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Steel Heat Treating Process Control—An Introduction

THE HEAT TREATMENT OF STEEL involves a number of processes (summarized in Table 1 and Refs 1,2) to condition the microstructure and obtain desired properties. Treatments include stress relieving, normalizing, and various types of annealing procedures (full annealing, intercritical annealing and subcritical annealing, recrystallization annealing, isothermal annealing, soft annealing, diffusion annealing). Heat treatment of steel also includes direct hardening and surface hardening. Direct hardening involves austenitization followed by rapid cooling (quenching) that produces a supersaturated solid solution of carbon in the form of a very hard metastable crystal structure known as

Table 1 Summary of typical heat treating processes for carbon and low-alloy steels

Process	Characteristics
Austenitization: Complete transformation to austenite by heating the steel above the critical temperature for austenitic formation	The optimal austenitization temperature is 30–50 °C (55–90 °F) above Ac_3 for hypoeutectoid steels and 30–50 °C (55–90 °F) above Ac_1 for hypereutectoid steels. Ac_3 is the temperature at which the transformation of ferrite to austenite is completed during heating. Ac_1 is the temperature at which austenite begins to form during heating. The heating rate must be limited and uniform to avoid cracking or warpage and to control thermal stresses in the range of 250–600 °C (480–1110 °F). The carbon equivalent controls the propensity for steel to crack. The holding time is dependent on geometrical factors related to the furnace (emissivities, temperature, and atmosphere composition) and load (type of steel and thermophysical properties).
Annealing: Heat treatment consisting of heating and soaking at suitable temperature followed by cooling under conditions such that, after return to ambient temperature, the metal will be in a structural state closer to that of equilibrium The primary purpose of annealing is to soften the steel to enhance its workability and machinability. Also, it relieves internal stresses, restores ductility and toughness, refines grains, reduces gaseous content in the steel, and improves homogenization of alloying elements.	Full annealing: Heat 30–50 °C (55–90 °F) above Ac_3 for hypoeutectoid steels, then furnace cool through the critical temperature range at a specified cooling rate. The aim is to break the continuous carbide network of high-carbon steels. It improves machinability. Partial (intercritical) annealing: Heating within the critical temperature range (Ac_1–Ac_3), followed by slow furnace cooling. It improves machinability. Subcritical annealing: Heating 10–20 °C (20–35 °F) below Ac_1 followed by cooling in still air. It can be used to temper bainitic or martensitic structures to produce softened microstructures containing spheroidal carbides in ferrite. Improves the cold working properties of low carbon steels (<25% C) or softens high-carbon and alloy steel Recrystallization annealing: Heat the steel for 30 min–1 h at temperature above the recrystallization temperature (T_R = 0.4 T_m), then the steel is cooled. The treatment temperature depends on prior deformation, grain size, and holding time. The recrystallization process produces strain-free grain nucleation, resulting in a ductile, spheroidized microstructure. Isothermal annealing: Heating the hypoeutectoid steel within the austenitic transformation range above Ac_3 for a time sufficient to complete the solution process, yielding a completely austenitic microstructure. At this time, the steel is cooled rapidly at a specific rate within the pearlite transformation range until the complete transformation to ferrite plus pearlite occurs, and then it is cooled rapidly. Spheroidizing (soft annealing): Involves the prolonged heating of steel at a temperature near the lower critical temperature (Ac_1), then furnace cooling Diffusion (Homogenizing annealed): Heat the steel rapidly to 1100–1200 °C (2010–2190 °F) for 8–16 h, furnace cool to 800–850 °C (1470–1560 °F), and then cool to room temperature in still air. It is performed on steel ingots and castings to minimize chemical segregation.
Normalizing: The aim is to provide a uniform microstructure of ferrite plus pearlite (small grains and finer lamellae than in annealing).	Heat the steel to 40–50 °C (80–90 °F) above Ac_3 for hypoeutectoid steels and 40–50 °C (80–90 °F) above A_{cm} for hypereutectoid steels. The holding time depends on the size, and then the steel is cooled in still air. It produces grain refinement and improved homogenization.
Stress relieving: It is typically used to remove residual stresses that have accumulated from prior manufacturing processes. Stress relieving results in a significant reduction of yield strength in addition to reducing the residual stresses to some "safe" value.	Heat to a temperature below Ac_1 for the required time to achieve the desired reduction in residual stresses, and then the steel is cooled at a rate sufficiently slow to avoid the formation of excessive thermal stresses. Below 300 °C (570 °F), faster cooling rates can be used. No microstructural changes occur during stress-relief processing. The recommended heating temperature range is 550–700 °C (1020–1290 °F), depending on the type of steel. These temperatures are above the recrystallization temperature. Little or no stress relief occurs at temperatures < 260 °C (500 °F), and approximately 90% of the stress is relieved at 540 °C (1005 °F). The maximum temperature for stress relief is limited to 30 °C (55 °F) below the tempering temperature used after quenching. The results of the stress-relief process are dependent on the temperature and time.
Hardenability: Ability to develop hardness to a given depth after having been austenitized and quenched	The hardenability depends on the concentration of dissolved carbon in the austenitic phase, alloying elements, austenitizing temperature, austenitic grain size at the moment of quenching, size and shape of the cross section, and quenching conditions.
Quenching: Quench severity is the ability of a quenching medium to extract heat from a hot steel workpiece.	Specific recommendations for quench media selection for use with various steel alloys are provided by standards such as SAE AMS 2759. Quench media include water, brine, aqueous polymer, gas or air quenching, and caustic quenching.
Tempering: Tempering is the thermal treatment of hardened and normalized steels to obtain the desired mechanical properties, which include improved toughness and ductility, lower hardness, and improved dimensional stability.	The tempering process involves heating steel to any temperature below the Ac_1 temperature. During tempering, as-quenched martensite is transformed into tempered martensite, which is composed of highly dispersed spheroids of cementite (carbides) dispersed in a soft matrix of ferrite, resulting in reduced hardness and increased toughness. The objective is to allow the hardness to decrease to the desired level and then stop the carbide decomposition by cooling. The extent of the tempering effect is determined by the temperature and time of the process.

Source: Ref 1, 2

martensite. Similarly, the surface of steel can be hardened by martensitic hardening, although a number of other thermochemical treatments (not included in Table 1) also are used to surface-harden steel. For more details see ASM Handbook Volume 4A, *Steel Heat Treating Fundamentals and Processes*.

Although the basic principles of steel heat treatment are well-understood, the link between principle and practice can be complex. Many variables are involved in the practice of steel heat treatment. Heat-treating specifications define minimum requirements but do not necessarily address all critical aspects of heat treatment practice. As can be seen in Table 2 (Ref 3), there are many variables that may influence the procedure and results of a heat treatment. The fish-bone diagram in Fig. 1 (Ref 4) also identifies variables in a neutral hardening operation.

The decision to control, monitor, or ignore a given variable is a decision that must be made by taking into account the importance of the variable and the ease with which it can be measured. This decision is ultimately a balance of economic and quality considerations. There are many process parameters of interest in almost any heat-treating operation. These fall into three categories:

- variables which *must* be closely controlled in order for the process to occur (for example, temperature, time, and so on);
- variables that are known to affect the process but are either ignored or simply monitored manually because of the complexity or cost involved; and
- variables which cannot be controlled at all (for example, natural gas composition, available heat input, and so on).

Some basic factors common to steel heat treatment procedure qualification in general are (Ref 3):

- Alloy grade/composition
- Maximum section size
- Time-temperature profile during heat treatment
- Furnace loading at full load condition

The critical variables depend on the process, of course. On a batch atmosphere carburizing furnace, for example, the common process variables that require control are:

- Temperature
- Atmosphere carbon potential
- Time
- Endothermic gas flow
- Enriching gas flow
- Atmosphere circulation
- Quenchant properties (temperature and agitation)

Whereas the first two on the list, temperature and carbon potential, will commonly be automatically controlled and the third, time, will usually be held fairly closely by either automatic or manual means, the last four on the list often go without notice *until a problem develops*, at which time they are examined. However, in keeping with the idea that process control represents *preventative* action, other critical variables should obviously be given more attention, possibly on an automatic, ongoing basis.

