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Decarburization-Free and Soot-Free Batch-Annealing with Automated Atmosphere Control

A new method of controlling a nitrogen/propylene furnace atmosphere was found to offer substantial and repeatable results annealing ferrous wire and wire rod.

By Chris Messina, Arno Muennich and Robert Blumenthal
(As presented on May 13th at Interwire 2003)

FOREWARD

The article deals specifically with a new method of controlling a nitrogen/ propylene protective atmosphere for the annealing process. This new method uses a zirconium oxide sensor in conjunction with a proprietary algorithm in order to determine the optimum atmosphere flow settings. The proprietary algorithm, known as "Annealing Process Equilibrium", is hereafter referred to as "APEx".

To understand the importance of APEx, it is necessary to know the historical development of the nitrogen/propylene atmosphere. The first half of the article describes the evolution of this atmosphere. The second half of the article details the APEx technology and gives some field results.

PART-I

BACKGROUND

The necessity for a protective atmosphere during the annealing of steel is well known with various atmospheres available to processors. Historically, gas generators were typically used because high purity gases were not available at an affordable rate. Gas generators produce a background gas of mostly nitrogen, with a small hydrocarbon component. As bulk, high-purity nitrogen became affordable, and as maintenance staffs became leaner, gas generators began being replaced by delivered nitrogen.

Processors soon realized that inert nitrogen alone could not replace generated gas for all applications. With a purely inert gas, the process relies on the dilution of decarburizing agents (O_2, H_2O, and CO_2). For end-uses that could not tolerate decarburized wire, high flows of high-purity nitrogen were necessary to keep the decarburization agent levels low. Further, the annealing equipment relied on atmosphere chamber integrity—a leaky furnace needed yet more nitrogen. Thus, the true cost of nitrogen began to reflect the desired product quality coupled with the condition of the annealing equipment.

Since most generated gases retain reactive hydrocarbons that scavenge oxygen to prevent damaging of the work, additives to pure nitrogen soon became commonplace to provide this reactivity. Propylene became the most utilized because of its low cracking temperature and its relative low cost. (Since very little propylene is needed, the operational cost impact goes virtually unnoticed.) Thus, by adding a small amount of propylene, less nitrogen could be used for diluting since the cracked propylene scavenges the oxygen and other decarburizing agents.

How much propylene to use? The answer to this central question has remained elusive since propylene began being used as a replacement for generated gas. Too little propylene provides insufficient protection against decarburizing agents, but with too much propylene, soot deposits are left on the charge. Soot leads to cleaning and coating issues, and ultimately higher operating costs.

TRADITIONAL PROPYLENE CONTROL

A typical plant will use predetermined gas flow rates to strive for a consistent atmosphere. The nitrogen and propylene flows are dictated by a predetermined recipe, and are coordinated with the temperature control events. Figure 1 shows a typical example.

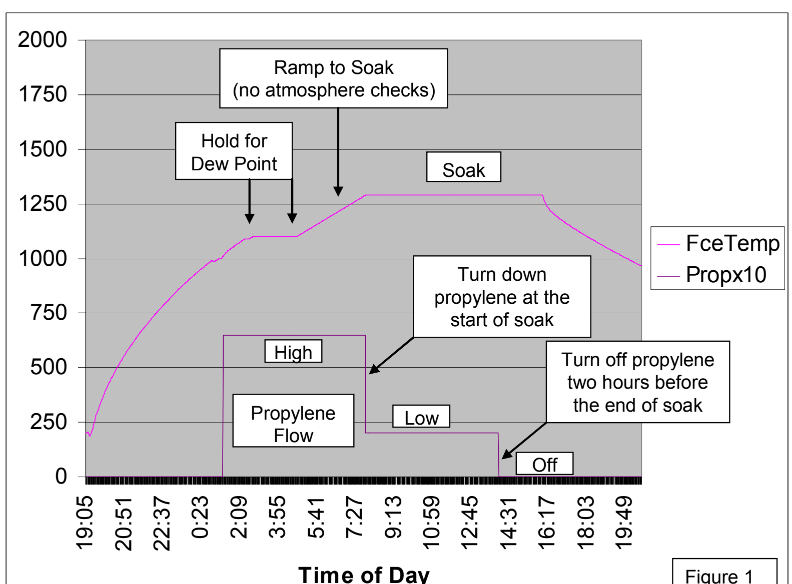

Figure 1

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This type of atmosphere control is known as "open-loop" because it does not use any immediate feedback from the process. Thus, the loop is only closed when the lab results are in—if there is decarburization, maybe the atmosphere can be corrected on the next cycle. But since the annealing cycles are long (more than a day), this type of feedback is difficult to implement. In practical terms, the metallurgist adds/lengthens cycle pre-holds and increases gas flows. Over time, the cost of a cycle slowly increases, costing the processor thousands of dollars in "hidden costs".

