



SuperSystems
incorporated

OPERATIONS MANUAL



Basic Nitriding Sampling System

Hydrogen Analyzer with calculated %DA, %NH₃, and Kn values)

P/N 13537

Please read, understand, and follow these instructions before operating this equipment. Super Systems, Inc. is not responsible for damages incurred due to a failure to comply with these instructions. If at any time there are questions regarding the proper use of this analyzer, please contact us at (800) 666-4330 for assistance.

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Specifications:

- Power Requirements: 100-240 VAC
- Current Draw: Max. 0.2 Amps
- Sensor Technology: Thermal Conductivity
- User Interface: 3.5" Color QVGA TFT LCD Touch Screen
- Measurement Range: 0-100% H₂
- Hydrogen Measurement Accuracy: +/- 0.01%
- Hydrogen Measurement Repeatability: +/- 0.01%
- Hydrogen Measurement Resolution: 0.01%
- Analog Outputs: Two Isolated 4-20mA (User Configurable)
- Analog Output Resolution: 0.005mA
- Analog Output Accuracy: +/- 0.01% of Range
- Analog Output Linearity: +/- 0.01%
- Analog Output Load Resistance: Minimum 0 Ohm, Maximum 500 Ohm
- Digital Communications: RS485 Modbus, Ethernet
- Enclosure Size (Without Filter): Approx. 13"L x 13"W x 6"D / 330mmL x 330mmW x 142mmD
- Enclosure Weight: 22.5 lbs / 10.2 kg.
- Enclosure Ventilation: Continuous Purge Fan, Dual Vents
- Recommended Flow Rate: 1.5 to 2.0 SCFH / 0.71 to 0.94 lpm
- Process Gas Fittings: Stainless Steel Compression for ¼" OD Tubing
- Calibration Gas Fitting: 1/8" Barb (can be removed for 1/8" Female NPT Port)
- Operating Environment: 10-90 %RH (Non Condensing)
- Operating Temperature: 32 to 122°F / 0 to 50°C
- Sample Gas Temperature: 32 to 158°F / 0 to 70°C

Basic Description:

This instrument uses the measurement of Hydrogen to display % Hydrogen (H₂), % Dissociation (DA), and % Ammonia (NH₃). When the flow rates of Nitrogen, Ammonia, and Dissociated Ammonia are manually entered, the instrument can also calculate Nitriding potential (Kn).

Electrical Connections / Terminal Assignments:

Wire Number	Function
1000	AC Line Power (100-240VAC)
1002	AC Neutral Power
Ground	AC Ground
1091	Analog Output Common (+)
1101	Analog Output #1 (-)
1091	Analog Output Common (+)
1111	Analog Output #2 (-)
1121	RS485 (-)
1131	RS 485 (+)

Getting Started:

Please refer to the enclosed drawing for instructions regarding the proper electrical and mechanical installation of this instrument. The flow meter on the door of the enclosure should be adjusted to maintain 1.5 SCFH of process gas flow.

Default Settings:

When the instrument is turned on, it will display % H₂ on the screen. To display additional values (% DA, % NH₃, or Kn), see the "Instrument Setup" screen. The first 4-20mA output will be set up to retransmit the %H₂ value scaled for 0 to 100%, and the second output will be set up for %DA also scaled for 0-100%. Any changes to these default parameters will be stored so they will not need to be re-entered when the power is shut off to the instrument

Modifying the Default Settings:

To access the operational and setup parameters, press the "menu" button at the lower left section of the screen. This will allow you to select only two options, Exit Program and Instrument Information. To prevent unwanted modification to the operation of the instrument, these are the only options available unless the user logs in. To access the other menu options, press the "Login" button and enter "2". This will give the operator access to the setup and operational parameters. Each individual menu option is described in detail below:

Description of Menu Items:

Exit Program

The touch screen display is constantly writing data to its flash card for storage, and it is important that the instrument not be shut down during this process. Before removing power from the enclosure, select "Exit Program". It will ask if you are sure that you want to shut down the interface. By answering yes, it closes the operating program in an orderly manner. Once the standard Microsoft Windows screen appears it is safe to remove power from the instrument. If this procedure

is not followed, there is a chance that there could be an error writing to the flash card that could cause problems with the operation of the instrument.

Instrument Information

This screen provides information on any applicable revision levels and serial numbers. It also shows if the instrument is logging data. There are no functions that can be performed on this screen; it is for informational purposes only.