Time-Temperature Profiles

Obviously, it is the time-temperature profile of the part, rather than the time and temperature of the furnace, that controls treated properties. This profile depends not only at the time-at-temperature but also on the time-to-temperature and furnace ramp-up. Section size as well as

Table 2 List of heat treatment variables to consider for procedure qualification

Heat treatment variable	Possible consideration in procedure qualification
Casting	. . .
Grade	Grade/or composition
Composition	Grade/composition
Min. section size	Not significant
Max. section size	Max. section size
Weight	Max. section size
Compactness or variation in part size	Max. section size
Microstructure	Not significant
Prior heat treatment	Not significant
Surface condition	Not significant
Heat treatment equipment	
Furnace type	Heat treatment, time, and temperature
Furnace size	Heat treatment, time, and temperature
Burner location	Heat treatment, time, and temperature
No. of burners	Heat treatment, time, and temperature
Insulation	Heat treatment, time, and temperature
Burner operating characteristics	Heat treatment, time, and temperature
Refractory supports	Heat treatment, time, and temperature
Door seal type	Heat treatment, time, and temperature
Door seal condition	Heat treatment, time, and temperature
Furnace control	Heat treatment, time, and temperature
Ramp-up control	Heat treatment, time, and temperature
Zone control	Heat treatment, time, and temperature
Cool-down control	Heat treatment, time, and temperature
Control T/C location	Heat treatment, time, and temperature
Control T/C response	Heat treatment, time, and temperature
Atmosphere	Not significant
Hold temperature variation	Heat treatment, time, and temperature
Heat treatment practice	. . .
Set temperature	Heat treatment, temperature
Load temperature	Heat treatment, temperature
Ramp-up time	Heat treatment, time
Hold time	Heat treatment, time
Basket/rack geometry	Full load condition
Weight of load	Full load condition
Density of load	Full load condition
Load location	Full load condition
Initial furnace temperature	Heat treatment, time, and temperature
Ramp/hold time criteria	Heat treatment, time, and temperature
Hold time variation	Heat treatment time
Quenching equipment	. . .
Quenchant type	Quenchant type
Tank volume	Initial quench temperature
Make-up water conditions	Initial quench temperature
Pump Inlet/outlet locations	Initial quench temperature
Quench practice/control	. . .
Quench velocity	Quench velocity
Delay time	Delay time
Initial quench temperature	Initial quench temperature
Initial load temperature	Not significant
Load volume	Full load condition
Load surface area	Full load condition
Quenched hardness	Minimum as-quenched hardness
Final casting temperature	Final quench temperature
Casting surface condition	Not significant
Localized quench velocity	Quench velocity
Localized quench temperature	Final quench temperature
Normalizing	. . .
Ambient temperature	Not significant
Air velocity	Air velocity
Air humidity (relative)	Less significant
Load volume	Full load condition
Load density	Full load condition
Localized air velocity	Air velocity
Localized temperature	Air

Source: Ref 3

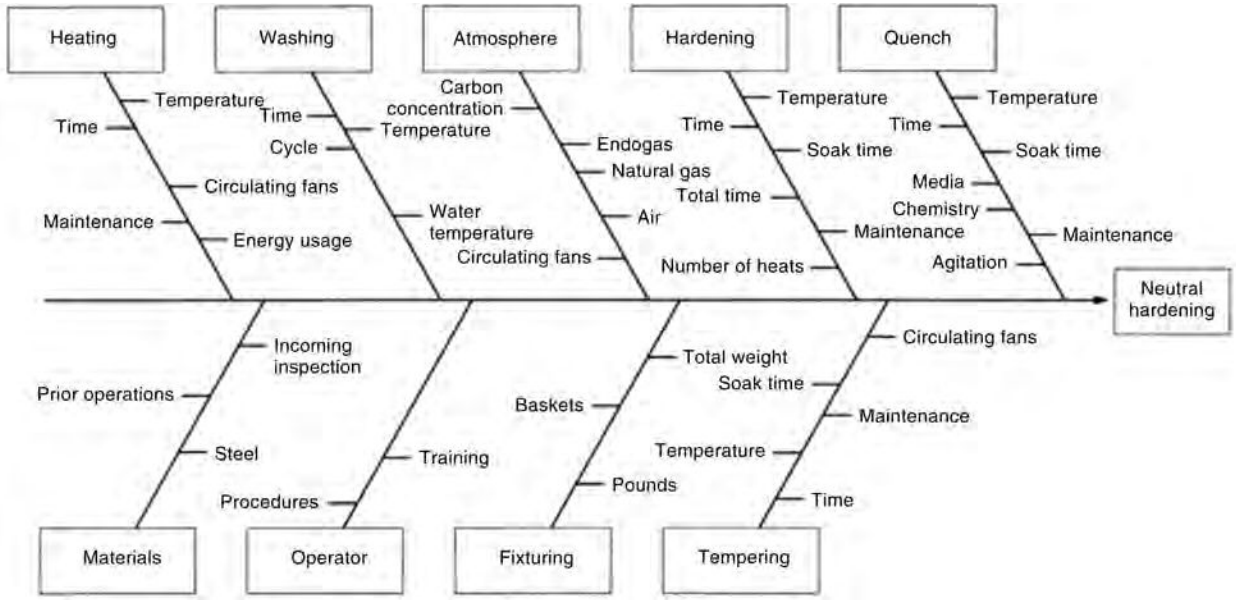

Fig. 1 Identification of heat-treating variables for the neutral hardening process

overall and local furnace loading will affect the time required to reach the austenitizing temperatures. Figure 2 (b) (Ref 5) shows the time difference to reach the set-point temperature for both a large load and a large section size. This figure also illustrates the importance of using load thermocouples rather than simply measuring time in the furnace when conducting heavy section heat treatments in loaded furnaces. Parts in the center of a heat treatment basket in fully loaded furnaces can be expected to have significantly different time-temperature profiles than those in a partially loaded furnace. Furnace loading also may be carefully planned to provide adequate circulation of furnace gases.

Challenges in temperature monitoring of loaded furnaces have led to overly conservative "hour-per-inch" rules to ensure that the centers of loads reach the appropriate temperature. These practices often result in holding at temperature far longer than is necessary. With modern temperature data acquisition systems, however, thermal conditions can be monitored in a furnace load, not just in the furnace itself. This improvement in technology affords the possibility of load-based heat treatment time and temperature cycles, which are more precise than heat treatment cycles based on furnace temperature (Ref 3).

Temperatures in the ranges of interest in heat treating are generally measured by one of two methods: thermocouple or infrared pyrometer. Because of its low cost, simplicity of construction, and inherent reliability, the thermocouple has always been and continues to be by far the most useful sensor in most situations. It is perfect for sensing gas temperatures, and even works well in vacuum furnaces by virtue of radiation. It is not very useful, however, for measurement of the actual temperature of parts going through a furnace.

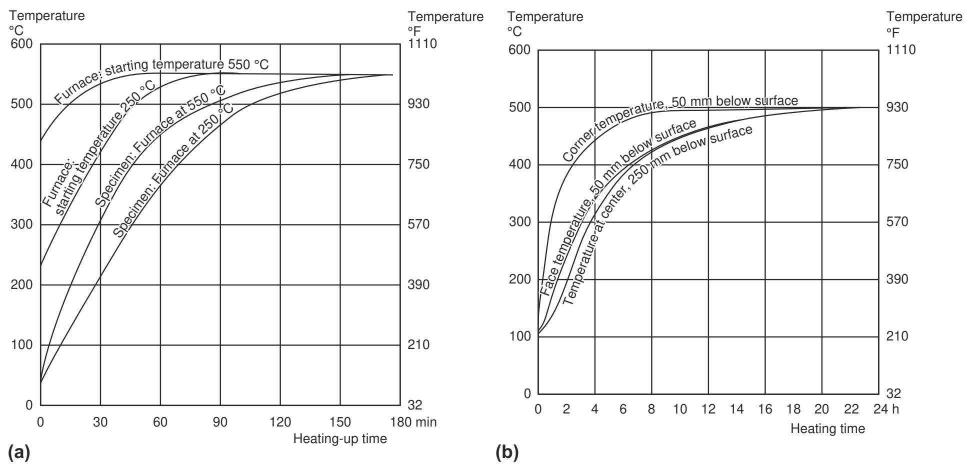

Fig. 2 Examples of heat-up time (a) effect of initial initial furnace temperature (b) temperature at different locations of a large load. Source: Ref 5

Because thermocouples often fail slowly by losing accuracy and because two thermocouples inserted at the same time will often agree even as they are both failing, it is important to change thermocouples on a regular schedule and to alternate replacements. Aside from the obvious process-related temperatures that are measured, it is possible that the temperatures of non-process-related items like cooling water, bearings, and door seals might be of interest. Thermocouples are most often used for these purposes.

The science of infrared temperature measurement has improved. This type of equipment is used in induction hardening but has been applied with mixed results in enclosed furnaces. In the latter, results are usually better if the furnace does not use a hydrocarbon atmosphere, as the presence of soot in the optical path presents a problem. More information is given in the article "Temperature Control in Heat Treating" in this Volume.