To get consistent results using pre-determined flows, the rest of the process must be made repeatable. The wire must go into the furnace with the same coatings and contamination. Also, the annealing equipment must be maintained in top shape. Deviation in any upstream parameters will lead to variability in final results. Commercial processors of ferrous wire are particularly susceptible to "variable upstream conditions". There is also a cost to "level" these initial conditions.

CLOSING THE LOOP WITH GAS SAMPLING

Since the introduction of propylene, many gas-sampling systems have been installed in an effort to close the process loop. The idea has been to sample the atmosphere, try to understand the reaction, and then make cycle adjustments online.

The first widely used, and still most common atmosphere measurement in the USA, is an Alnor dew pointer. This is a manual unit, requiring an operator to pressurize a sample and visually find the point at which fog is formed. Processors hold at a pre-soak (a.k.a. "Dew Point Hold", see Figure 1) for a specific dew point in an effort to level the conditions. It is the operator's responsibility to "resume" the cycle after he reaches the specified condition.

As processors realize that the critical period of the cycle is between 1100°F and 1300°F, an attempt is made to integrate into the process: automatic dew pointers, carbon dioxide detectors, and oxygen analyzers. Due to the cost of analyzers, it is very common to see one analyzer multiplexed to multiple annealing furnaces.

What these processors soon discover is that it is practically impossible to maintain consistent measurement of these parameters. The key point: the decarburizing agents (O_2, H_2O, and CO_2) all occur in small quantities in a nitrogen/propylene atmosphere. Thus, the analysis must be extremely robust in order to work consistently long term. In fact, with the probability of some soot at some point, and the various coatings that are volatized, keeping sample lines clean is a maintenance-intensive prospect. In addition, a "shift" occurs as the sample is cooled, leading to variations in measurement. So, development of an understanding of this atmosphere has been hampered by the fact that consistent process measurement has not been achieved.

PART II

PROPYLENE CONTROL USING APEx

The basic philosophy of APEx is to use an in-situ probe to directly measure the partial pressure of oxygen, and then combine that value with the temperature and a carbon monoxide measurement in order to fully understand all the reactions that are occurring. The reactions are summarized in the APEx heating factor (HF) and the APEx cooling factor (CF). These are the values that are used to determine gas flows and cycle holds.

The carbon monoxide is measured using a traditional multiplexed sampling system, but the critical trace values are determined using the in-situ probe. Carbon monoxide in this process varies from 0.0-3.0%, but is typically over 0.25% in the critical portions of the cycle. Thus, the sampling system can be a traditional design requiring very little maintenance. The key trace element is measured using the proven technology of a zirconium oxide sensor, in-situ (i.e. the sensor is inside a probe that is inserted into the process atmosphere). The probe also contains a thermocouple to give temperature feedback. See Figure 2 for a system schematic.

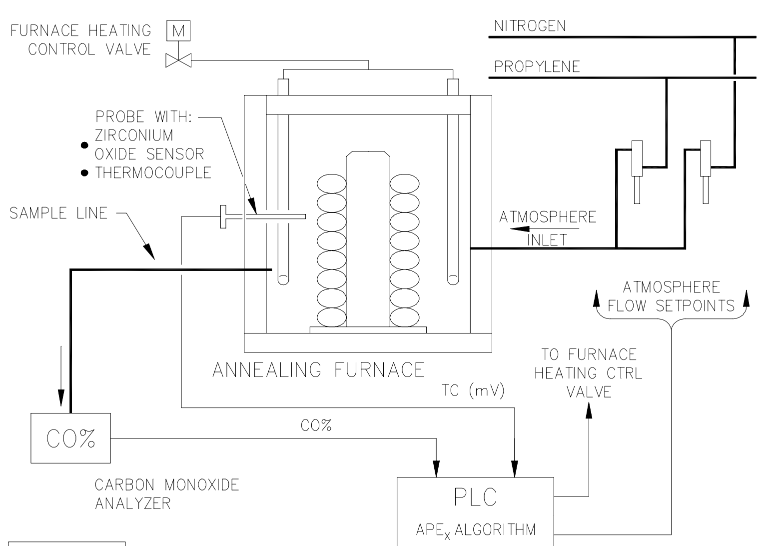

FIGURE 2

THE IDEALIZED APEx CYCLE

The APEx algorithm is involved in two distinct segments of the cycle: the heat-up from 1100°F to soak ~1300°F (using APEx HF), and the propylene turn-down phase during soak (using APEx CF). See Figure 3.