Communication Setup

The communication methods shown on this screen are for display only and cannot be modified. The baud rates can be adjusted but they have been optimized for this instrument and modification is not recommended.

Instrument Configuration

This instrument is capable of displaying four different parameters. These include:

- Percent Hydrogen (H₂)
- Percent Dissociation (DA)
- Percent Ammonia (NH₃)
- Nitriding Potential (Kn)

The Percent Hydrogen is displayed on the main screen at all times. One additional parameter can be displayed along with Hydrogen by highlighting it and pressing the "OK" button. A clearer description of each of these parameters can be found in Appendix "A", Gas Nitriding Technical paper.

Percent Hydrogen

The percent hydrogen is the amount of Hydrogen that is being detected by the thermal conductivity sensor inside the instrument. There are no additional calculations being performed to this value.

Percent Dissociation

Dissociation is derived from the amount of Hydrogen in the sample gas.

Percent Ammonia

The amount of Ammonia can also be inferred from the Hydrogen value.

Nitriding Potential

The accurate calculation of Nitriding potential requires the flow rates of other gases that are being introduced into the process. These flow rates are measured in SCFH (Standard Cubic Feet per Hour). Kn can only be calculated by entering the flow of Nitrogen (N₂), Ammonia (NH₃) and Dissociated Ammonia (DA). These values are entered at the bottom of the main screen when Kn is selected.

Output Configuration

There are two 4-20mA outputs that can be configured for any of the four parameters. For each input, the operator can select the Source (H₂, DA, NH₃, Kn), the zero value (the value to be represented by 4mA) and the span value (the value to be represented by 20mA).

- Output 1 can be measured from Terminal #1091(+) and 1101 (-).
- Output 2 can be measured from Terminal #1091(+) and 1111 (-).

Output Calibration

Accurate retransmission of the selected parameters can only be obtained through calibrating both of the analog outputs. This is done at the factory prior to shipment, however it is a relatively simple procedure that can be performed in the field if desired. To perform a calibration, a multimeter with

a current input is required. Please keep in mind that the accuracy of the instrument used to calibrate the outputs is directly related to the accuracy of the outputs after calibration, so a meter calibrated against NIST (National Institute of Standards and Technology) standards is preferred. Before performing any calibrations, all other devices must be disconnected from the analog outputs. Multiple devices on the outputs will cause inaccurate measurement of current.

Attach the leads of the multimeter to the terminals for Output 1. The positive lead should be attached to Terminal #1091 and the negative lead to Terminal 1101. Select "Zero Output 1" and press the "Prep for Cal" button. The low limit of the output is 4mA, so 4.000 is shown as the default measured value. Ideally the meter connected to the outputs will also show 4.000. If the two values are not close enough to obtain the desired level of accuracy, a calibration should be performed. If, for example, the meter connected to the outputs reads 4.216mA, then the value "4.216" should be entered on the screen as the Measured Value. Once the value has been entered, press the "calibrate" button. This will offset the mA output of the instrument in an amount that results in an exact output of 4.000mA. When the calibration is complete, the multimeter should be reading 4.000mA (+/- .005).

The procedure can be repeated for the Span of Output 1. When "Span Output 1" is selected and "Prep for Cal" is pressed, the instrument will output the high output limit, which is 20mA. The multimeter will display the actual output from the instrument, and if it is not within the desired tolerance it can be calibrated using the same procedure as above.

To calibrate Output 2, attach the leads of the multimeter to Terminal #1091 (+) and the negative lead to Terminal 1111 (-) and follow the same procedure that was used for the first output.

Sensor Calibration

A proper calibration of the sensor requires two gases. The first gas should be pure Nitrogen or Argon. This contains no Hydrogen, and is therefore referred to as the Zero Gas. The second gas is the Span Gas. The Span Gas should ideally contain a quantity of Hydrogen similar to the amount of Hydrogen in the process gas. The Span Gas should also include any other gases that are present in the process gas in their respective percentages. The more similar to the process gas the calibration gas is, the more accurate the calibration will be.

