Heat Treatment and Testing of Carbon and Low Alloy Steel Large Cross Section and Critical Section Components

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Foreword

This recommended practice was formulated by Subcommittee 6, Ad-Hoc Task Group under ISO Standard 10423 on Heat Treatment of Large Cross Section and Critical Section Components. It is a report of the conclusion of a task group study of heat treatment as covered by API Specification 6A, *Specification for Wellhead and Christmas Tree Equipment*.

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Suggested revisions are invited and should be submitted to the Standards Department, API, 1220 L Street, NW, Washington, DC 20005, standards@api.org.

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1 Scope

This recommended practice (RP) may supplement the API equipment specifications for carbon and low alloy steel large cross section and critical components. The recommend practice described herein suggests the requirements for batch-type bath quench and water spray quench-type heat treating practices.

2 Normative References

Standards referenced in this specification may be replaced by other international or national standards that can be shown to meet or exceed the requirements of the referenced standard. Manufacturers who use other standards in lieu of standards referenced herein are responsible for documenting the equivalency of the standards. Referenced standards used by the manufacturer may be either the applicable revision shown in Section 2 and herein or the latest revision. When the latest edition is specified it may be used on issue and shall become mandatory six months from the date of the revision.

API Specification 6A, *Specification for Wellhead and Christmas Tree Equipment*

ASTM A255[1], *Standard Test Methods for Determining Hardenability of Steel*

NACE MR0175 [2]/ISO 15156 [3], *Petroleum and natural gas industries—Materials for use in* H_2S*-containing environments in oil and gas production*

SAE AMS-H-6875 [4], *Heat Treatment of Steel Raw Materials*

3 Terms and Definitions

For the purposes of this document, the following definitions apply.

3.1
critical section components
Any part having a cross section thickness with an equivalent round (ER) that exceeds the depth of hardenability of the alloy selected for the part.

3.2
large cross section
Any part having a cross section thickness with an equivalent round (ER) greater than 5 in. (125 mm).

3.3
prolongation
An extension of a piece of raw material or an extension of a production part made integrally during forging, hot working, cold working or casting for the purpose of performing mechanical testing and metallurgical evaluation.

3.4
QTC
Qualification test coupon.

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1 ASTM International, 100 Barr Harbor Drive, West Conshohocken, Pennsylvania 19428, www.astm.org.
2 NACE International (formerly the National Association of Corrosion Engineers), 1440 South Creek Drive, Houston, Texas 77218-8340, www.nace.org.
3 International Organization for Standardization, 1, ch. de la Voie-Creuse, Case postale 56, CH-1211 Geneva 20, Switzerland, www.iso.org.
4 SAE International, 400 Commonwealth Drive, Warrendale, Pennsylvania 15096-0001, www.sae.org.

3.5
soak time
The time that the entire part (throughout its cross section) is at the specified temperature.

4 Purpose

Heat treatment is a critical process that must be appropriate and controlled in order to produce parts that comply with design requirements. Per API 6A, "The properties exhibited by the QTCs shall represent the properties of the thermal response of the material comprising the production parts it qualifies. Depending upon the hardenability of a given material, the QTC results might not always correspond to the properties of the actual components at all locations throughout their cross section."

The specified mechanical properties may not necessarily be required or achieved through the entire section thickness of the production part(s). These procedures are intended to provide the manufacturer and end user with a means of ensuring that the qualification test coupon (QTC) is more representative of the mechanical properties in a large cross section component than can be expected with a standard API equipment specification QTC. Furthermore, these procedures are intended to provide to optimize the heat treatment and heat treatment response of large cross section components, thereby insuring that the component has the required mechanical properties at the depth below the surface established by the manufacture at all critical locations.

It should be noted that the required mechanical properties as established by the manufacturer may be different from the mechanical properties required by the API equipment specification.

This recommended practice is intended to supplement the heat treatment and testing requirements found in the API equipment specification and not to replace them altogether.

5 Application

This recommended practice is intended for use on large cross section components being manufactured for conformance to API equipment specifications.