Most temperature control applications in heat treating are of the on-off type, such as electrical contactors for heating elements or high-low fire systems for combustion burners. Others are proportional, utilizing stoichiometric gas-air ratios for burners or variable current systems for heating elements.

Each of these systems has the effect of delivering a certain amount of energy to achieve and maintain the furnace at a given temperature. The amount of energy may be known by *direct* measurement with gas or electrical meters, but *relative* measurements may also be made by more advanced control instrumentation.

The amount of energy consumed is of course important from a cost standpoint. It is equally interesting, however, from the process/data acquisition standpoint. If the relative amount of energy required to heat and maintain a given furnace at a given temperature is known, then any additional energy consumed by any process run in this furnace must be absorbed by the load. This effect has been successfully utilized in helping to determine when loads are at heat, as shown in the following figures.

Notice in Fig. 3 how the heat input to the process drops down to some equilibrium value after the process has reached setpoint. This is the point at which heat input to the furnace equals heat loss through the walls and atmosphere effluent.

When load size is varied, as happens in most shops, the process suffers from a lack of information about how this might affect the results. By taking into account a very inexpensively acquired piece of information such as the percent output of a properly-tuned proportioning temperature controller, it is possible to begin compensating for load size by taking advantage of the fact that a heavier load requires more heat to reach setpoint.

The effect of load size on heat input is shown in Fig. 4. Notice that although the light and heavy loads have a relatively minor effect on how long it takes for furnace temperature recovery, load size has a potentially dramatic effect on heat balance equilibrium. In terms of data acquisition, the heat input to a furnace is a piece of information that is usually easy to capture and might be of significant value in learning to more tightly control the process.

Furnace temperature uniformity is always of tremendous concern, especially in vacuum applications where there is no convection to help even things out. Although the process of making a furnace uniform in temperature may be difficult, the actual uniformity results are easily documented with multiple thermocouples. It is not at all out of the question to monitor uniformity by permanently placing thermocouples in several furnace locations and automatically calculating and alarming against their average and spread. This approach has been used for years with vacuum furnaces (where uniformity is more a function of loading). The monitoring thermocouple is of value in evaluating uniformity and the repeatability of uniformity.

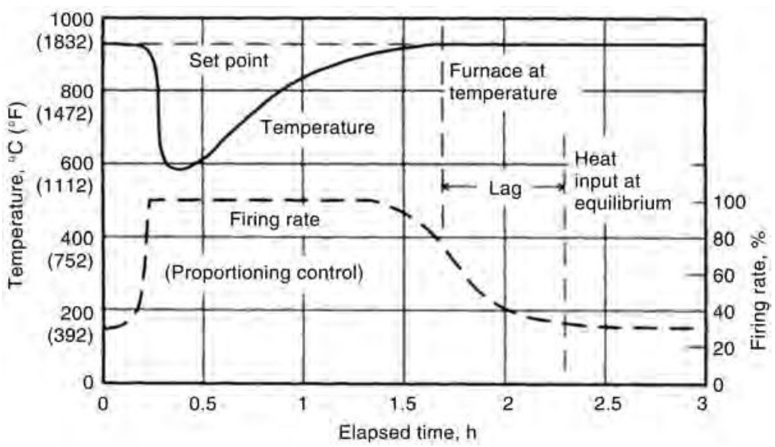

Fig. 3 Plot of furnace temperature versus elapsed time to show that heat input equilibrium lags behind attaining of setpoint temperature after furnace loading

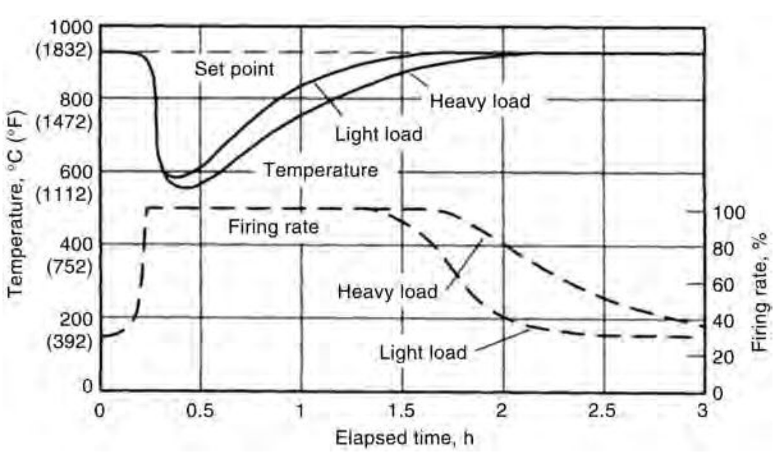

Fig. 4 Plot of furnace temperature versus elapsed time to show effect of load size on heat balance equilibrium

Temperature Uniformity Surveys

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Although the need for precise control of temperature during the heat treatment process is well understood, it must be recognized that temperatures throughout the furnace work zone can deviate significantly from the temperature controller's indicated value.

For this reason, several pyrometry-related specifications such as AMS2750E (Ref 6), CQI-9 3rd ed. (Ref 7) or ASTM A991/A 991M-08 (Ref 8) require that the heat treater perform regularly scheduled temperature uniformity surveys so as to confirm that temperatures throughout the furnace work zone remain within acceptable limits from the temperature control setpoint value.

These specifications also require that furnace temperature controls and their associated thermocouples meet specified accuracy requirements and be maintained on a regularly scheduled basis, since both of these factors have significant impact upon temperature control and temperature uniformity of the furnace work zone.

Furnace temperature control, monitoring, and recording devices must be calibrated at regular intervals to insure that their accuracies have not drifted outside of specified tolerances. Likewise, process thermocouples must be checked, typically via a systems accuracy test, to ensure that they have not accumulated sufficient error to place them outside of their required specified tolerance.

In the aggregate, data collected from the calibrations, system accuracy tests, and temperature uniformity surveys will provide the heat treater with objective evidence of the furnace system's capability to achieve and hold setpoint temperature values throughout the furnace work zone.

Temperature Uniformity Survey. A Temperature Uniformity Survey, commonly referred to as a TUS, is a testing procedure intended to map variations in temperature throughout the furnace work zone and, most importantly, to enable the heat treater to define the furnace system's qualified work zone volume and qualified operating temperature range.

AMS2750E (Ref 9) defines the qualified work zone volume as "[t]he defined portion of a furnace volume where temperature variation conforms to the required uniformity tolerance". CQI-9 3rd ed. has a similar definition, whereas ASTM A991/A 991M-08 simply acknowledges the importance of identifying this qualified work zone volume.

Work that is processed outside of the qualified work zone dimensions is deemed to have been run in a non-conforming furnace (Fig. 5) The qualified operating temperature range is defined in AMS2750E as "[t]he temperature range of thermal processing equipment where temperature uniformity has been tested and found to be within required tolerances" (Ref 10).

Whether the entire furnace work zone volume or only a portion of the furnace work zone volume is deemed to be qualified, it should be well documented and clearly communicated to furnace operators to ensure that materials to be processed are placed within the limitations of the qualified work zone dimensions.

Likewise, the qualified operating temperature ranges for the furnace system should be well documented and clearly communicated to the furnace operators to ensure that materials are not processed at temperatures outside this range.

Development of Temperature Uniformity Survey Procedures. Although the conducting of TUSs on a regularly scheduled basis can represent a significant investment for the heat treater, this testing procedure can provide valuable information, particularly over time, when performed correctly and in a consistent manner.

Hence, the initial TUS is of critical importance because it establishes the baseline position against which future TUS data for the furnace under test will be compared.

It is during this initial TUS that the temperature control tuning parameters will be confirmed, the need for temperature controller offset/bias will be evaluated, the control thermocouple location will be evaluated for effectiveness, combustion settings (in the case of a gas fired furnace) will be confirmed as suitable, and the furnace system's qualified work zone and qualified operating temperature range will be determined.

These baseline positions during our initial, successful TUS must be well documented, as they represent key furnace parameters required to achieve a favorable TUS outcome. As subsequent or periodic TUSs are performed, these baseline positions can assist in a root cause analysis in the event that a comparative study of historical TUS data indicates changes in furnace performance.

It is important to note that specifications such as AMS2750E (Ref 11), CQI-9 3rd ed. (Sections 3.4.1.1 and 3.4.1.2, (Ref 12)) or ASTM A991/A 991M-08 (Ref 13) require that the heat treater perform a new (initial/primary) TUS after any furnace modification that might alter the temperature uniformity characteristics of the furnace. Although these specifications list specific items that are deemed to be furnace modifications and thereby require a new (initial/primary) TUS, these lists are not intended to be exhaustive.