Performing a Zero Calibration

On the Sensor Calibration page, select "Zero Hydrogen". Turn the valve on the side of the enclosure to the "Calibration Gas" position, and attach the Zero gas to the "Calibration Gas Inlet" port. Begin the flow of gas at a rate of 1.5 to 2.0 SCFH as measured on the flow meter on the door of the enclosure. The gas should not be under any pressure other than the amount required to maintain the appropriate flow amount. The target Value is shown on the screen. For a Zero Calibration, this will be 0.00 (the amount of Hydrogen in the Zero Gas). The Measured H2 Value can be seen at the bottom of the screen. When this value comes to equilibrium, it will not be showing any upward or downward trends, only the slight oscillation of the readings. This usually occurs in approximately 30 seconds. When the sensor is at equilibrium, press the green "Calibrate" button to perform the zero calibration. After the Zero Calibration is complete, turn off the flow of gas and disconnect it from the enclosure.

Performing a Span Calibration

To perform a span calibration, select "Span Hydrogen", attach the Span Gas to the Calibration Gas Inlet port, and begin the flow of gas at 1.5 to 2.0 SCFH. The Target Value should be set to the exact amount of Hydrogen that is in the Span Gas cylinder. Then the same procedure should be followed as the Zero calibration, with the "Calibrate" button being pressed after the readings reach equilibrium. After the Span Gas calibration is complete, turn off the flow of gas, disconnect the

cylinder from the enclosure, and restore the valve on the side of the instrument to the "Sample Gas" position. This will re-connect the sensor to the process gas stream.

Appendix A – Gas Nitriding Technical Paper

Introduction of the nitriding factor, KN as the driving force for gas nitriding, provides for precise, continuous control. The traditional measurement of % dissociation provides an alternative parameter for checking the accuracy of the calculated KN and in many cases, as the primary control parameter.

INTRODUCTION

The metallurgical processes of carburizing and nitriding have followed similar paths as the technology has advanced, and in a process of continuous evolution both procedures have progressed through similar developmental stages. Carburizing, early on, was conducted by packing the work pieces in a thick layer of carbon powder and raising them to temperatures conducive to diffusion of carbon into the work. While the process was effective, it was excessively slow, and difficult to control, so it progressed into a process utilizing a carbonaceous gas atmosphere. The only effective way of controlling this process was to establish a relationship between the enriching gas flow, and measurement of the carbon potential, using either periodic shim stock or dew point measurements. This technique persisted until the early seventies when the zirconia carbon sensor was first introduced. This device provided continuous measurement of the carbon potential, rather than the discontinuous measurements from shim stock or dew point. The measurement has been subsequently refined, by adjusting the sensor calibration using 3-gas (CO, CO₂ and CH₄) IR analyzers to calculate a more accurate carbon potential. The technique is currently applied to carbon control using continuous non-dispersive infrared analyzers, which measure continuously rather than periodically.

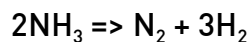
Nitriding has followed a similar path. The process provides several advantages for the alloys treated, such as high surface hardness, wear resistance, anti-galling, good fatigue life, corrosion resistance and improved sag resistance at temperatures up to the nitriding temperatures. Since the early beginnings of gas nitriding, the % dissociated ammonia (PD) was considered the appropriate control parameter. This parameter is easily (but discontinuously) measured using the ammonia dissociation burette, which is calibrated directly in PD. In the 1990's, however, a new control parameter, the nitriding potential, was introduced. This parameter is defined by the equation

$$Kn = pNH_3/pH_2^{3/2}$$

It is now the specified control parameter for many industry material specifications.

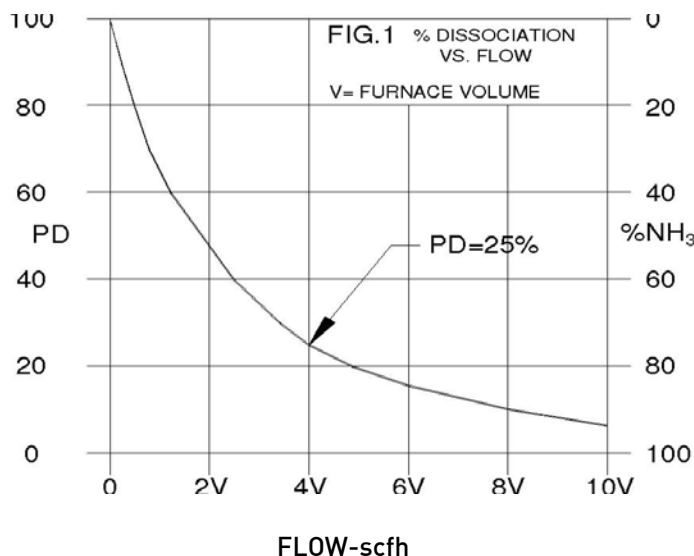
THE TECHNOLOGY

When ammonia is introduced into a furnace, it dissociates according to the following reaction:



Although this reaction will ultimately go to completion, it is basically very slow. It has been determined that, if the furnace atmosphere is changed four times every hour, the amount that the ammonia is dissociated is 25% ±10 %. An approximate relationship between ammonia flow rate and % dissociation is shown in Fig. 1. The general shape of the curve will vary with the particular nitriding

furnace, and will also likely be affected by the size and surface area of the load. Fig. 1 also illustrates the basic relationship of ammonia concentration in the furnace atmosphere to the % dissociation, i.e. $PD = 100 - \%NH_3$.