6 Recommended Heat Treating Practices

6.1 General

Heat treating may be defined as the controlled heating and cooling of a metal in order to obtain a desired microstructure and consequently desired properties. Carbon and low alloy steels are the most widely used alloys in oil and gas exploration and production. One of the reasons for this is their versatility: a wide range of properties can be obtained through an appropriate heat treatment. The basis for heat treating carbon and low alloy steels is that they have several different stable crystal structures depending on the heat treatment process used. By transforming the crystal structures during heat treatment, the desired microstructure and mechanical properties can be obtained in the end product, provided the size of the product does not exceed the hardenability limits of the alloy.

The most common type of heat treatment imposed on carbon and low alloy steels is a three- to four-step process consisting of austenitizing, quenching, and tempering (Q&T) or normalizing, austenitizing, quenching, and tempering (N-Q&T). The austenitizing cycle consists of heating the steel up to a temperature high enough to completely transform its microstructure into austenite (typically about 1500 °F to 1700 °F or 816 °C to 927 °C for most common low alloy steels). Austenite is a phase of steel having a face-centered cubic structure.

The quenching cycle consists of removing the steel from the furnace and rapidly cooling it in a suitable liquid quench medium such as water, polymer, or oil before the temperature of any section of the component falls below the upper critical temperature. Ideally, the austenite transforms into a structure known as martensite during the quench and greatly harden the steel. Martensite generally has high strength but very low ductility, toughness, and resistance to brittle fracture. Austenite transforms into martensite only if a certain critical cooling rate is achieved. Slower rates result in other softer transformation products such as bainite, pearlite, and ferrite (in descending order of hardness). The actual cooling rate required to produce martensite is dependent on the alloying content of the steel.

The final stage of heat treating carbon and low alloy steel is tempering. This consists of reheating the steel to an elevated temperature, but below where it would again transform into austenite, and letting it soften. This lowers the strength but greatly increases the ductility and toughness of the steel. Tempered martensite exhibits the best combination of mechanical properties (hardness, strength, ductility, toughness, fatigue, etc.) of any of the transformation hardening microstructures.

As indicated above, in some cases an additional operation is utilized prior to the austenitizing cycle. This process is called normalizing. Normalizing consists of heating uniformly to temperature at least 100 °F (56 °C) above the critical range and cooling in still air to ambient temperature. The treatment produces a recrystallization and gives refinement and uniformity to the grain structure. The redistribution of the elements that occurs during normalizing produces a microstructure that responds to heat treatment in a more uniform manner. For many low alloy steels, normalizing prior to austenitizing can improve the toughness of the material and reduce the tendency toward a banded structure.

The mechanical properties of carbon or low alloy steel are dependent on the type, relative amounts, and distribution of the various microstructural components that form in response to a heat treatment. The surface of a part always heats up or cools down at a faster rate than the center.

Thus, some variation in microstructures and properties can be expected within the same part, particularly if there is variation in section thickness. The variation in microstructure can be reduced and the desired microstructure obtained by selecting an alloy grade with appropriate hardenability and performing rough machining to near net shape prior to heat treatment.

Heat treating is the controlled heating and cooling of a metal to obtain a desired microstructure. Good heat treat practice involves having the proper equipment and procedures in place to ensure that the necessary control is maintained.

6.2 Requirements for Heat Treating Equipment

6.2.1 Requirements for Heat Treat Furnaces

Furnaces must be adequately sized for the load to be heat treated. The load must fit entirely within the calibrated working zone. The furnace must be capable of bringing the load up to temperature within a reasonable time period. The furnace must be adequately insulated to prevent heat loss and maintain temperature uniformity. Electric furnaces should have some mechanical means of circulating the air during heating. Furnaces shall have automatic temperature indicating, controlling, and recording devices.

The controlling and recording instruments used for heat treating shall posses an accuracy of ±1 % of their full scale range.

Furnaces shall be properly calibrated no less than once a year to an internationally recognized standard such as SAE AMS-H-6875 or API 6A. Furnaces shall be capable of maintaining a uniform temperature within the working zone of ±25 °F (±14 °C) of the set point temperature for austenitizing and normalizing and ±15 °F (±8 °C) of the set point temperature for tempering.

Temperature controlling and recording instruments shall be calibrated at least once every three months. Thermocouples also shall be calibrated or replaced at least once every three months.

