Further progress on crystal growth on-line monitoring

C. Bouché and B. Gaillac

ITECA SOCADEI. France

Contact author: catherine.bouche@iteca.fr

Abstract

A good characterization of the main variables affecting a strike can lead to an improved operation of any type of pan. On-line monitoring of crystal growth performed by ITECA' new generation of microscopes provides a better knowledge of those variables, which are essential for achieving highest yield and cost effective production of homogeneous crystals. The paper describes and discusses digital microscope operation at different stages of the process (i.e. before seeding, at the seeding stage and during graining phase). A particular attention must be paid to the location of the microscope onto the pan: it has to be mounted in front of a representative area to provide good monitoring of the crystallization and valuable statistical information, especially in the first stages of the process. This new tool has become indispensable for creating standard strikes - the ultimate goal being to achieve full automation of the pan operation.

Keywords: ***crystal growth; batch pan; seeding; microscope; strike; statistical information; CV; MA, ITECA, online monitoring***

Introduction

There are many different parameters affecting a batch pan sugar boiling operation: some of them are fixed by the original design (type of pan or circulation inside it) while others are constantly evolving and interacting between each other (syrup purity, seed size and quality, steam supply, temperature, vacuum, concentration, level of super saturation, duration of each step, etc…). To get a massecuite of good quality, it is essential to establish the best possible sequence of operations involving these parameters and make sure the chosen strategy is correctly applied for every type of pan and for each strike.

Crystal growth on-line monitoring using ITECA MCC3000 microscope gives a very good knowledge of the sugar boiling process. The number of crystals, the Coefficient of Variation (CV) and the Mean Aperture (MA) real-time measurements provide the operator with the information necessary to ensure that the graining is focused, on schedule and moving toward the goal: a cost effective production of homogeneous crystals with the required size. In order to homogenize and stabilize the production regardless of the type of pan or of the operator, standard strikes can be set up while ensuring optimum operating points.

This paper describes how the pan microscope can play an important role at different stages of the process, especially when it is well positioned on the pan. Before seeding it checks the syrup quality and potentially detects contaminants or super coarse crystals that will limit the production of high quality crystals. At the seeding stage, it counts and measures the crystals to make sure the good volume with the correct size have entered the pan at the right time. During the graining phase, the CV and the MA are monitored to check normal crystal crop. Any non conformity (air bubbles, bad circulation, bad crystal size, etc…) can trigger alarms.

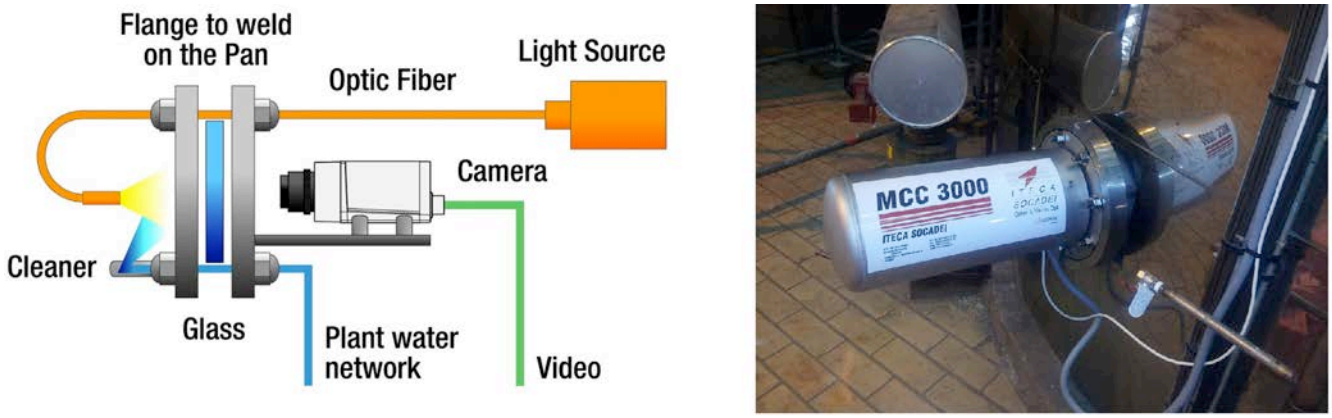
On the whole, every single data is registered for future analysis and complete traceability. On batch pans, different strikes can be compared at fixed intervals or at predefined key-points to easily follow the global trends of the measured data over time and closely run the pan.

Measurement principle

Crystal growth is analyzed using a pan microscope coupled to a high-resolution digital camera placed in front of a sight glass of the pan (figure 1).