Examples of furnace modifications and actions that change the furnace from its original documented state include (Ref 14):

- Increase in the maximum qualified operating temperature
- Decrease in the minimum qualified operating temperature
- Change in burner size, number, or location
- Change in heating element number, type, or location
- Changes to airflow (baffle positions, fan speed, fan quantity, etc.)
- Change of refractory thickness
- New refractory with different thermal properties
- Change of vacuum furnace hot zone design or materials
- Change of control sensor location
- Change of combustion pressure settings from original settings
- Temperature control scheme changes (proportional vs high-low/on-off)
- Changes in temperature control tuning constants
- Work zone volume increase covering area not previously tested

This is not intended to be an exhaustive list, but gives typical examples of furnace modifications that could alter the temperature uniformity characteristics of the furnace and thus require an additional TUS.

Therefore, it is important for the heat treater to evaluate maintenance work performed on the furnace system and to determine whether such work could have an effect on temperature uniformity. If it is determined that the furnace qualified work zone temperature uniformity characteristics could be affected, production in this furnace cannot resume until a successful TUS has been achieved.

Ongoing TUS testing not only provides evidence concerning furnace process capability (which, as we shall see later, is the reproducibility of a process over a long time period), but also provides the heat treater an evaluation of the effectiveness of the furnace maintenance program.

Over time, the TUS test results will provide objective evidence as to whether the furnace maintenance program is adequate to allow the furnace, on a repeatable basis, to maintain temperature control meeting required tolerances as well as maintaining its established qualified work zone volume.

If successful TUS outcomes are to be achieved on a continuing basis, it is the maintenance program, specifically as it relates to pyrometry, that must be well developed.

Again, for this reason, several pyrometry-related specifications such as AMS2750E, CQI-9 3rd ed., and ASTM A991/A 991M-08 require that the heat treater adhere to specific requirements for such things as accuracy of control devices, accuracy of thermocouples, calibration intervals for temperature control devices, and system accuracy testing to confirm thermocouple integrity.

In the absence of an ongoing calibration program for our furnace temperature control devices or an ongoing system accuracy testing program to verify thermocouple integrity, the heat treater has little chance of achieving favorable TUS results for his furnace system.

Specifications such as AMS2750E, CQI-9 3rd ed. and ASTM A991/A 991M-08 require that the TUS be performed while mirroring typical furnace parameters used in production. This approach provides the heat treater with the most meaningful data, as it will allow for the analysis of furnace performance in a typical production mode of operation.

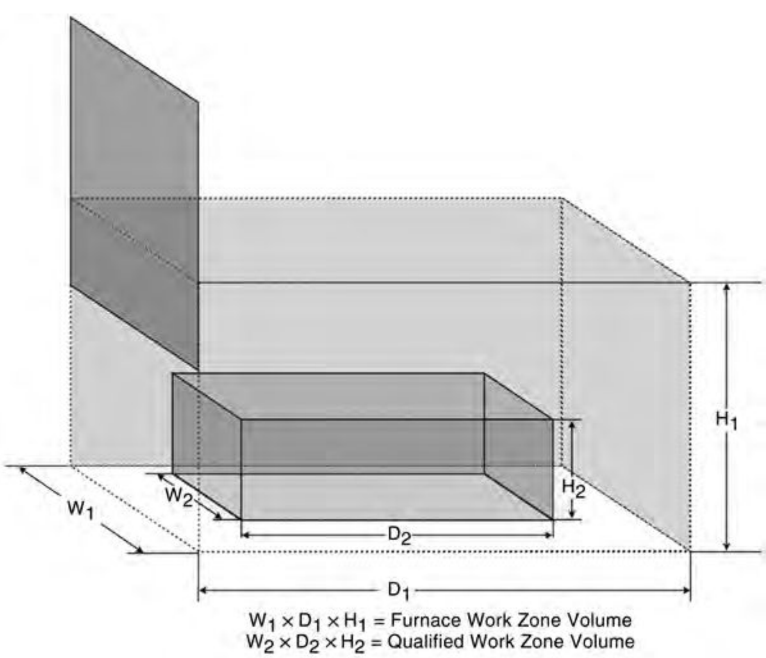

Fig. 5 Illustration of furnace work zone and qualified work zone

For example, if the doors of a continuous furnace are normally open during production, they must be open for the TUS; if slow heat-up rates and stabilization temperatures are not used in production, they must not be used during the TUS; if excess combustion air is used during production, it must also be used during the TUS; if fans are operated during production, they must also be operated during the TUS (AMS2750E).

It should be noted that the use of atmospheres may make it impossible to completely replicate production parameters as potential safety issues, and/or TUS test sensor issues may arise.

The goal is to determine the furnace system's process capability, and the more closely the heat treater can mirror production parameters the more meaningful the data collected will become. Also, once an approach is decided, adherence to this same TUS method for subsequent testing will facilitate the critical evaluation of furnace performance over time.

The analysis of current TUS data should always include a comparison against previously collected TUS data to identify and evaluate any changes that may have taken place. Changes such as longer times to temperature, changes in overall temperature uniformity, or changes in the location of hot spots or cold spots should be duly noted. These variants could be indicative of furnace degradation issues such as worn door seals, breakdown of insulation, burner issues, or any number of other causes.

This historical comparative analysis, however, has significantly less value if the TUS test procedures were not identical from test to test. For this reason careful consideration should be given to the approach taken to ensure that it is efficient, provides meaningful data, and is likely to be followed on a consistent basis.

With a consistent approach to the TUS, the data collected will provide objective evidence as to the furnace system's capability to perform satisfactorily over time, and the TUS will become not only a useful tool but an integral component of an effective furnace maintenance program.

Governing Specifications for Temperature Uniformity Surveys. Specifications such as AMS2750E, CQI-9 3rd ed., ASTM A991/A 991M-08, and many others speak directly to the importance of TUS testing as an integral component of a sound pyrometry maintenance program. These specifications recognize that when TUSs are performed correctly, analysis of the data thereby gathered greatly assists in identifying process capability.

It must be noted, however, that the specific requirements imposed by these specifications can vary significantly. Complicating matters, it is not uncommon for the heat treater to be working with multiple pyrometry specifications, each with its own approach to TUS requirements (Table 3).

For this reason, it is of the utmost importance that the heat treater understand these differences when developing his approach to TUSs. Further, it must be understood that AMS2750E and CQI-9 3rd ed. both represent minimum requirements and any other applicable process specifications with more stringent pyrometry requirements must be followed.

As a general rule, heat treaters supplying the aerospace and defense industries will need to satisfy the requirements of AMS2750E, whereas heat treaters supplying the automotive industry will typically be required to satisfy the requirements of CQI-9 3rd ed.. ASTM A991/A 991M-08, on the other hand, is not specific to any market segment but intended simply to cover TUS testing requirements for any furnace used to heat treat steel products.

Test Thermocouples for Temperature Uniformity Surveys. Temperature data for a TUS is collected by a field test equipment system. This system comprises a data acquisition device connected to the TUS test thermocouples. The data acquisition portion of this system usually remains outside the furnace during the TUS; however, data acquisition devices that reside within the furnace are available.

There are several variables to be considered when selecting the thermocouple type and style to be used during the TUS testing process. Some of these variables include the cost, the desire or need to reuse the TUS test thermocouples, and possible access issues associated with placing the test thermocouples into the furnace work zone.

However, there are two critically important points to be addressed if we are to achieve a high level of confidence in the TUS temperature data being collected: at what temperature will the TUS be conducted, and what is the atmosphere within the furnace to be tested?

Care must be taken to ensure that thermocouple wire size and type are appropriate for their intended use and are not used outside of their recommended upper temperature limit. Guidance on these limitations can be found in current revisions of publications such as ASTM E 230 (Table 4, Ref 15), ASTM MNL 12, and ASTM E 608 (Table 5, Ref 16), or can be acquired directly from the thermocouple supplier.

In addition, the insulation type selected must be appropriate for the proposed TUS test temperature. The selection should also take into account such factors as the need for abrasion or moisture resistance and the potential effect of furnace atmosphere upon the test thermocouples.

The atmosphere present in the furnace during the TUS can have a significant effect upon the performance of our test thermocouples. Type K thermocouples, for example, are subject to failure when exposed to sulfur. Further, a preferential oxidation of chromium in the positive leg of this thermocouple type can take place in low oxygen concentrations, resulting in significant negative calibration drifts.