Equipment used to calibrate production equipment shall have an accuracy of ±0.25 % of full scale range and shall be traceable to an industry recognized industry standard such as the National Institute for Standards and Technology (NIST).

6.2.2 Requirements for Quenching Facilities

Quench tanks shall be located in close proximity to the austenitizing furnace and be easily accessible. This minimizes transfer time and heat loss of the load during the transfer. Transfer time from furnace to the quench tank should be as quick as possible but no more than 90 seconds. The transfer time shall be measured from the time the furnace door is fully open or the furnace roof is fully removed and until the component(s) is completely submerged into the quench bath. In cases where the 90-second transfer time is not achievable, objective evidence shall be provided to demonstrate that the material meets material specification properties.

Quench tanks shall be adequately sized for the loads. In the case of water quenching, the volume of water quench tanks shall be such that the temperature of the water does not exceed 100 °F (40 °C) at the start of the quench and does not exceed 120 °F (50 °C) at the end of the quench. This may require the use of supplemental heat exchangers or chillers. As a general guideline, quench tanks should have approximately one gallon of quench media for every pound of load being quenched.

Proper agitation is critical. Quench tanks should have some means (propellers, pumps, etc.) of circulating the quench media to optimize the cooling rate. In the case of water quenching, agitation must be sufficient to break up the steam blanket that forms at the surfaces of the hot immersed part. The steam blanket acts as an insulator and greatly reduce the cooling rate. A quench tank with proper agitation has a noticeable rise in the level of quench medium in the quench tank when the agitators are turned on. Agitators shall be placed so that circulation is maintained throughout the quench tank when a load is being quenched. A quench tank with a single pump located at one end, for example, may not be acceptable because part of the load would be shielded from the quench media flow. Air agitation is unacceptable.

When oil quenching is performed, only oil formulated by the quench oil manufacturer specifically for heat treat quenching operations shall be used. Additionally, oil quench media shall be maintained within the manufacturers' recommended temperature range. These requirements are necessary to minimize the possibility of oil quench tank fires.

Polymer quench media shall be maintained within the manufacturers' recommended temperature range, and the concentration of the polymer shall be routinely monitored and adjusted as necessary.

Spray quench facilities shall consist of one or more high pressure and high volume spray quench rings. These spray quench facilities are normally used for quenching cylindrical cross section parts such as bar and tubing. Essential parameters such as nozzle size, jet spacing, standoff, flow rate of quench medium, and traverse rate of component shall be quantified and controlled.

Quench baths shall permit complete immersion of parts, shall provide for agitation of the quench medium of the parts, shall be of sufficient volume to absorb the heat rejected by the most massive part to be quenched, and shall have a temperature indicator with a sensor in the quench media. Quenching baths shall be free from visible contamination that could detrimentally affect the quenching process. Bath maintenance programs should be established. A system check shall be made prior to production use to ensure the adequacy the agitation system and that the system is designed to minimize susceptibility to agitation variation. When using polymers, a concentration control system shall be established prior to production use.

Fixtures, jigs, hangers, trays, snorkels, etc. shall be employed as needed for proper handling of parts. Fixtures and fixture materials shall not cause contamination of parts and shall not reduce the heating, cooling, or quenching rates to less than that required for adequate hardening of the parts.

Equipment shall be provided to clean parts before heat treatment and to remove oil from parts quenched in oil baths and salt residue from parts heated or quenched in salt baths. When using polymer quenchants, a rinsing system shall be in place to remove quenchant residue from the parts.

6.3 Recommendations for Heat Treatment Procedures and Practices

6.3.1 Specifying Heat Treatment Parameters

The manufacturer shall provide the following technical information to the heat treat facility:

— material grade;

— ladle analysis or product chemical analysis;

— description of the parts to be heat treated including the quantity;

— QTC requirements (see 6.4);

— qualification testing and acceptance criteria (e.g. mechanical properties, metallurgical requirements, and test method specifications);

— any restrictions on specific furnace size or type used, when applicable;

— heat treating times and temperatures for all cycles;

— quenching medium, including start and finish temperature limits for water;

— allowable methods of determining time at temperature for each cycle;

— allowable reheat treatment provisions for nonconforming material;

— hardness test method, locations, frequency, and acceptance criteria;

— any special requirements;

— certification and records requirements.

