Technology for Productivity

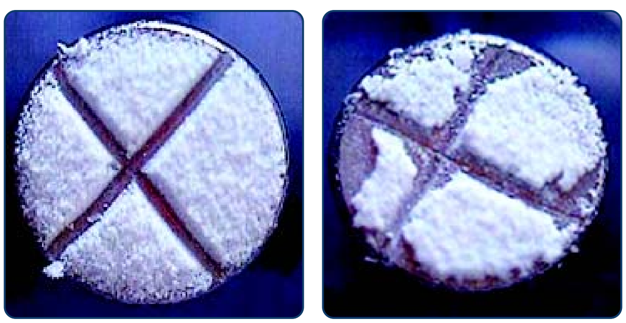
# Application Note

## Multi-Inlet Cirrus™ to Monitor Hydrogen Reduction Process

Note #05/07

## PROBLEM

A major manufacturer of TV and Computer CRT monitors experienced an outbreak of mis-processed components caused by equipment failure. The problem occurred in a thermal reduction process used in the production of nickel caps for thermionic emission cathodes, which are used in electron gun assemblies. The cause of the problem was quickly isolated to contamination of the high purity hydrogen supply to the reduction vessel with nitrogen, causing nitrides to form on the cap surface. The nitrides interfered with the critical interface between the cap nickel and the emission determining spray layer, causing failures at pre-delivery testing of the cathodes. The equipment was modified and short term control measures were put in place to ensure detection if the problem reappeared. However, the control measures required expensive offline scientific analysis using LA-ICPMS to be performed after every reduction cycle on a sample of caps, and so a more cost effective real-time solution was desired without an increase in risk to the component quality.



*Cathode cap with no Nitride layer, showing good spray layer adhesion (left); Cathode cap with Nitride present, showing poor spray layer adhesion (right)*

## BACKGROUND

The nickel caps are placed in a reduction vessel that is connected to one of two gases. The first gas introduced is a purge gas consisting of 95% Nitrogen and 5% Hydrogen. The second is 100% Hydrogen. After the reduction vessel has been loaded, it is purged at room temperature with the $N_2/H_2$ mixture for 5 minutes. The gas is then switched to Hydrogen for 15 minutes. The rate of gas flow is such that the gas escapes through the lid of the reduction vessel. In the case of Hydrogen, the gas is lit to burn off the excess. After 15 minutes, the reduction vessel is placed in an oven at 950°C for 30 minutes. The vessel is then left to cool for 15 minutes with a continued flow of hydrogen, before switching to the purge gas for 75 minutes.

In essence, this is a manual procedure with the operator being responsible for timing each stage of the process as well as the physical transfer of the vessel. Nitrides can form if the cathode is exposed to nitrogen when at a temperature greater than 400°C. This may occur if the process gases become contaminated, if the operator switches over to the purge gas prematurely, or from the purge gas as the lines converge before entering the pot.

## SOLUTION

The application required the simultaneous monitoring of two reduction vessels and therefore a Cirrus with a Multi-Stream Inlet was identified as the optimum solution. The high levels of Hydrogen encountered during certain phases of the process had to be considered in relation to the configuration of the Cirrus pumping system. It was decided, therefore, that the best approach would be to use two external rotary pumps, one to back the turbo and the other to pump the inlet bypass "Tee" assembly. The capillary bypass is used to maintain a constant flow of the ambient pressure gas sampled through the inlet, thereby maximizing the response time of the Cirrus to changes in the gas mixture. The capillary inlet then interfaces with the multi-stream inlet (rotary valve) as shown in Figure 1.

As the system was to be used in a production environment, the information provided to the operator has to be as straight forward as possible. It was decided, therefore, that the user interface should consist of two lights on each reduction assembly plus a

## SOLUTION (CONT'D)

system warning light on the Cirrus analysis system where:

- Green indicated everything was satisfactory on the reduction assembly being monitored
- Red indicated the Cirrus had detected an excessive nitrogen level on that particular reduction vessel
- Blue indicated that the Cirrus was not available for monitoring

The operation of the Cirrus is completely automated - indeed, the computer is locked away so the operator does not have access to it. Figures 2 and 3 provide an overview of the analog and digital I/O and other interfaces used to achieve this level of automation.