A controlled LED light source illuminates the crystals moving

Figure 1. ITECA MCC3000 installed in front a sight glass on a batch pan



inside the pan behind the sight glass in front of the microscope and the high-resolution CCD camera. Very sharp images of the crystals are continuously sent to a computer in control room where dedicated software applies specific mathematical algorithms to each image and calculates, among other parameters, the Coefficient of Variation (CV) and the Mean aperture (MA) of the crystals in real-time. Adjustable thresholds on different variables are used to detect and trigger alarms on non-conformities.

Direct communication I/O must be set up between the microscope and the plant PLC to send information (Alarms, CV and MA values) to the plant supervision system but also receive information from the plant (Beginning of the process, seeding "top", drop of the pan, washing, etc…) that are necessary to correctly characterize the process.

The position of the microscope on the pan must be carefully chosen to ensure representative measurements. It has to be located below the syrup level, ideally at the calandria level in an area with good agitation and free from air bubbles (See left hand image taken at Nangis's plant – France, 2012).

At Imperial Sugar in Savannah – USA, the microscope was originally placed at the calandria level but because there were numerous air bubbles in this selected area, especially during the seeding phase, it was later moved underneath the pan (as shown in the middle panel in figure 2), where the agitation is even better. At this particular location, an air pressure cooling system was mounted onto the microscope housing to maintain a stable temperature below 55°C, essential for proper functioning of the electronic components.

In another plant in Germany, the air bubbles issue during seeding phase was addressed differently to avoid moving the welded flange from its original location. The software was upgraded to detect the air

Figure 2. Examples of positions of the ITECA MCC3000 on the pan

Good location – calandria level

Good location – underneath the pan

MCC too high above the seeding level

bubbles and take them out of the crystal size calculation.

Checking the syrup quality before seeding

Small contaminants detection

Impurities are first eliminated during the clarification step using screening, heating or liming techniques, but the syrup produced after evaporation always contains contaminants that will limit the production of high quality crystals.

Detecting particles from a few µm, the microscope can count and measure the contaminant sizes and characterize the syrup quality for each strike, checking that it will remain constant and within predefined limits, and potentially trigger alarms in case of non-conformities.

Contaminant particles of about 30 µm are highlighted in the figure 3.

Figure 3. Analysis of the contaminants present in the syrup

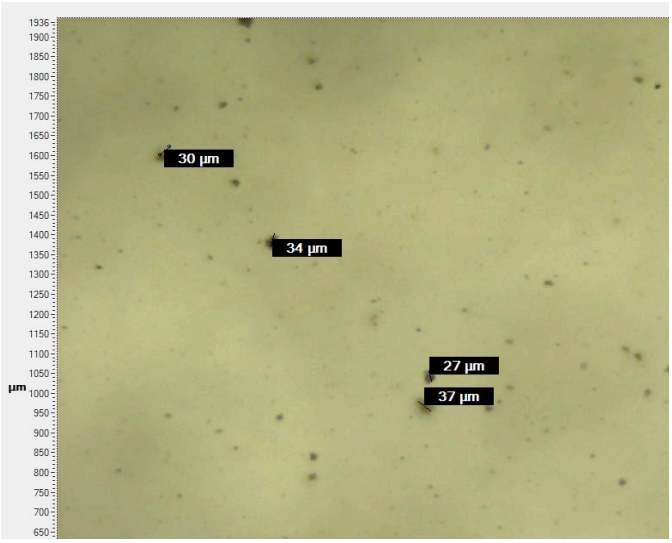
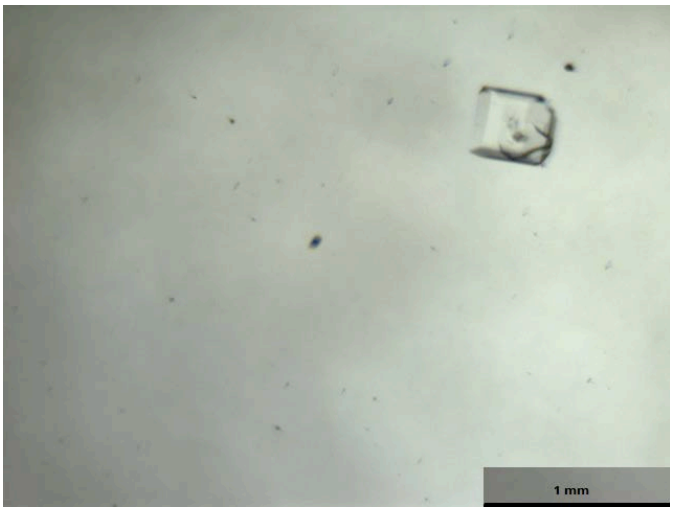


