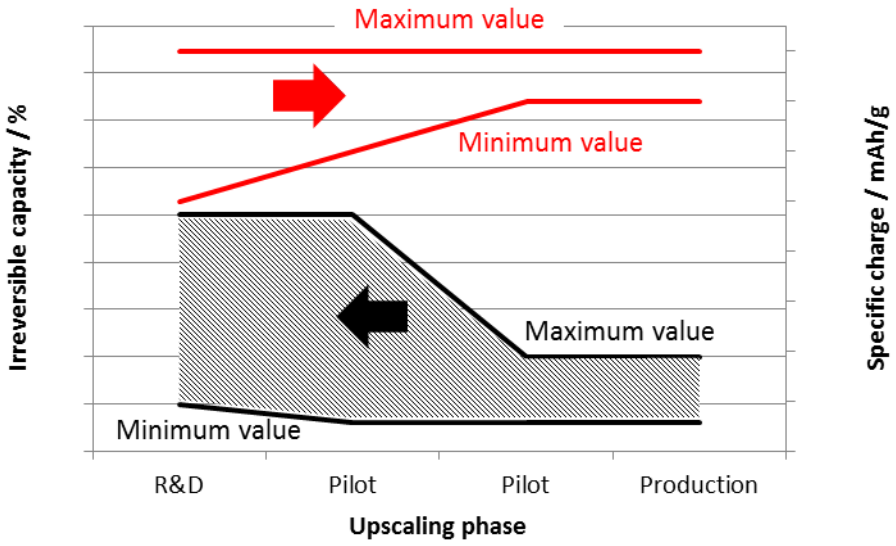

Figure 4 Evolution of the desired specific capacity and irreversible capacity during the upscaling activity

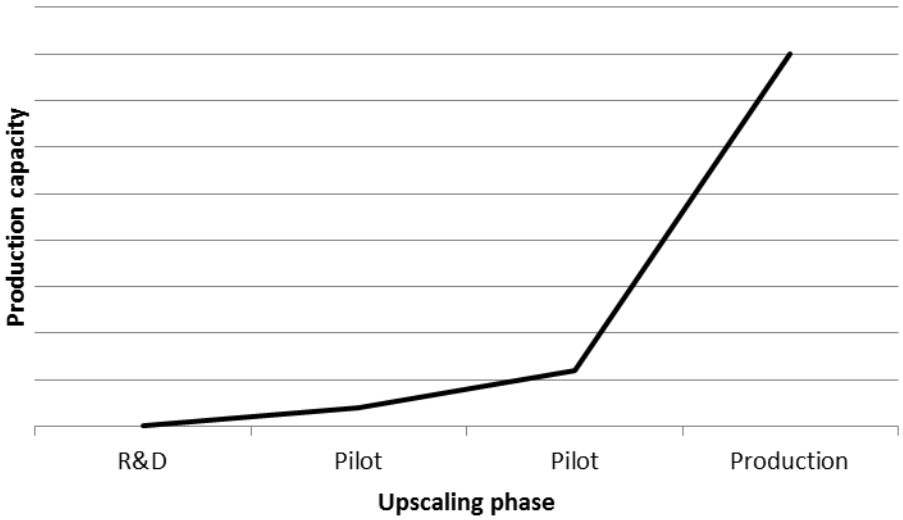

Figure 5 Production capacity during the various phases of the up-scaling process for novel graphites