It can be seen that the APEx cycle does not have a "dew point hold". Rather, an "APEx HF Setpoint" is used to control the ramp from 1100°F to soak (~1300°F). Starting at 1100°F, the temperature setpoint is ramped up as fast as atmosphere conditions permit. When the measured HF is less than the HF Setpoint, the temperature setpoint is prevented from advancing higher, thus allowing the reactions to complete before decarburizing conditions occur. The advantage of this approach is that heat can continue to be increased providing a steady force for all the gas reactions to complete. In particular, the oxides entrained in the charge and furnace fixtures, continue to be liberated as heat is increased. The effectiveness of APEx HF centers around keeping the conditions "on the edge" while driving off and reacting with these contaminants.

As the charge and furnace fixtures approach an equilibrium temperature during soak, these reactions complete, and the need for an aggressive gas scavenger diminishes. It is important to stop the propylene when it is no longer necessary, otherwise the cracked gas, having no contaminants to react with, will simply deposit soot. The APEx CF expresses the level of residual contaminants. When the measured CF is lower than the CF setpoint, propylene is shutoff. Actually, it is first turned down to a secondary flow, and then shutoff. The only contaminants at this point are a result of furnace leaks—the tighter the furnace, the greater the atmosphere gas conservation. An extremely leaky furnace may require that propylene flow be maintained at a minimum level for some longer period of time.

AN APEx APPLICATION

APEx has been proven on both bell-type furnaces and box-type furnaces of a batch nature. With the courtesy of Bluff City Steel, LLC, recent field results on a box furnace in their Memphis, TN USA plant are shared. It is important to understand that APEx was implemented on existing furnaces, modifying an existing process.

General results after implementing APEx at Bluff City Steel on THREE furnaces:

- Immediate reduction of nitrogen by 20% (with further reductions contemplated).
- Reduction to near-zero additional decarburization on virtually every charge.
- Consistent cleanliness out of anneal, improving coating consistency.
- Removal of operator dew point holds—thus eliminating inconsistency due to human factors (i.e. dew pointer operation, timing of dew point checks, etc.).

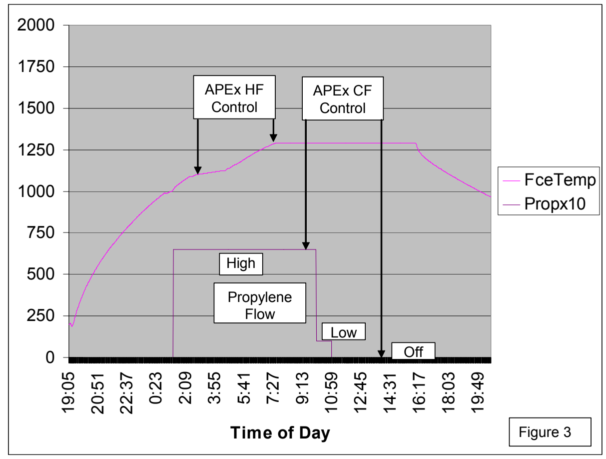

Figure 3

CYCLE RESULTS

The general results can be seen in figure 4. This cycle was run in Bluff City Steel's #2 Box Furnace, a radiant tube heated unit with one large circulation fan in back and one large overhead door in the front. This furnace is loaded with a boom-truck through this front door.

The APEx HF was below its 1.5 setpoint when the cycle reached 1100°F, so the cycle was halted until the HF reached 1.5. At that point, there was a series of stops/restarts as the temperature was raised "carefully". The total time to get from 1000°F to the start of soak was a time similar to Bluff City's previous practice of hold and check dew point, but with much better decarburization performance and less nitrogen.

After 2.5 hours into soak, APEx CF turned the propylene flow down from maximum flow down to a "bleed" flow. After another hour, the propylene was turned off altogether.

As the charge is unloaded, the soot deposition is measured visually against a standard scale of 1-5, 1 being the cleanest. This charge was deemed a 1. With subsequent tuning, coils that are removed from the APEx controlled furnaces at Bluff City Steel are consistently able to achieve a "1" rating.