Figure 4. Detection of a 500 µm super coarse crystal



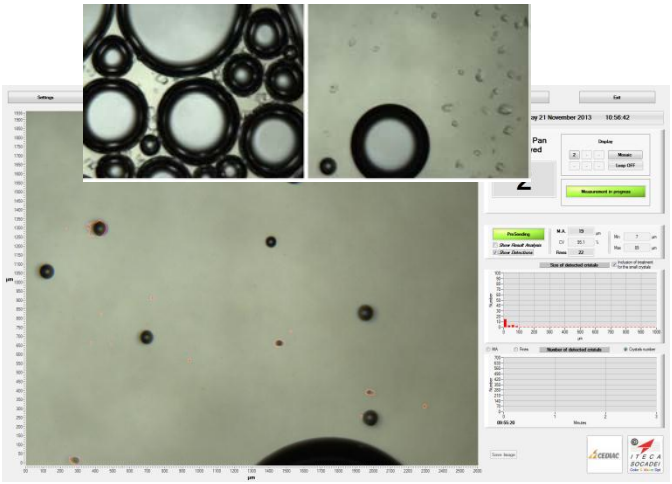
Super coarse detection

When the pan is not washed properly at the end of a strike, super coarse crystals remain inside and reappear in the syrup. Their presence induces difficulties in dispersing the seeds at the seeding stage and increases the risks of conglomerate formation. Big crystals will also continue growing during the entire process and contaminate the final massecuite quality

Air bubbles detection

In case of vacuum issue or when not enough antifoam is used, air bubbles can appear in the pan. Too many of them could for instance cool the syrup down and jeopardize the massecuite production.

Figure 5. Air bubbles detection



As their totally round shape can easily be dissociated from the crystals ones, the software automatically detects the air bubbles and disregard them for the crystal size and number calculation (figure 5). It still controls their number to allow the operator to react promptly and efficiently at this stage of the process.

Using the pan microscope at the seeding stage

Description

Much of the responsibility for good quality final crystals resides in the seed quality (Rogé and Mathlouthi, 2005): big crystals, free from fines particles and without conglomerates grains will preserve the homogeneity and the size of the crystals at the end of the crystallization process. In addition, the total amount of crystals produced at the end of a strike depends on the seed quantity. It is therefore essential to closely monitor the seeding stage in order to control final crystal amount and size.

The microscope can characterize these criteria in real time.

The measurement starts when receiving the “seeding” information from the plant PLC. It then constantly counts the number of particles present in the illuminated measurement area and displays the graph “number of crystals as a function of time” (red curve in Figure 6). The curve increases rapidly when the seeds enter the pan and it usually stabilizes after about three minutes, once all the crystals are spread evenly inside the pan. This is a good indicator of the seed preparation and injection procedure: it checks that the seeds enter the pan at the right time

Figure 6. Analysis of the seeds quantity and quality

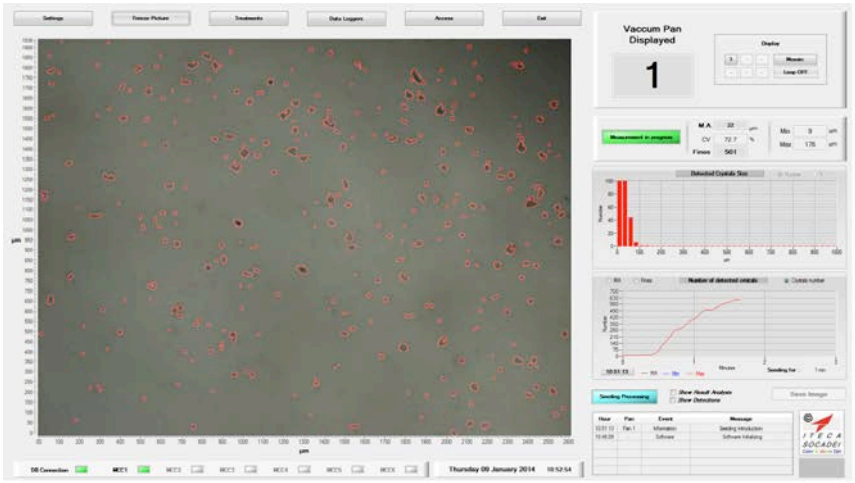


Figure 7. Bad agitation detection

and that the number of crystals remains stable for each strike.