6.3.2 Rough Machining Practices

Machining prior to heat treat should be considered to minimize the stock remaining on parts made from carbon and low alloy steels with relatively low hardenability.

Parts requiring rough machining prior to heat treatment shall have sharp corners radiused or chamfered prior to the austenitize and quench operation. Such radiused or chamfered corners help to prevent quench cracking in these areas.

There should be generous radii on all corners of parts being heat treated to prevent quench cracking. A $^1/_8$-in. (3-mm) radius is the minimum, but $^1/_4$ in. (6 mm) or larger is recommended.

Rough machining to within $^1/_8$ in. (3 mm) to $^1/_2$ in. (13 mm) per side of major finished dimensions prior to heat treatment is recommended. Additional material may need to be left on the component for the following reasons:

— to prevent quench cracking;

— to allow for the removal of surface scale and decarburization;

— to allow for the removal of surface imperfections;

— to allow for the distortion of part geometry;

— to ensure cleanup to the finished dimensions during final machining.

In some cases the placement or rough machining of internal through bores and/or internal part configuration bears on the selection of the appropriate alloy. Since the through bores and internal configuration substantially reduce the section thickness, the hardenability of the alloy selected may be less than that required for the same part without the through bores.

Contiguous thickness variations should be minimized to help prevent quench cracking. Larger section thicknesses quench at a much different rate than do the smaller sections and create a potential for contractual stress cracking between the sections during the quench. A generous radius should be left between these section thicknesses prior to heat treatment.

6.3.3 Furnace Loading Practices

To ensure that all parts are evenly heated and quenched, provide sufficient part spacing within the working zone of the furnace. Do not stack or bundle parts. Fixtures may be required.

Parts should not be placed directly on the furnace hearth (floor). Use a metal tray or fixture that allows the furnace atmosphere to circulate around and under the part. The refractory on the furnace floor is a large heat sink that may result in uneven heating of the part.

Support long parts as needed to prevent sagging during heat treating. Sagging may occur especially during the austenitizing cycle.

Consider using heat treating fixtures for parts with complex geometries to prevent distortion during heat treating.

6.3.4 Specification of Normalizing Temperature

Recommended normalizing temperature ranges for low alloy steels are available in many heat treating handbooks. In general, a temperature at least 100 °F (56 °C) above the critical range is chosen. This is followed by cooling in still air to room temperature.

6.3.5 Specification of Austenitizing Temperatures

Recommended austenitizing temperature ranges for low alloy steels are available in many heat treating handbooks. In general the minimum temperature is selected based on the minimum temperature at which the transformation into austenite is complete plus an added safety factor, often in the range of 50 °F to 100 °F (28 °C to 56 °C). The upper limit is based upon giving the heat treater a practical range to work to but limiting the temperature to minimize excessive grain growth.

6.3.6 Specification of Tempering Temperatures

Recommended tempering temperature ranges for a prescribed set of mechanical properties in a given carbon or low alloy steel are also readily available in many heat treating handbooks. The minimum temperature may have to be adjusted in order to comply with some standards such as NACE MR0175/ ISO 15156 or, if possible, to ensure that the tempering temperature is well above any weld stress relieving that may subsequently be performed on the part. The maximum temperature is kept well below the temperature where reaustinetizing may occur. It may be adjusted to ensure the end hardness and strength of the heat treated part is on the high side. The range should be wide enough to allow the heat treater some leeway in determining the actual temperatures to be used on specific parts with specific compositions. Care should be taken to avoid any tempering temperatures that may result in the formation of deleterious phases that could cause embrittlement or increase susceptibility to some forms of corrosion. After the tempering cycle, parts are usually cooled in air. Sometimes it may be appropriate to liquid quench certain steels from the tempering temperature to avoid slow cooling through temperature ranges that may lead to various forms of temper embrittlement.