Even in an air atmosphere there can be adverse effects upon TUS test thermocouples. For example, in a TUS performed at an elevated temperature with ceramic fiber insulated thermocouple wire, in a furnace loaded with steel product, carbon off-gassing could create a bridge between the two legs of the TUS test thermocouples, with the result that unreliable temperature data are collected.

Many of the concerns regarding atmospheres and the resulting potential negative effect on thermocouples can be alleviated by the utilization of non-expendable sheathed-style thermocouples

Table 3 Comparison of different requirements in governing specifications for temperature uniformity surveys

Not intended as a complete list of differences

Topic of requirement	AMS2750E	CQI-9 3rd edition	A 991/A 991M-08
Performance of an initial or primary TUS	Required	Not required	Required (primary)
Performance of a periodic or secondary TUS	Required	Required	Required (secondary)
Specific TUS frequency requirement	Varies per furnace class/instrument type	Annual or quarterly per process tables	Annual
TUS frequency reductions allowed	Yes	No	No
Furnace temperature uniformity requirements	Per furnace class	Per process tables	Per customer product specification
Number of required TUS test temperatures	Varies whether Initial or Periodic TUS and the furnace operating temperature range	One if operating range not greater than 85 °C (155 °F) for aluminum; 170 °C (305 °F) for other metals.	One if span of operating temperature range is not greater than 150 °C (300 °F).
Number of required TUS test points	Follow AMS2750E specific formulas	Follow CQI-9 3rd edition specific formulas	Three for continuous belt furnace with work zone height less than 300 mm (12 in.). Other furnace types as deemed necessary.
Sensor accuracy requirements	± 2.2 °C (±4.0 °F) or ± 0.75% of reading	± 1.1 °C (±2.0 °F) or ± 0.4% of reading	Not specified
Furnace loaded/unloaded	Either	Either	Loaded
Pass/fail criteria	Per AMS2750E specific	Per CQI-9 3rd edition specific	Per customer product specification
Alternative test methods	Allowed per AMS2750E specific requirements	Allowed per CQI-9 3rd edition specific requirements	Allowed per A991/A 991M-08 specific requirements

Table 4 Suggested upper temperature limits for protected thermocouples (ASTM E230-02)

Thermocouple type	Upper temperature limit °C (°F) for various wire sizes (AWG)			
	No. 8 gage	No. 14 gage	No. 20 gage	No. 24 gage
T	...	370 (700)	260 (500)	200 (400)
J	760 (1400)	590 (1100)	480 (900)	370 (700)
K and N	1260 (2300)	1090 (2000)	980 (1800)	870 (1600)

Table 5 Suggested upper temperature limits for sheathed thermocouples (ASTM E608)

Thermocouple type	Upper temperature limit °C (°F) for various sheath diameters			
	1.6 mm (0.062 in.)	3.2 mm (0.125 in.)	4.8 mm (0.188 in.)	6.4 mm (0.250 in.)
T	260 (500)	315 (600)	370 (700)	370 (700)
J	440 (825)	520 (970)	620 (1150)	720 (1330)
K and N	920 (1690)	1070 (1960)	1150 (2100)	1150 (2100)

rather than the lower cost, expendable, fabric-insulated thermocouple wire.

To further help minimize problems related to thermocouples during a TUS, the utilization of Type N versus Type K thermocouples should be considered, as the former are less susceptible to oxidation issues, tend to be more stable overall, and require less consideration regarding insertion depth.

Specifications such as AMS2750E (Ref 17) and CQI-9 3rd (Ref 18) ed. require that TUS test thermocouples be calibrated traceable to the National Institute of Standards and Technology (NIST) or some other equivalent national institute of calibration prior to their first use, and that certain accuracy requirements be met.

These specifications also require that the TUS test thermocouples be calibrated in the temperature range in which they will be used with, calibration intervals not greater than prescribed amounts which can vary from one specification to another.

Calibration reports for the TUS test thermocouples should contain the date of calibration, the source of the calibration data, the nominal test temperature, the actual test temperature readings, the calibration technique used, and the correction factor or deviation amount for each calibration temperature.

Knowing the correction factor or deviation amount at each calibration temperature for both the thermocouple and the TUS recording device will allow the heat treater to determine true temperature values for data collected during the TUS testing process. True temperature data are also referred to as *corrected data*.

If the required TUS test temperatures should fall between calibration points of the test thermocouple or its associated recording device, interpolation of calibration correction factor data is allowed so that the precise correction factor or deviation value to be applied can be determined. It also is important to note that AMS2750E requires rounding be performed in accordance with ASTM E29 or some other national standard (Ref 19).

Each furnace system and furnace system type will present its own set of challenges in the placing of TUS test thermocouples into the furnace work zone. In some instances it will make sense to trail thermocouple wire through the furnace system and connect it to an externally located data acquisition system. However, variables such as furnace doors, particularly interior furnace doors, could preclude the ability to utilize this approach.

When trailing thermocouple wire through the system is not an option, data acquisition devices that can remain in the furnace work zone during the TUS process may provide the solution. When these data acquisition systems are used care must be taken not to exceed their time at temperature limitations or to immerse them in quench baths if they are not specifically designed to withstand such immersion.

Because TUSs are performed on a regularly scheduled basis, it is of considerable importance to develop an approach to the TUS setup that minimizes its time to completion. It should also provide for the critically important need to achieve a consistent placement of the TUS test thermocouples from one test to the next.

For this reason, consideration should be given to the utilization of a test fixture to which the TUS test thermocouples can be attached. This approach helps to ensure a consistent, efficient setup from one test to the next while also ensuring that the qualified work zone dimensions are precisely mapped.

If thermocouples are to be inserted into heat sinks, it is important to note that both AMS2750E and CQI-9 3rd ed. place limitations on their size and also require their material composition be consistent with the thermal characteristics of product typically processed in the furnace under test.

Careful consideration must be given to the decision whether to weld heat sinks to the test fixture. Doing so can effectively result in a heat sink larger than allowed for, and overshoot conditions may be missed or understated.

When wiring the TUS test fixture it is always important to leave sufficient slack in the test thermocouples to allow for contraction. This applies to both expendable thermocouple wire and non-expendable sheath-style thermocouples, particularly when test temperatures reach or exceed 925 °C (1700 °F).

It is essential for the collection of reliable TUS data to select the appropriate thermocouples, have them calibrated in the range of their use, and install them properly and in a consistent location within the furnace work zone.

In the absence of accurate TUS temperature data being collected, the heat treater may be accepting data that appears to meet specified requirements when in fact it does not. On the other hand, the heat treater could erroneously conclude the TUS results do not meet specified requirements and find himself chasing problems that do not really exist.

Use of Atmosphere During Temperature Uniformity Surveys. Although the intent is to mirror production parameters while conducting the TUS, the use of atmospheres is deserving of careful consideration. Not only can certain atmospheres have a negative effect on the TUS test thermocouples, they can also present safety issues.

It should be noted that while the effect of the atmosphere itself upon temperature uniformity is generally negligible, the atmosphere delivery system has the potential to create significant furnace performance issues that may be revealed in the temperature uniformity results.

Although specifications such as AMS2750E and CQI-9 3rd ed. allow for exclusions regarding atmospheres, the use of an inert gas, if possible, or the utilization of sheathed-style TUS test sensors may alleviate the concerns associated with potentially problematic atmospheres.

In any event, it is critically important that safety issues be fully evaluated in deciding the approach to be taken.

Temperature Survey with Loaded or Unloaded Furnace. Some pyrometry specifications require that uniformity surveys be conducted initially with the furnace both loaded and unloaded. Other pyrometry specifications give the heat treater the option of conducting a TUS while the furnace is either loaded or unloaded.

Both AMS2750E and CQI-9 3rd ed. allow the heat treater to perform the TUS with a load or simulated load, with a test rack or empty. AMS2750E, however, requires that once an approach is decided upon, the heat treater must stay with that approach unless he is willing to perform an initial TUS and restart the process of earning reduced-TUS-frequency permissions.

The decision to conduct the TUS loaded or unloaded should be driven by the heat treater's desire for data meaningful to his operation. The question simply becomes: which approach provides information representing the greater value to the heat treater, part temperature or air temperature?

For example, although allowed by both AMS2750E and CQI-9 3rd ed., a TUS performed on an empty continuous belt furnace will provide the heat treater with little information about how well the furnace system responds to a load-related drawdown on temperature. Subsequently, information concerning soak time capabilities when product is run at typical production rates will not be provided.

The same issues apply to batch-type furnaces. In absence of a load, the temperature data collected will not provide any indication of the time required to bring parts to temperature and will do little to help determine whether product recipes are efficient or adequate.