Using adequate image treatments and optical algorithms, the software also measures the crystal sizes, displaying the distribution in size with the Coefficient of Variation (CV) and the Mean aperture (MA) in real-time. These indicators check that the seeds are homogeneous and with the requested sizes.

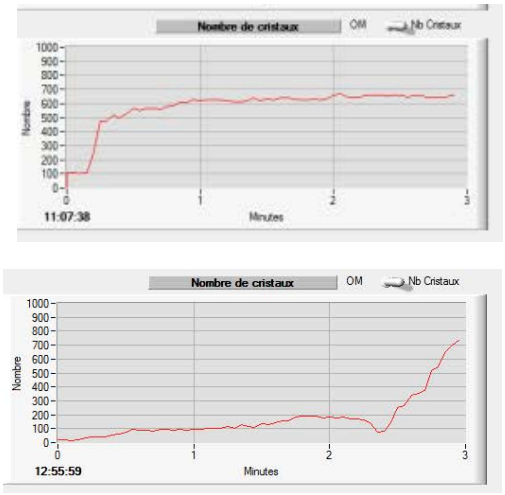
Bad agitation detection

During the seeding phase, the micro crystals are dispersed into the pan by mechanical agitation to get a homogeneous spatial distribution. In case of bad agitation, the crystals are not evenly distributed inside the pan (see figure 7): it leads to an increase of conglomerate formation and increases the risk of wasteful fines production.

Depending on the microscope position on the pan, the crystals might not be detected straight after the seeding top but might appear in the measurement area after a certain delay. As crystals have been growing in another area of the pan prior to arriving, they are bigger than expected (80 µm in the example shown in the previous figure).

The shape of the graphs “Number of crystals as a function of time” in Figure 8 is a good indicator of the agitation state. With a correct agitation, the number of crystals rapidly grows to finally stabilize when all crystals have been detected while in case of bad steering, the number of crystals stays very low for a certain delay before increasing.

Figure 8. Number of crystals as a function of time



Crystal growth measurement during graining phase

General measurement strategies

Different measurement strategies are used and applied according to the density of crystals inside the pan, the type and sizes of the crystals, which of course continuously change along the process (As shown in the crystal growth curve on figure 9) but also strongly depend upon the type of operating pan.

Up to four different strategies or specific algorithms can be applied (Each one using filters, morphology, pattern recognition or segmentation). The simplest strategy is used prior and at the seeding stage where crystals are little and perfectly separated one from the other. When crystals are getting bigger, they start touching one another and several algorithms must be applied in parallel and to each image to correctly characterize the crystals sizes.

As the crystals are moving in three dimensions inside the pan, and are not always presenting the same face in the two dimensions images of the measurement area, statistics must be made over several images to make sure the measured diameter is representative of the real average crystal size. The number of images taken for the statistics increases as the crystal sizes increase and as the number of crystals in each image decreases (i.e for a fixed measurement area of 2.6 mm x 2 mm, the number of crystal at seeding stage might be of 500, as at the end of the process it will go down to one or two crystals). As a consequence, the precision of the average size measurement being very high at the beginning of the process slowly diminishes along the strike.