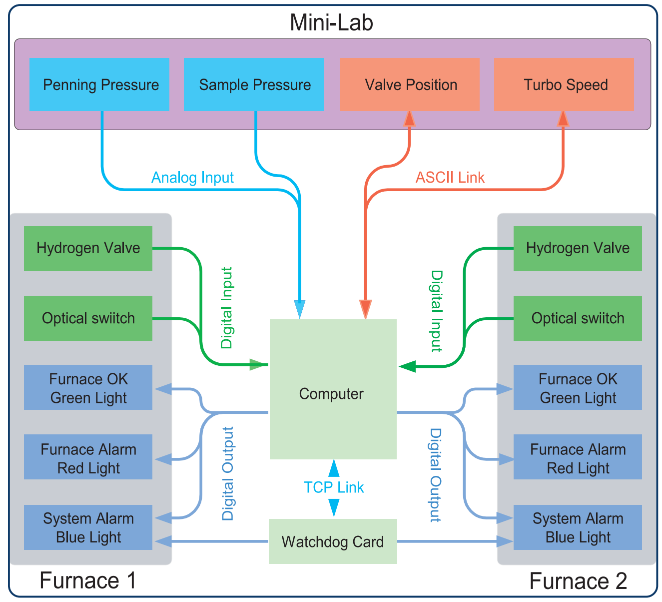



*Figure 2 - Cirrus™ analog and digital I/O schematic diagram*


*Figure 1: Cirrus™ with a Multi-Stream Inlet*

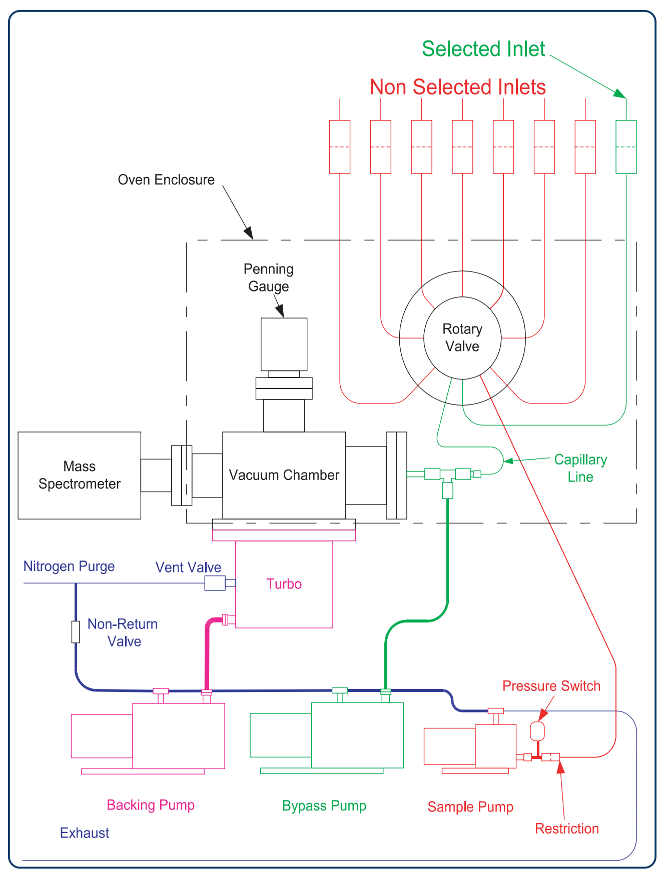



*Figure 3 - Schematic Diagram*

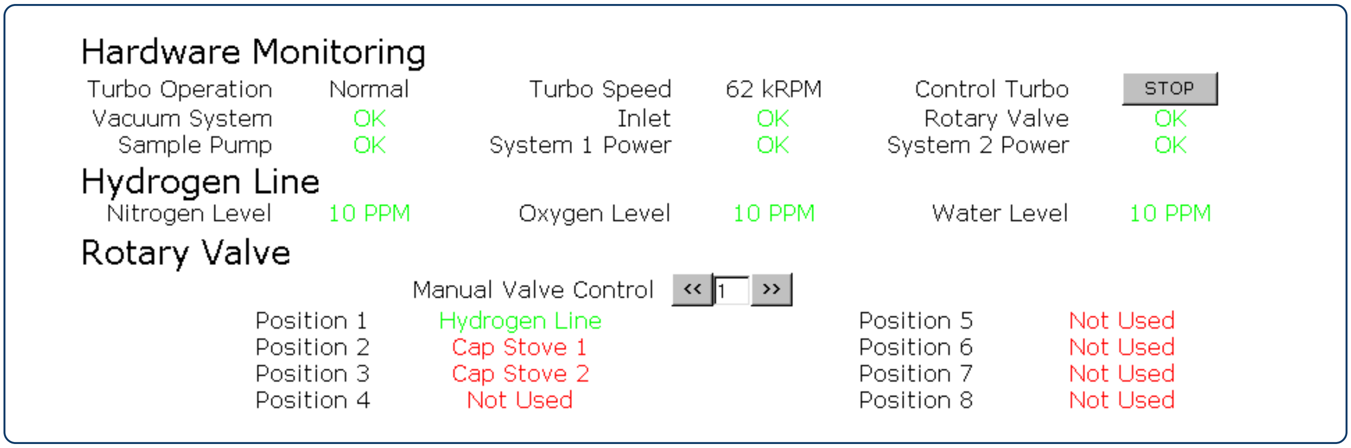



*Figure 4: Initial Start-Up Display*

# SOLUTION (CONT'D)

## PC based control platform

The Cirrus functions are controlled using Process Eye™ Professional, a 32-bit recipe-based application running a series of interlinked recipes. The computer is equipped with a watchdog card that would automatically re-boot the PC in the event of a failure. In addition to the inputs from the reduction ovens (hydrogen valve status and optical switch position), the Penning gauge pressure was also monitored as an analog input along with the output of the pressure switch. This is done to detect line blockages and/or pump failure so that any errors are detected immediately to avoid false readings. On initial start-up, the operator is presented with the display shown above (in Figure 4).

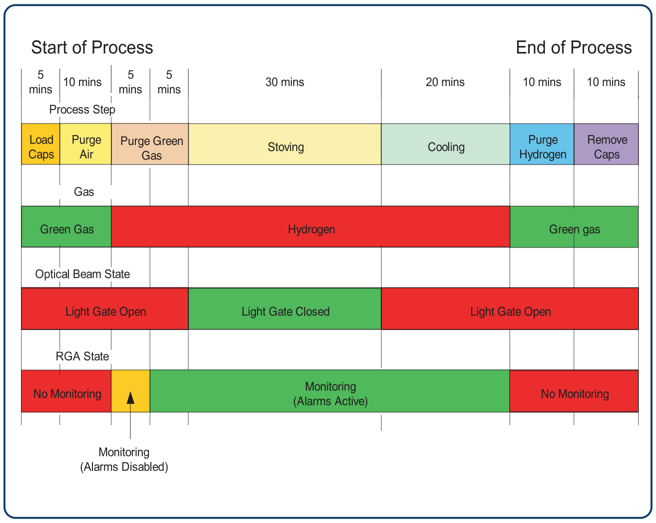
Communication with the Cirrus pumping system is with ASCII commands via an RS232 communications. Some examples of typical commands are:

- When the turbo operating parameters are within normal limits, the "Vacuum System" alarm state is "OK."
- If the pressure measured by the Penning gauge is too low, an inlet alarm is generated to indicate that the inlet line is possibly blocked, otherwise the "Inlet Alarm" state is "OK."
- On initial start-up, the system cycles the valve to each available position. On receiving confirmation that each position has been achieved, the "Rotary Valve" alarm state is "OK." If during normal operation the valve position does not achieve the requested position within a certain time limit, then the "Rotary Valve" alarm state will be set to "FAIL."
- The pressure on the inlet of the sample pump is received via a 0-10V analog input signal. If the pressure is below a certain limit, then the "Sample Pump" alarm state is "OK." If the pressure is too high, indicating a pump failure, then the state will change to "FAIL."
- If any of the alarms above go to "FAIL," the system alarm condition will be set which will light the blue alarm light on the Cirrus.
- If the 24V power supply to each furnace is "OK," then the "System 1 Power" and "System 2 Power" alarm states will be "OK." If either power supply fails, then a red alarm light will be illuminated on the relevant furnace.