Nitriding has historically been conducted as either a single-stage or a double stage process using anhydrous ammonia. Temperatures in the single stage process range from about 925°F to 975°F, and dissociation from 15% to 30%. The first stage normally lasts for a period of 4 to 10 hours. The process creates an intense layer at the work surface, which is known as the white nitride layer. This layer is rich in dissolved nitrogen and nitrides. When the two-stage process, known also as the Floe process (U.S. Patent 2,437,249) is used, it has the advantage of narrowing and modifying the intensity of the white layer. The second stage may proceed at the same temperature as the first stage, or may be raised to from 1025°F to 1050°F.

The second stage PD is increased to 65% to 80% which allows the further diffusion of white layer components toward the core, eventually eliminating the layer. This PD may be achieved by reducing ammonia flow rate, or by adding diluents such as nitrogen, dissociated ammonia or hydrogen, in order to ensure adequate positive flow. The nitriding process is carried out by the catalytic generation of nascent (monatomic) nitrogen at the surface of ferrous alloys. The nascent nitrogen diffuses into the surface and exists in the work surface as either dissolved nitrogen or as iron nitrides such as the α' phase (Fe_4N) or the γ' phase ($Fe_{1-2}N$). There are several alloying elements in alloy steels that also contribute to the favorable characteristics provided by nitriding. Notable are aluminum, chromium, molybdenum, tungsten and vanadium, all of which form nitrides that are stable at nitriding temperatures.

In addition to microscopic cross section evaluation of work samples, effective processes are often evaluated by plotting hardness profiles with depth. Because these profiles are significantly affected by the core hardness, the work should be pre-tempered (before nitriding) at the lowest possible temperature, usually 30°F to 50°F above the nitriding temperature.

PROCESSING

Metallurgists have developed proprietary recipes for a large variety of products, tailored to suit their requirements for these products. An extensive list of these recipes is provided in the ASM Handbook, Vol. 4, Heat Treating, March 2001. Single stage processes vary in time from 8 hrs to 48 hrs. Two stage processes vary in time between 42 and 127 hours, with the second stage time being between 2 and 5 times the first stage time.

An aerospace industry standard was published in 1999, SAE Aerospace Material Standard AMS2759/10, which stipulates control with the Kn nitriding potential as the primary control

parameter. This standard specifies three classes of processes. Class 0-has no white layer, Class 1-0.0005" maximum white layer and Class 2-0.001" maximum white layer. Nitriding potentials for all classes range from 4 to 15 for the first stage. They range from 0.2 to 0.8 for the second stage of Class 0, from 0.4 to 2.6 for the second stage of Class 1 and from 1.2 to 5.5 for the second stage of Class 2. Treatment times are not stipulated. This specification provides a brief description of essential controls for nitriding processes:

Super Systems nitriding control systems meet this specification. SSi control systems also provide cascade temperature controls, and sufficient memory for 300 twelve-step programs.

SUMMARY

Nitriding control by % dissociation will continue to be a significant portion of the nitriding market place. The introduction of the nitriding potential, K_n presents a viable alternative, but there is insufficient data on effectiveness of the two parameters to make a judgment about which is preferable. Super Systems addresses this paradox by providing both parameters as a feature of all systems. As with carbon control, where %C is calculated from the measurement of oxygen, both nitriding control parameters (PD and K_n) are precisely calculated by an SSi proprietary analytical technique.

It is important to note that, unlike in carburizing, the gases used in gas nitriding are extremely pure, and accordingly the calculations used in nitriding process control are stable, and reproducible. The importance of superior temperature control has been demonstrated, and is related to the fact that retort temperature will have a significant effect on % dissociation. A change in retort temperature will cause a change in the PD curve displayed in Fig. 1. This characteristic is addressed by the provision of advanced cascade temperature controls in SSi nitriding systems.

(Written by Thomas H Lotze)