Figure 9. Crystal growth curve – Laboratory measurement

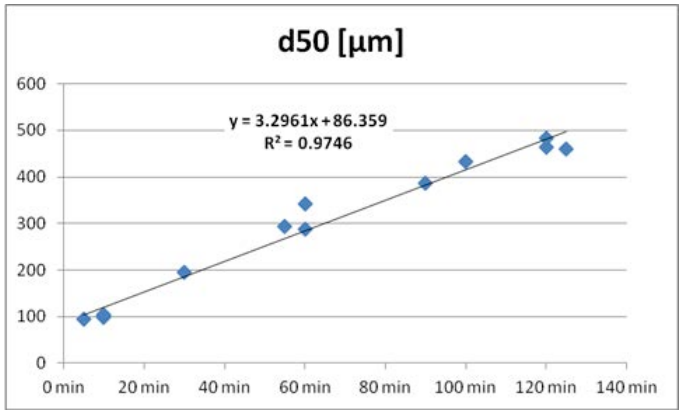


Figure 10. Crystal growth measurement evolution over 20 minutes of a strike

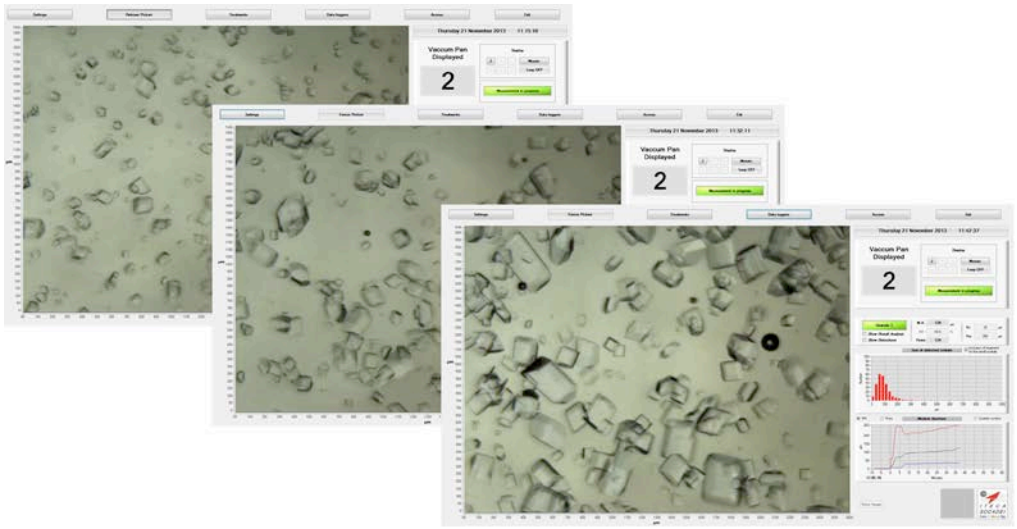
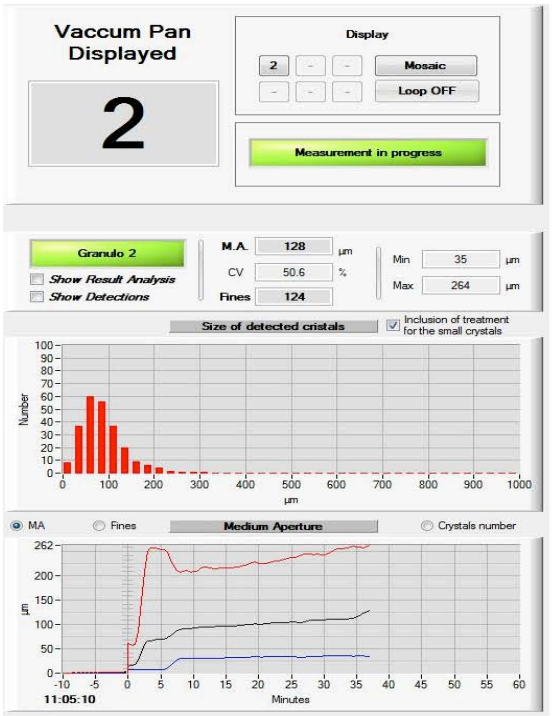


Figure 11. Zoom on the central right side of the main display



Crystal growth measurement

Every image being analyzed in real-time, the statistical information are calculated and displayed in real-time (figure 10).

The microscope follows the crystal growth until they are all agglomerated in front of the measuring area. The video of the crystals moving inside the pan is displayed on the left hand side of the screen and the operation and statistical information (Pan in operation, distribution in size, number of crystals, MA, CV, etc...) on the right hand side of the screen.

The microscope follows the crystal growth until they are all agglomerated in front of the measuring area. The video of the crystals moving inside the pan is displayed on the left hand side of the screen and the operation and statistical information (Pan in operation, distribution in size, number of crystals, MA, CV, etc...) on the right hand side of the screen.

The first table of figure 11 a displays the MA values, with the minimum and maximum measured crystal sizes in µm, and the CV plus the number of fines being calculated in percent. Each value is a sliding average over a specific number of images.

Below the first table, the distribution in size graph gives the number of crystals as a function of their respective size.

On the last display, the operator can choose to only monitor the number of fines, the number of crystals or the Mean aperture over time. The Mean Aperture (black curve) being the sliding average crystal size over 100 images is plotted with the maximum and minimum detected crystals (Red and blue curves). The red curve being the maximum value detected over the last 100 images gives a measurement of the presence of big crystals or conglomerates in the pan. The blue lower curve being the minimum detected crystal size is only interesting when compared to the two previous ones to monitor the CV. Indeed, the three slopes will stay parallel with a constant CV and deviate one from the other in case the dispersion increases.