When the system is in idle mode and neither furnace is being used, the Cirrus monitors the Hydrogen supply line to check the levels of Nitrogen, Oxygen and Water contaminants. These levels are displayed on the status page; in green, if below alarm levels and in red if above the user-defined limit. Should the nitrogen level exceed the alarm limit then the red alarm light will be illuminated on both furnaces. If all alarm conditions are "OK" then the green status light will be illuminated on both furnaces.

## SOLUTION (CONT'D)

The process sequence typically run in this application is:

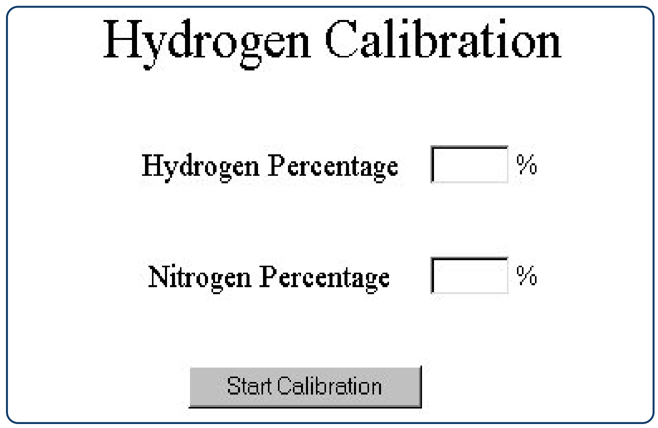



Once an operator has completed the initial nitrogen purge on a batch and switches to hydrogen, this functions as the trigger to start the process monitor recipe. The valve position is selected to monitor gas from that furnace. If both furnaces are in operation then 5 scans are taken from each furnace in turn. The point at which the operator switches back to purge gas marks the end of the run.

Factors critical to this process include the time that the hydrogen is on before the reduction vessel is placed in the furnace, the concentration of nitrogen contaminant in the hydrogen while the vessel is in the furnace and the length of time the hydrogen flow is maintained after the vessel has been taken out of the furnace. At the end of the run, the software generates a report and prints a hard copy.

### Calibration

In order to calibrate the system, a calibration gas was connected to the multi-inlet valve, position #4. The calibration gas was a mixture of low levels of nitrogen in a balance of hydrogen and originates from a separate, traceable bottled supply. The sensitivity for nitrogen was assumed to be 1 and the sensitivity was calibrated using air with a nitrogen level of 78%. A recipe was then created to calibrate for hydrogen sensitivity. This allowed the user to enter the concentrations of hydrogen and nitrogen in the calibration mix as shown below:

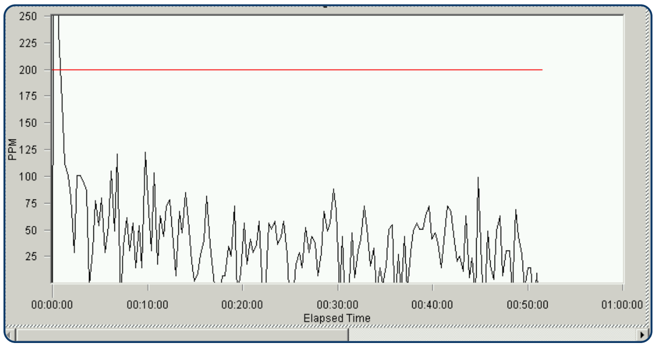



A sensitivity factor for hydrogen was then calculated and this value was stored as a reference and used for all recipes.