Another detail to be considered is variation in load size. Variations in load configurations can have significant effects upon temperature uniformity characteristics. Although not a specific requirement in AMS2750E or CQI-9 3rd ed., consideration should be given to performing additional TUSs to insure that load size variations are not creating out-of-tolerance temperature uniformity conditions within the furnace qualified work zone.

Throughout this process of developing approaches to be taken when performing TUSs, the heat treater should remain focused not only on compliance with his applicable governing specifications but also with the developing of data that will provide value added benefits pertinent to his operation.

Data Collection and Reporting. Temperature uniformity survey data should be collected at a sample rate sufficient to detect any potential out-of-tolerance conditions. Further, the data collection should begin before any TUS test thermocouple achieves the lower temperature tolerance value so that any TUS test thermocouple exceeding the upper tolerance value can be easily detected.

When a TUS test fixture is being placed into a preheated furnace or into a liquid bath, the data collection should begin as the fixture is being placed into the vessel if at all possible.

It is important to note that the sample rate requirements for data collection during a TUS are not the same in AMS275E and CQI-9 3rd ed.. Generally speaking, AMS275E requires that temperature data from all TUS test thermocouples be collected at a minimum of each two-minute period, , though additional requirements exist in AMS2750E addressing data collection in certain specific situations. In contrast, CQI-9 3rd ed. requires that temperature data from all TUS test thermocouples be collected at a minimum of each two-minute period unless the furnace system is a continuous or semi-continuous type, in which case all TUS test thermocouple data must be recorded at least every thirty seconds.

In addition to the temperature data being collected from the TUS test thermocouples, both AMS2750E and CQI-9 3rd ed. require that the furnace system's process temperature recording device is collecting zone temperature data throughout the duration of the TUS. The provision of this process record as a component of the TUS report is a requirement of both AMS2750E and CQI-9 3rd ed.

Moreover, AMS2750E also requires the process record to include the temperature data generated by all thermocouples defining the furnace system's applicable instrumentation type. This could potentially include the temperature high limit, cold monitoring device, and load thermocouples.

Whether working with AMS2750E or CQI-9 3rd ed., when performing a TUS it is imperative that stabilization of furnace temperature can be identified. Although stabilization is defined differently in AMS2750E and CQI-9 3rd ed., it is essentially that point at which all TUS test thermocouples and all furnace zone process sensors are within tolerance and display a recurrent temperature pattern or cycle. (The pyrometry specification ASTM A991/A 991M-08 does not use the term "stabilization" while addressing TUS requirements, but it does indicate the need to identify a recurrent temperature profile during the testing process.)

Regardless of the governing specification, the requirement is essentially the same: to provide objective evidence that the furnace system has the ability to remain within the required temperature uniformity tolerance.

Once the temperature data have been collected, there are specific requirements governing the information that must be included in the TUS reporting. These reporting requirements can vary from specification to specification so it is critically important that the heat treater understand which specific items must be included in their documentation.

Upon close review it will become obvious that the TUS data required by any of these specifications will greatly assist the heat treater in performing this test in a consistent manner, thereby allowing the greatest value for the resources invested to be achieved.

Alternative Test Methods. In some instances the challenges presented in conducting a TUS simply cannot be overcome and standard TUS methodologies will not work. This does not excuse the heat treater from having to prove the temperature uniformity capabilities of his furnace system.

In this instance both AMS2750E and CQI-9 3rd ed. allow for alternative test methods to help determine the adequacy of the furnace system's temperature uniformity capability. Each of these specifications has a unique approach to alternative test methods and what is acceptable to one may not be to the other.

For instance, in AMS2750E it is permissible to map the qualified work zone volume by repeatedly inserting a single test thermocouple through the furnace walls, hearth or roof until the qualified work zone dimensions are adequately tested.

It is also permissible to perform property surveys whereby the heat treater shall analyze those characteristics that are sensitive to variations in process temperatures and establish a baseline against which regularly scheduled property trends are to be performed.

CQI-9 3rd ed. leaves the approach to the alternative TUS test method up to the heat treater to develop. The proposed method must meet the requirements of the applicable process table, satisfy the intent of CQI-9 3rd ed., and be approved by the customer.

If the heat treater is unable to develop an acceptable alternative TUS test method, CQI-9 3rd ed. also allows property surveys to be performed as a means to provide evidence of thermal processing capabilities.

If the alternative test method is used as an approach to confirm temperature uniformity capability, it is crucial that the heat treater understand fully the requirements of his applicable process or pyrometry specification to ensure that compliance is being maintained.

Furnace Atmospheres

Measurement of various parameters associated with gases in a furnace chamber is of particular importance for controlling many heat-treating processes. When steel is being heat-treated in a controlled atmosphere furnace, there are a maximum of four phases present:

- the gas
- ferrite
- austenite
- cementite

Because pearlite is a lamellar mixture of ferrite and cementite, and ledeburite is the cementite-austenite eutectic mixture, these are not separate phases. If scaling or sooting occurs then there will be five or six phases.

Inside a furnace used for the heat treatment of steel parts the following reactions may occur:

$$Fe + H_2O \rightleftharpoons FeO + H_2 \quad \text{(Eq 1)}$$

$$Fe + CO_2 \rightleftharpoons FeO + CO \quad \text{(Eq 2)}$$

$$CO + H_2O \rightleftharpoons CO_2 + H_2 \quad \text{(Eq 3)}$$

$$CH_4 + CO_2 \rightleftharpoons 2H_2 + 2CO \quad \text{(Eq 4)}$$

$$Fe + CO + H_2 \rightleftharpoons (Fe, C) + H_2O \quad \text{(Eq 5)}$$

$$2CO + Fe \rightleftharpoons (Fe, C) + CO_2 \quad \text{(Eq 6)}$$

$$Fe + CH_4 \rightleftharpoons (Fe, C) + 2H_2 \quad \text{(Eq 7)}$$

$$2CO + O_2 \rightleftharpoons 2CO_2 \quad \text{(Eq 8)}$$

$$CO + 3H_2 \rightleftharpoons CH_4 + H_2O \quad \text{(Eq 9)}$$

Each of these reactions is reversible, depending on the ambient conditions, and its direction is determined by the LeChatelier-Braun principle. These reactions need not all occur at the same time, nor are they all equally desirable or beneficial. Reactions Eq 1 and 2 are indicative of the formulation of scale and, hence, often represent undesirable conditions. Carburizing and carbon restoration reactions involve mainly reactions Eq 5, 6, and 7. Reactions Eq 5 and 6 are the most important ones taking place within a furnace used for neutral hardening.

An analysis of a furnace atmosphere containing eight or ten (or more) constituents and having these connected by five or six equilibrium reactions (any of which may proceed in either direction) can be complex and unwieldy.

Fortunately, the application of Gibbs phase rule determines the number of variables that must be independent in a heterogeneous system. By applying the principles of heterogeneous equilibria, simplifications can be made that enable metallurgists and metallurgical engineers to understand and control furnace atmospheres with a minimum of effort.

For more details on atmosphere control, see article "Furnace Atmosphere Controls in Heat Treating" in this Volume.

Gas Pressure Level. The manual measurement of pressure in a furnace atmosphere is usually done with a water manometer, with levels generally in the range of 0 to 25 mm (0 to 1 in.) in the water column. The reading is actually a differential reading between the inside and outside of the furnace. Inexpensive pressure transducers that produce high-level signals (for example, 4 to 20 mA) for this range are readily available, but care must be used to ensure long-term reliability of this signal in case the tubing to the transducer becomes plugged or partially closed.

Transducers that feature an analog display of the pressure reading as well as the retransmission signal are most useful when the signal is trimmed for calibration. The transducer should have its own zero and span adjustments for maximum flexibility.

Furnaces in which the pressure is constantly varying over a wide range because of opening and closing doors or other upsets will require intelligent signal conditioning that ignores the peaks and valleys in the pressure. Atmosphere pressure is sometimes controlled in a closed loop with an actuator-driven atmosphere effluent damper. This device also helps to keep the atmosphere pressure constant.

Vacuum Level. A typical vacuum furnace will generally require at least two sensors for accurate monitoring of conditions within the furnace. As low oxygen levels are desirable when high vacuums are used, the zirconia oxygen sensor provides a direct measurement of this gas. Measurement of vacuum level has been performed by a variety of methods, but two of the most common have been the thermocouple gage for lower vacuum levels and the cold cathode gage for higher vacuum levels. Another method for measurement down to 13 nPa (10^{-10} torr) is based on a hot filament ionizing the residual gas. All methods except capacitance manometer are subject to error if the composition of the residual gas is different than expected.