Figure 12. Monitoring of the number of fines in percent

Figure 13. Key points along the strike

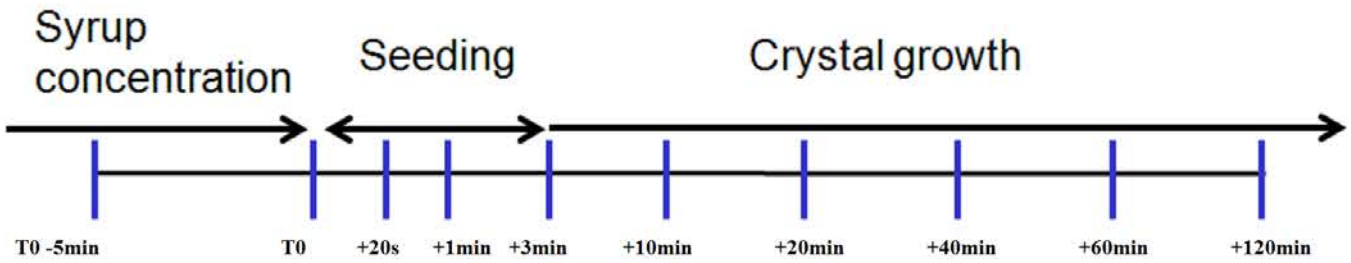
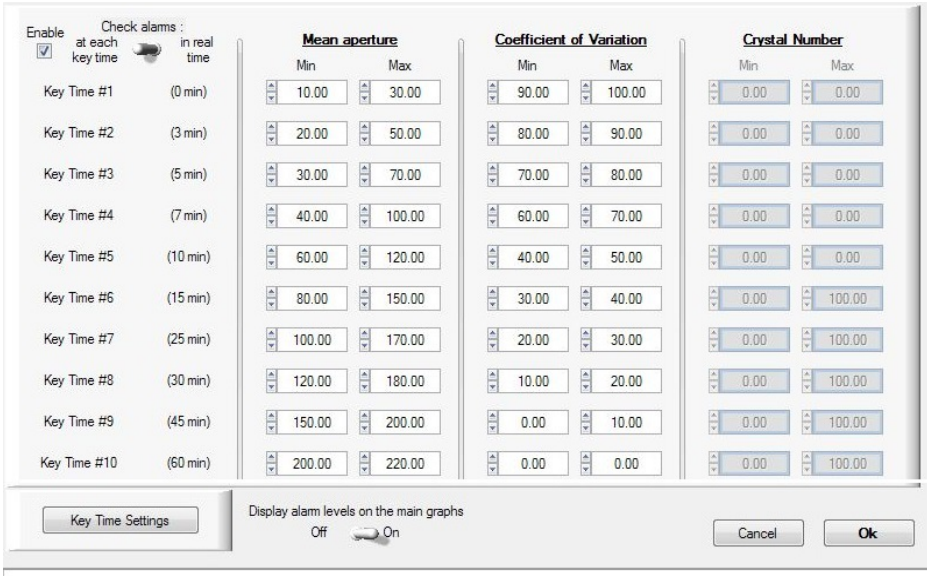
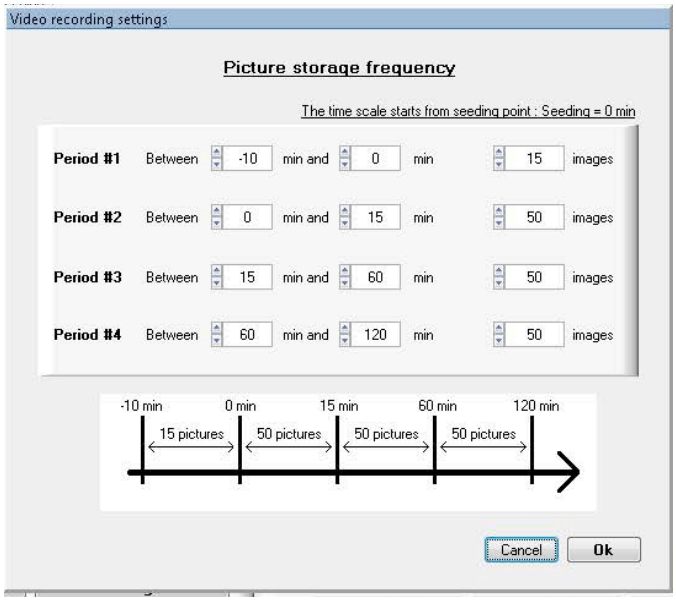


Figure 14. Key points configuration



Operational limits of the system are reached when all the crystals are agglomerated in front of the sight glass. They strongly depend upon the original crystal density inside the pan and the duration of the different steps of the process.