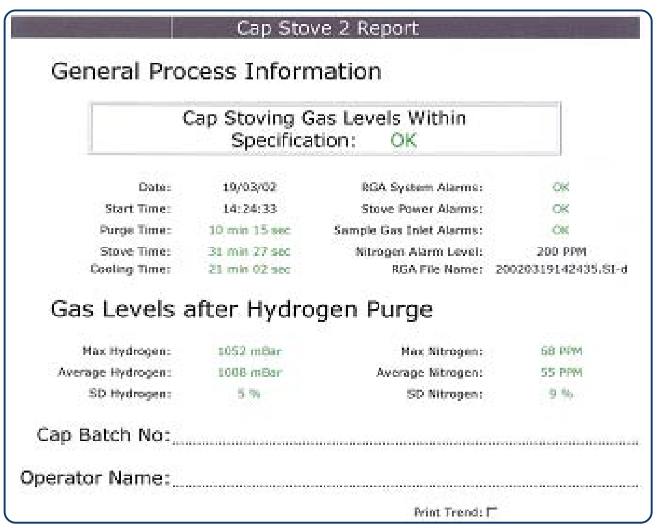
# SOLUTION (CONT'D)

## Results

A typical composition versus time trend of the nitrogen level during a process is seen below:




The nitrogen level drops quite quickly during the initial phase of the process. The alarm level is set at 200ppm but is only active after 5 minutes has elapsed. This delay is used to eliminate unnecessary alarms. The operator is not presented with this view, however, a summary report is updated during the run and then printed out at the end as follows:



Cap Stove 2 Report

General Process Information

Cap Stoving Gas Levels Within Specification: OK

| | | | |
|---|---|---|---|
| Date: | 19/03/02 | RGA System Alarms: | OK |
| Start Time: | 14:24:33 | Stove Power Alarms: | OK |
| Purge Time: | 10 min 15 sec | Sample Gas Inlet Alarms: | OK |
| Stove Time: | 31 min 27 sec | Nitrogen Alarm Level: | 200 PPM |
| Cooling Time: | 21 min 02 sec | RGA File Name: | 20020319142435.SI-d |

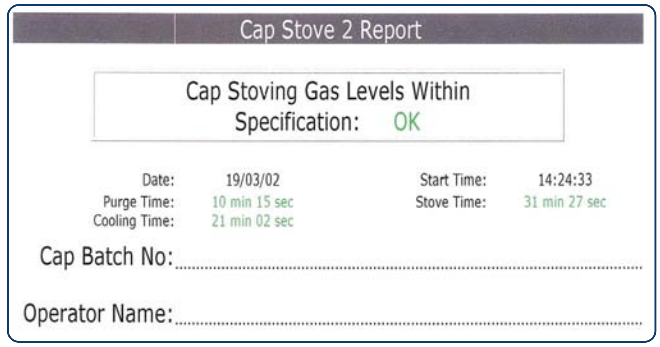
Gas Levels after Hydrogen Purge

| | | | |
|---|---|---|---|
| Max Hydrogen: | 1052 mBar | Max Nitrogen: | 68 PPM |
| Average Hydrogen: | 1008 mBar | Average Nitrogen: | 55 PPM |
| SD Hydrogen: | 5 % | SD Nitrogen: | 9 % |

Cap Batch No:.........................................

Operator Name:.........................................

Print Trend: ☐



Cap Stove 2 Report

Cap Stoving Gas Levels Within Specification: OK

| | | | |
|---|---|---|---|
| Date: | 19/03/02 | Start Time: | 14:24:33 |
| Purge Time: | 10 min 15 sec | Stove Time: | 31 min 27 sec |
| Cooling Time: | 21 min 02 sec | | |

Cap Batch No:.........................................

Operator Name:.........................................

The lower part of the report is sent with the caps while the top half is filed for quality assurance purposes. If any of the parameters are outside the allowed range, they appear red on the printout.

## BENEFITS

The customer has observed significant benefits in several key areas:

- Significantly improved process reproducibility and has virtually eliminated batch-to-batch variability
- Eliminated undetected operator induced process variations
  - Aided in operator training issues by identifying and quantifying operator errors, by operator, over time
  - Allowed for additional process optimizations to occur through utilization of extra data acquisition capabilities and PC control of the Cirrus
- Eliminated the expense of routine testing of the components, resulting in a savings of more than **$120,000 annually**
- Reduced the component lead time through the use of in-line real-time detection

Utilizing the Cirrus with its powerful Process Eye Professional software has led to further use of process monitors as a diagnostic and process optimization tool. This customer also purchased process monitors powered by Process Eye Professional in their vacuum sputtering areas to monitor the vacuum quality and to detect and quantify machine and operator variability.

For further information, call your local MKS Sales Engineer or contact the MKS Applications Engineering Group at 800-227-8766.



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