Process Gas Flows. Measurement of process gas flows in controlled atmosphere furnaces generated interest recently. There are several methods available, varying widely in cost. The selection of a particular method must be made with extreme care because the cost of gas flow measurement must be kept in balance with the other data acquisition elements in the overall instrumentation plan.

The least expensive method to measure gas flows is with simple flow switch devices. These devices almost always operate on the principle of a pressure drop (and pressure switch) across a fixed orifice in the gas stream. Their limitation is that they can sense only whether flow above a certain fixed amount is present or absent. However, by combining two flow switches in the gas stream set at different points, it is possible to ascertain whether a gas flow is above the high limit, in the desired range, or below the low limit.

Flow switch arrangements are especially suited for gas flows that do not vary (for example, endothermic or exothermic generated gases and nitrogen) or even liquids like methanol. A dual flow switch arrangement is a control setup in which the data acquisition system sounds an alarm whenever the gas flow has dropped out of the desired range, with human intervention required to correct the flow.

When an exact value of gas flow is required, two techniques are available:

- Electronic true mass flowmeters
- Rotameters with electronic adaptors

Each of these may require external power supplies and care in system wiring.

The true mass technique uses the principle of measuring the amount of heat that the gas stream can remove from a heated bulb of controlled temperature. This technique has the advantage of being inherently accurate (if the flowmeter has been calibrated against a known flow) regardless of pressure fluctuations. It has the disadvantage of being a blind technique, with the electronic signal (typically 4 to 20 mA) being the only indication of flow.

The rotameter technique, while slightly more expensive than the true mass technique, has the advantage of having two outputs: a visual one by virtue of the float scale (see Fig. 6), and an electronic one by virtue of electronic position sensing of the same float assembly. The disadvantages of the rotameter technique lie in accuracy and resolution: the measurement is sensitive to temperature and pressure variations (good upstream regulation is required), and the electronic signal may resolve to only ±2.5% of scale. However, this technique is still preferred because of long-term calibration considerations.

Measurement of low liquid flows, such as in nitrogen-methanol systems, is best done with the rotameter system. However, the viscosity of methanol changes quickly with temperature, and for maximum accuracy it is necessary to electronically compensate the temperature readings from a rotameter-type device accordingly.

Control of gas flows is usually performed with an adjustable port valve and motor actuator. The most easily installed and maintained motor actuator is one that accepts an electronic signal (for example, 4 to 20 mA) and positions itself accordingly. Motors that employ a slidewire feedback technique are not always desirable because of long-term maintenance problems.

One problem with motor-actuated control valves is that the flow is not proportional to the value position. For example, it is possible to have signals of 0% open and 100% open do just what is expected, while the 50% open signal gives a measured gas flow of 20% of scale. This phenomenon is due to a variety of factors, including linkage adjustment, pressure drops in the piping, and the inherent nonlinearity of valve ports.

Corrosive gases such as ammonia require stainless steel construction that makes these valves more expensive. Another completely different technique of measuring and controlling gas flow that can be used in many cases is the pulse time-proportioning system. For example, if a simple on-off valve is set up in such a way that when the valve is open there is a fixed, known flow through it, then the average gas flow is always easily calculated by the formula:

$$\text{Average flow} = \frac{\text{Value ON time}}{\text{Value ON time} + \text{Value OFF}} \times \text{Flow when ON}$$

If the ON and OFF time are automatically varied and kept short with respect to the furnace size, this type of arrangement will provide a most cost-effective, easily calibrated system for measuring and controlling gas flows. The power to perform ON and OFF time cycle control is easily found in many of the most powerful process controllers on the market today. In the case of two process gases that must stay in a fixed ratio with each other (for example, nitrogen-methanol), it is possible to design an electronic control system to measure both process gases and control one or both of them to maintain a constant ratio, using the techniques outlined above. The cost of such a system can be very high, but varying methanol flow is a common problem that can significantly affect process results.

Analysis of Gas Composition. The following measurement techniques for the composition

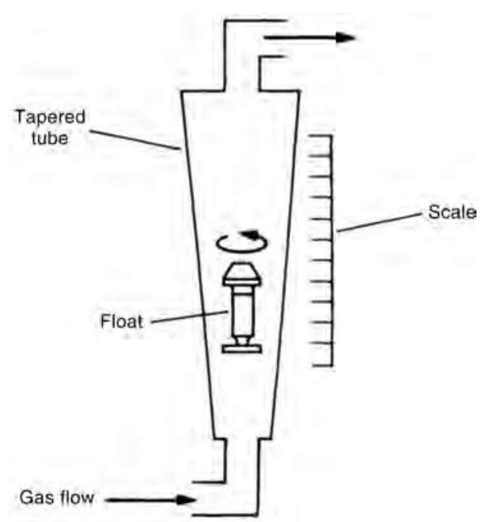

Fig. 6 Schematic showing key visual output components of a rotameter

of furnace atmospheres have successfully been used:

- *Oxygen probe analysis:* This technique is good for measurement of oxygen levels below 0.01%, and with inference can be used to calculate percent carbon potential in a known CO gas, or percent water in a known H_2 gas. It has the advantages of in-situ measurement and good reliability. Frequent calibration is not required.
- *Infrared absorption analysis:* This technique is good for measurement of CO, CO_2, or CH_4 concentration in ranges of interest in hydrocarbon atmospheres. The same unit can be configured to measure all of the above gases simultaneously if desired, which allows accurate calculation of percent carbon potential. The cost is high, with multiple furnaces usually routed to a single analyzer. Frequent calibration is required. Its major disadvantage is that a sample of gas must be transported to the analyzer.
- *Dew point:* If measured by variable pressure change/condensation method, results by inference can be used to calculate percent carbon potential against known carbon and hydrogen gas levels. The disadvantage is that measurements are physically difficult and operator technique and interpretation may play a major role. The sample must be transported to the analyzer. Although there are automatic dew point-measuring systems, none has worked for extended periods in hydrocarbon atmospheres without a significant amount of preventative maintenance.
- *Mass spectrometry:* This technique can determine the composition of gas completely, except for inaccuracies at low levels with some gases. Calibration is difficult. Other disadvantages are cost, the required operator skill level, and the need for sample transport. It is rarely used on-line in heat treatment.
- *Gas chromatography:* See *mass spectrometry*.

While complex gas analysis systems may not have day-to-day process control functions in heat treating, they may be of value in an off-line mode in analyzing trends of shifting gas supplies.

Atmosphere Agitation. The measurement of atmosphere agitation has not yet been successfully performed at the time of this writing, but this certainly would be of interest, as failed atmosphere circulation systems are a common problem. A system based upon a windmill is the most obvious, but other possibilities exist, including measurement of heat flow into a cooled bulb in the atmosphere circulation path. In the not-so-distant future, some such system will almost certainly have to be developed.

Quenching Parameters

Quenching is perhaps the greatest "art" of steel heat-treating operations. Like the heating stage, several variables influence the transformation kinetics during cooling, especially when rapid cooling rates are needed for quench hardening. During the quench, the objective is to cool the part uniformly and with sufficient quench severity for rapid cooling to prevent the formation of pearlite prior to reaching the martensite start (M_s) temperature.

An effective quench severity for a casting is influenced by many factors, as discussed in more detail in the article "Quenching of Steel" *Steel Heat Treating Fundamentals and Processes*, Volume 4A of the *ASM Handbook*. Critical variables include (Ref 3):

- Quenchant type
- Initial quench tank temperature
- Quench tank velocity
- Final quench tank temperature after quenching

The tank volume and quenchant type are critical. Make-up water (to control temperature increases in the tank) and local quenchant velocities also influence the cooling rates. In addition, quenching practice is as important as the quenching equipment. Assessment of transformation kinetics can be difficult to assess from precise recommendations of minimum and maximum quench delays when transferring a basket of complex parts into a quench tank. The quench load size, density, and surface condition also will affect the local heat transfer coefficient at the casting-quenchant interface.

Quenchant Bulk Temperature. The measurement of quenchant bulk temperature is generally the *only* measurement undertaken in most shops. Often this is a monitor-only function, with no feedback control of system heating or cooling involved. From a data acquisition standpoint, it is interesting to look at the bulk temperature during the actual quenching operation, noting any changes. Typically the starting temperature and the highest temperature reached are recorded. In low-agitation quench tanks, care must be taken to ensure that the temperature measured is truly indicative of the bulk temperature.

Quenchant Viscosity. The viscosity of liquid quenchants, especially polymers, is an important indication of composition. In oils, it can be an indication of oil aging or contamination. Viscosity may be measured in-situ, but the results will vary with temperature, making it necessary to compensate for variations in temperature.