Figure 15. Time interval selection box



Fines detection

When the super saturation is not properly contained, it can lead to the spontaneous formation of crystals or false grains that will have a significant influence on the massecuite production (Libelle, 2007).

The software has a dedicated section that continuously monitors the presence of fines particles. In figure 12 the number of fines is plotted as a percentage of the overall crystal sizes.

The "fines" definition is easily adjusted for every pan (i.e it is the number of crystals with size lower than x µm; x being configurable by the operator).

Typical shape is shown in Figure 12 : the number of fines is optimum during the first minutes of the process because the crystals are still small. The proportion of fines decreases steadily as the crystals size rises before stabilizing to about 15% (The number of fines can never be zero due to the residual noise and because there is always a certain amount of fines remaining in the pan).

Powerful verification and comparison tools

Monitoring the ongoing strike at specific key points

One of the key features of the software is its ability to check the value of the measured variables at predefined key points and possibly trigger alarms when thresholds are exceeded (figure 13).

Ten key points are automatically registered on a standard basis but the operator can modify and adjust them for each strike if requested, completing the table in Figure 14.

The key points are placed on the requested time scale and for each one, minimum and maximum alarms thresholds are set up for the variables MA, CV and Crystal number. An overrun of any of these thresholds will trigger an alarm or a switch.

Additional parameters such as the number of contaminants, the number of conglomerates in the pan prior to seeding, or a sudden increase in the number of fines can be monitored as well, with two levels of alarms according to the importance of the situation.

Figure 16. Comparison of three different strikes, 21mn and 88mn after seeding

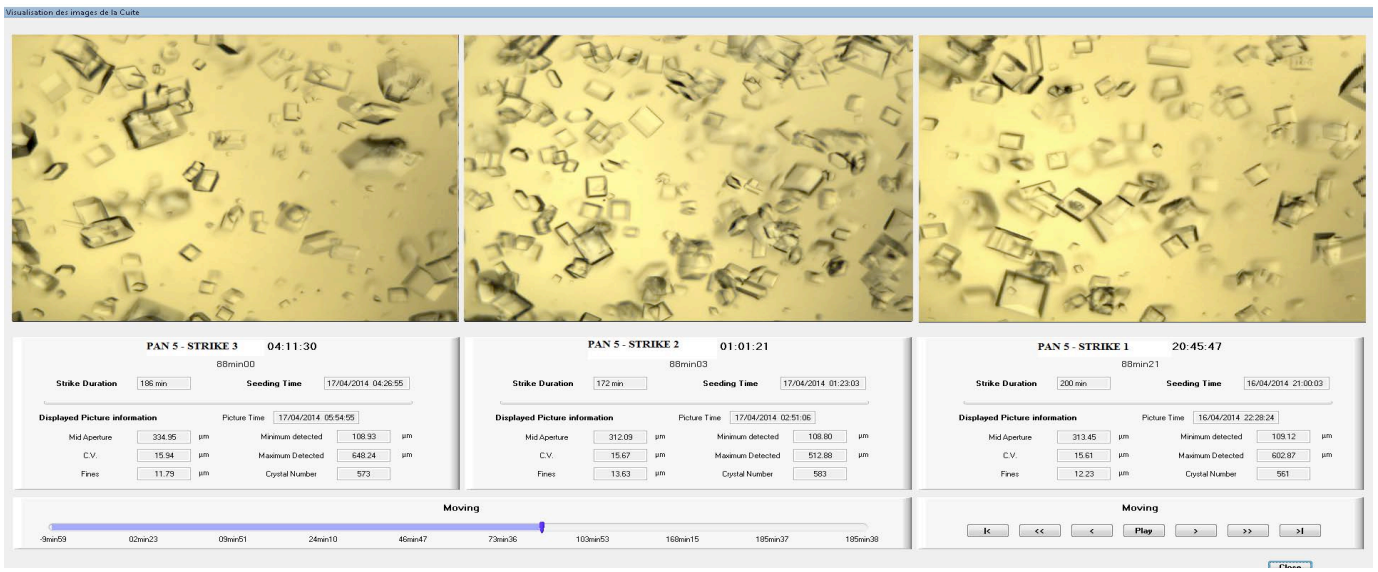
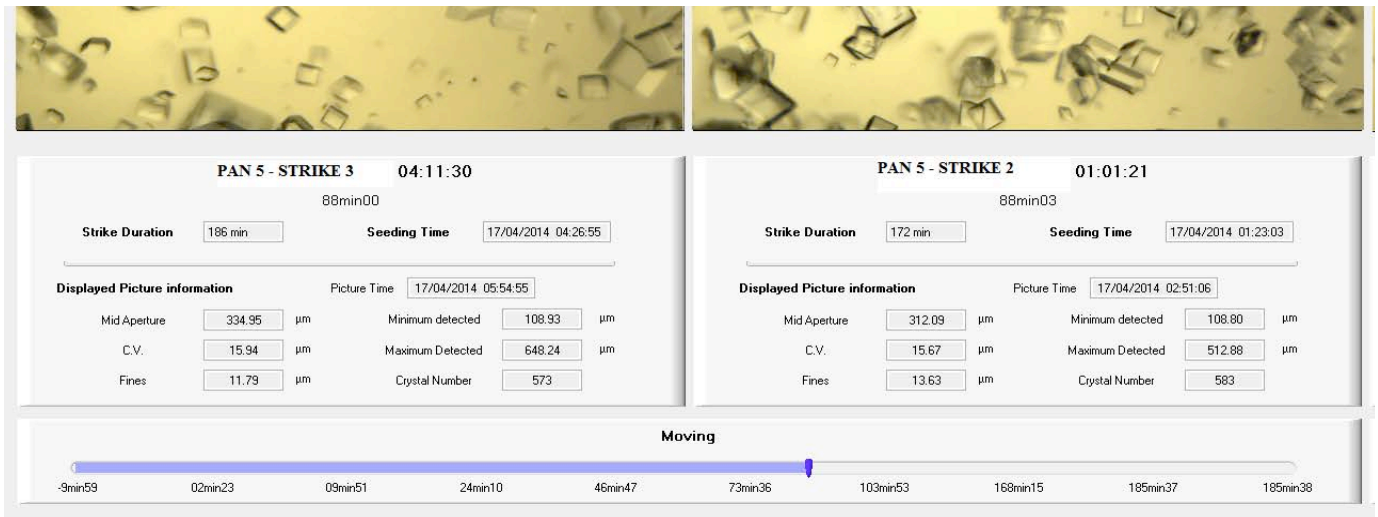