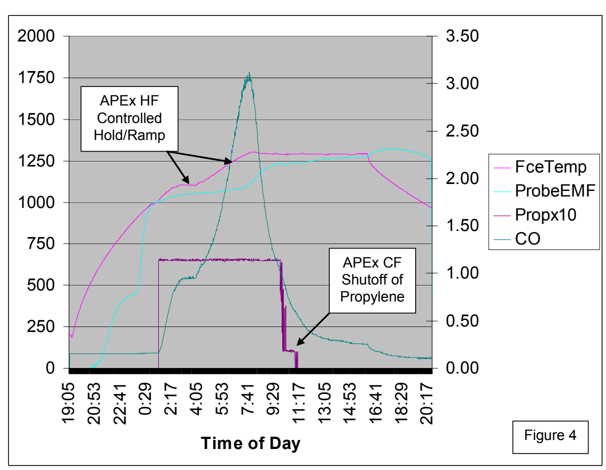

Figure 4

LAB RESULTS

Mounts of the AISI 1040 material annealed in the above cycle using the APEx process can be seen in figures 5a/5b. Although the excellent 100% spheroidization can be seen, APEx cannot take credit as this is a result of time-at-temperature and controlled cooling. The effect of APEx can be seen at the left edge of the material where a consistent carbide matrix extends all the way to the surface. These results were reported as an average 0.001" depth of partial decarburization, with 0.000" depth of total decarburization.

Figure 5a: Before Annealing

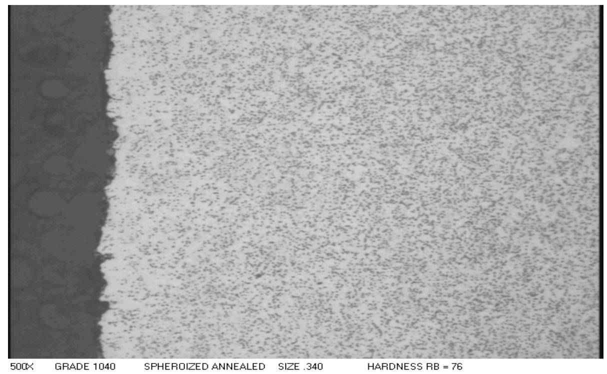

Figure 5b: After APEx Annealing

CONCLUSIONS

A properly controlled nitrogen/propylene atmosphere can achieve repeatable results on subcritical annealing of ferrous wire and wire rod. The keys to success are:

- Accurate and repeatable measurement/inference of decarburizing agents that occur at trace levels (O_2, H_2O, and CO_2).
- Integration of the temperature control with the atmosphere control.
- Control of the critical ramp from 1100°F to soak (~1300°F) to prevent decarburization.
- Avoidance of carbon deposition by shutting off the propylene during soak after all major reactions are complete.

The mark of a breakthrough technology is one that opens the door to many additional possibilities. Some of the additional benefits being considered/pursued:

- Lowering nitrogen flows to absolute minimum levels.
- Using additional higher flows of propylene to speed up cycle time (leading to cost savings in fuel, electricity, and nitrogen).
- Controlling depth of partial decarburization to meet specific manufacturing requirements of wire users.
- Reducing/eliminating the air burnoff segment to introduce less oxidation and speed-up the APEx ramp time on subsequent cycles.
- Applying APEx to continuous annealing processes that use a protective atmosphere.

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As Vice President of Technology at RAD-CON, Mr. Messina has been developing various innovations in the batch annealing process and specifically in the area of atmosphere control. He has worked in the heat-treating field in various engineering and developmental capacities since 1987. Mr. Messina holds a Bachelor of Science degree in Engineering Physics from John Carroll University (Cleveland, OH USA).	Mr. Muennich currently heads RAD-CON's control system development since assuming the Chief Systems Engineer position in 1996. Mr. Muennich has been in the industrial controls field since 1987. He holds a Bachelor of Science degree from the Electronic Technology Institute (Cleveland, OH USA).	As both president and co-founder of Furnace Control Corporation and Advanced Atmosphere Corporation, Dr. Blumenthal developed zirconium oxide technology for controlling heat-treating atmospheres throughout the 1970s, 80s, and 90s. Dr. Blumenthal taught Materials Engineering for 32 years at Marquette University and was granted the title of Professor Emeritus upon retirement in 1996.