Quenchant Media Composition. Measurement of composition (and contamination) of quenching media has become an important area of concern. In the case of quench oils, the parameters of additive levels, alkalinity, oxidation or sludge content, and water content have come to be recognized as being of significant importance. When the costs of these products have increased significantly, much effort has been expended learning how to make a tank of oil last longer through the use of additives and cleaning. Unfortunately, none of the tests required for oil composition determination are suited to real-time data acquisition, except for water content analysis. Also unfortunately, many commercially available water-in-oil analyzers are subject to frequent malfunctions and, though required for safety reasons, are not really suited to the purposes being discussed here.

Solutions of various polymers in water have the common characteristic that the concentration of polymer (which, together with the degree of agitation, determines quenching performance) is usually easily measured with temperature-compensated viscosity sensors, as mentioned above.

Solutions of salts and other additives in water are commonly monitored with specific gravity determination. While this test is easily done with a hydrometer in the lab, it is not so easy to do *in situ* in an automated data acquisition environment.

Quenchant Agitation. Although the primary heat removal rate in a quenching system is generally considered to be a function of the quench medium, there is a significant modifying effect from agitation. Agitation of a quenchant is a parameter that is easily controlled with variable-speed propellers or pumps. However, measurement of this agitation, which may be different from time to time depending on load size and configuration, is another matter.

First, there must be agreement on the units in which agitation is to be measured. Most people would agree that units of velocity (for example, feet per second) or volume (for example, gallons per hour) are logical, but these units do not account for the *pattern* of flow in a given load and how this pattern might change from load to load, with identical velocities.

It is intuitive that measurement of pump or propeller and angular velocity and subsequent linear velocity calculation is not going to be indicative of what is really happening in the quench tank. (What if the propeller motor is still running but the propeller has fallen off, or the flow of quenchant is blocked somewhere else in the system?) It may then be necessary to place a sensor, commonly configured as a tiny free-spinning turbine, in the flow stream directly ahead of or behind the load to directly monitor the quenchant flow rate.

The problems with these systems are twofold. First, the turbine may become clogged with solid contaminants and give a falsely low reading. Secondly, it is often difficult to position the spinner so that it can provide an accurate measurement.

Quenchant Cooling Efficiency. Perhaps the largest area of interest in terms of quenching parameter control is in the measurement of actual cooling performance of a quenchant with a standardized test. In the past few years, a number of quenching evaluation tests have been developed for laboratory use that operate by heating and quenching a probe with an embedded thermocouple into a sample of the quench media under test. The results are often presented as raw cooling curves, or perhaps as plots of instantaneous cooling rate against probe temperature (see Fig. 7). The availability and use of this equipment has led to a push for a standard quenchant evaluation procedure. Unfortunately, the SE tests are all manual and cannot to date be made in-situ by an automated data acquisition system.

Process and Product Capabilities

Process capability studies also are conducted on all types of manufacturing processes to determine the statistical variation of a product with respect to a measured characteristic. *Process capability* is the reproducibility of a process over a long time period with normal changes in workers, material, and other conditions. The wear and tear of constant operation inevitably requires corrective action before out-of-specification parts are produced. As noted, process results also depend on material-related characteristics such as hardenability, material chemistry, and part geometry.

For heat-treating processes, characteristics frequently measured are hardness and case depth. Specifications may initially need to be clarified with regard to the exact test scales or test methods to be used and the critical locations where these tests are to be made before a capability study is conducted.

After the metallurgical requirements are clearly established, a basic process capability study may be conducted. Care should be taken so that the parts tested are from the loading locations representing the extremes in process variability. A good guideline for test sample locations is to use those loading locations prescribed for temperature uniformity surveys in specification MIL-H-6875.

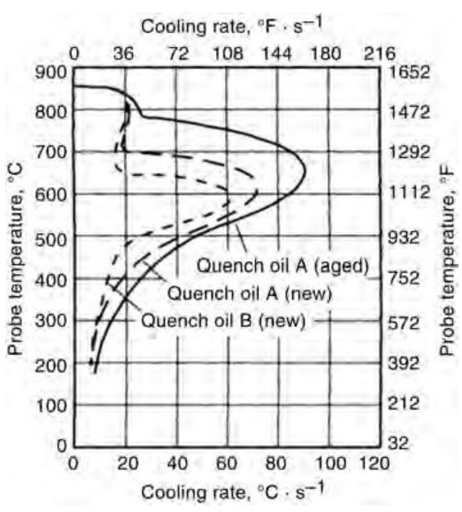

Fig. 7 Probe cooling rate curves for quench oils having approximately the same GM quenchometer and viscosity values

The use of normal probability paper for data representation and plotting is highly recommended. If the data do not plot as a straight line (indicating a normal distribution), a metallurgical or process-related reason for the skewness should be apparent or be determined. For continuous processes, it also is important to collect the samples over a sufficiently long period of time in order to reflect process heating power fluctuations or other process abnormalities that could be time-dependent.

The overall process capability (Fig. 8) is the result of many factors, which may be grouped into four categories and classified as intrinsic or extrinsic sources of undesirable background signals (noise):

- *Base material contributions* (intrinsic noise): These are unique material characteristics, material defects, and hardenability differences. These can vary from lot to lot and also between materials.
- *Part-related contributions* (intrinsic noise): These are part geometry and section-size variations.
- *Process-related contributions* (extrinsic noise): These include temperature uniformity as affected by process control and mass effects, time control, atmosphere control, and cooling method (as determined by uniformity and average severity).
- *Evaluation method contribution* (extrinsic noise): These include standards accuracy and testing method accuracy.

By using properly standardized test coupons as the basis of a process capability study, one can separate out the variability due to intrinsic noise factors and arrive at the inherent process capability. However, extrinsic contributions should be kept in mind. Additionally, a GR&R (gage repeatability and reproducibility) study may be performed on the evaluation method to determine the contribution of these factors to variability.

To successfully use the process capability study as a dynamic tool to refine and narrow process variability, the following three steps should be used in conjunction with process capability studies:

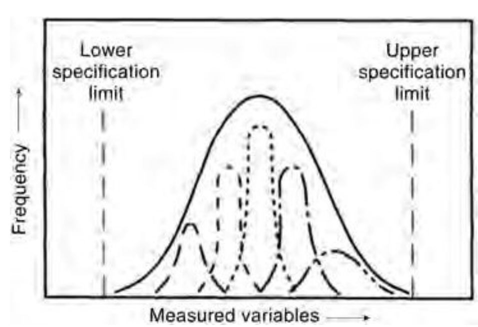

Fig. 8 Factors contributing to overall heat-treating process result variations

- Step No. 1:
  - Identify critical control variables and their relative contributions to process attribute variations (this can be done by process modeling techniques).
  - Measure process inputs with corresponding process output results.
  - Document process control procedures.
- Step No. 2:
  - Modify control procedures, manufacturing procedures, or equipment in order to reduce process variability.
- Step No. 3:
  - Remeasure process capability (as in Step No. 1 above) to ascertain the effectiveness of the changes.

Material Variability. It is obviously important to characterize the incoming product to be processed, so that the controllable incoming material variability can be isolated and corrected independently from the product variations that are due to the process. Before applying statistical control techniques to monitor process or product uniformity, it also is important to understand how the raw material uniformity is controlled prior to heat treatment—that is, whether or not the incoming material is identified and kept separate by heat numbers in the case of steel or by batch numbers in the case of cast materials.

One important material factor is *decarburization*, which occurs to a greater or lesser degree exists on most steels having more than 0.30% C. This defect results from basic steel manufacturing and if not removed in the part manufacturing process prior to heat treatment can influence the surface hardness of parts after induction, flame, or direct hardening processes that may not be capable of correcting the surface decarburization condition. It also should be recognized that many heat-treating processes can also cause this same problem.

Another type of problem is *banding*. Many steels, particularly a resulfurized one such as AISI 1100 or 1200 series, exhibit banding or microalloy segregation. The bands exist prior to heat treatment and the ferrite-rich and pearlite-rich areas run in bands across the longitudinal rolling direction of the bar stock from which parts are made. It has been found that this condition can result in a 4- to 10-point variation in Rockwell C hardness after hardening between these bands of different chemical composition. This problem is greatest when the bands are widest and the heat treatment times are very short, as in induction hardening processes.

For some materials (notably cast irons), hardness test results can vary sensitively with the hardness testing scale used. This sensitivity of hardness value to the testing method and the hardness scale used occurs because the different phases present in the workpiece vary significantly in hardness. This same effect exists in other materials that are heat-treated.

Part-Related Variability. Each part evaluated by statistical means after heat treatment