Figure 17. Zoom on the measured variables of the second and third strike 88 mn after seeding



Comparing various strikes between them or to a standard one

All the data being stored in a SQL database, it can be easily accessed and manipulated to be analyzed. Another key feature of the software is its ability to compare up to three strikes over a common time axis, starting at their own seeding point.

The videos of each strike being fully recorded from minus 10 mn before seeding until the end of the strike, the first step of the comparison consists in sampling the images within four preselected time intervals (cf figure 15) in order to streamline and accelerate the treatment of the selected images.

The user can choose a specific number of images to be used in each interval: a larger number of images for a close monitoring in a certain time interval; a lower number of images when less precision is needed in another time interval.

The videos of the three strikes can then be run in parallel and compared over the same time scale starting ten minutes before each seeding points until the end of the process (figure 16).

The videos are either played in a single run over the entire length of the strikes or the cursor can be moved anywhere along the time axis to visualize the three videos at the same specific times, each strike being synchronized at their seeding point. The indicators are displayed in parallel and compared with each other.

The images themselves give a lot of information as it is easy to check whether the crystals are similar and distributed evenly inside the pan at the same time for each strike, but the value of each variable is also displayed for more precise monitoring and comparison (figure 17).

The MA, the crystal number or the number of fines can be compared at specific times.

The information makes even more sense when compared to the pan operation flow sheet as any change in the process is tracked and its impact directly measured on the crystal growth.

Another major use of this feature is to set a standard strike and compare it to the other strikes to make sure that the pan operation remains stable and within predefined limits, which will ensure a stable and good quality massecuite production.

Conclusion

This new generation of digital pan microscope will soon become indispensable in sugar factories and refineries. Considerable benefits have already been demonstrated in optimizing the crystallization process. The ability of creating standard strikes now helps stabilizing the massecuite production irrespective of the type of pan or of the operator in charge of the operation, the ultimate goal being to achieve full automation of the pan operation.

References

Barbara Rogé, Mohamed Mathlouthi (2005) Qualité de la semence, contrôle de l'ensemencement et de la qualité des cristaux de sucre. Proceedings of the AVH Association – 12th Symposium.

Teddy Jeannick Libelle (2007) Modèles de connaissance de la cristallisation de troisième jet en sucrerie de cannes – Expérimentations et simulations. PhD Thesis presented on September the 28th, 2007 by– Université de la Réunion – UFR de Sciences et Technologies