6.3.7 Specification of Heat Treatment Time at Temperature

Soak time is the time that the entire part (throughout its cross section) is at the specified normalizing, austenitizing, or tempering temperature. The actual furnace times are considerably longer to allow the parts to be heated up to the specified temperature. The austenitizing time should be sufficient to allow the complete transformation to austenite and to dissolve any undesirable phases but not excessively long in order to prevent excessive grain growth or excessive decarburization of the surface. There are several factors that can influence the furnace and soak times for heat treat cycles such as the specific alloy, temperature monitoring methods, required material properties, and material configuration.

When the furnace atmosphere thermocouple monitoring method is used to monitor time at temperature, the furnace time should be specified as opposed to soak time. The furnace time is dependent on the cross section of the material being heat treated, and times are typically around $^1/_2$ hour to 1 hour per inch of cross section. Heat-up times vary based on convective and radiant conditions of the furnace heating environment and need to be considered when determining time at temperature.

When the attached thermocouple monitoring or heat sink thermocouple monitoring methods are used to monitor temperature, the soak time should be specified instead of the furnace time. When using these temperature-monitoring methods, the soak time specified is not a function of the cross section, and times are typically $^1/_2$-hour to 1-hour minimum.

6.3.8 Specification of Heat Treatment Temperature Monitoring Method

Having the part being heat treated at the specified austenitizing and tempering temperatures for a sufficient period of time is critical to obtaining the desired properties throughout the entire cross section of the part. Thus determining when a part first reaches the prescribed temperature is important. The actual method utilized must take into account the fact that heavier cross section parts take longer to heat up. The three common methods of determining time at temperature are as follows.

— *Furnace Atmosphere Thermocouple Monitoring*—In this method, a thermocouple, generally suspended from the ceiling of the furnace, is used to monitor when the furnace atmosphere reaches the desired set point temperature. The total furnace time after the furnace atmosphere recovers to the set point temperature is calculated based upon on the heaviest cross section of any part in the furnace load. SAE AMS-H-6875 provides suggested hold times that can be utilized as a reference.

— *Attached Thermocouple Monitoring*—In this method, a thermocouple is attached to the surface of the heaviest cross section of the largest part in a heat treatment load. The specified normalizing (if required), austenitizing, or tempering time at temperature begins when the surface thermocouple reaches the desired set point temperature. In some cases a drop thermocouple is utilized. This is a thermocouple attached to the ceiling of the furnace that can be lowered until it physically comes into contact with the part.

— *Heat Sink Thermocouple Monitoring*—In this method, a thermocouple is imbedded in a separate block of material (made from the same general type of material as the parts being heat treated, e.g. carbon/low alloy steel). The temperature sensing tip of the thermocouple must be at least 1 in. below the nearest surface. The size of the heat sink should be equal to or greater than that of the heaviest cross section of the parts being heat treated. The specified normalizing (if required), austenitizing, or tempering time at temperature begins when the thermocouple reaches the desired set point temperature. An acceptable alternative to a separate heat sink is to imbed the temperature sensing tip of the thermocouple at least 1 in. below the surface into one of the production parts' heaviest cross section. Obviously this small hole must not be in a location that would interfere with the use of the finished part.

6.3.9 Specification of Quench Medium

A quench medium should be chosen that gives the fastest possible cooling rate without causing quench cracking. Recommended quench media for given carbon and low alloy steels can be found in any standard heat treating handbook. Any deviation from the commonly recommended media should be considered very carefully before it is permitted.

There may be times when a slower quench medium is desired in order to minimize distortion or the likelihood of quench cracking because of part geometry. The effects of using a slower quenchant on the end properties must be considered. Although slower quenchant reduces the likelihood of cracking, it also results in lower mechanical properties across the section thickness for a given alloy.

When liquid quenching is required, oil, water, or a polymer/water solution may be used as specified for the alloy and temper indicated. The consistency of quench effectiveness shall be determined for each tank by testing initially and periodically. The heat treating facility shall establish control limits for each quenching system.

Problems, such as cracking and high residual stress, due to an inappropriate quenchant or improperly designed system that is not suitable for a particular alloy and configuration shall be avoided. Because of wide variations in quenching characteristics of different quenchants in different quenching systems, a quenchant validation procedure shall be implemented when initially establishing the quenching procedure or when changing from one quenchant to another.

When substituting a polymer quenchant for an existing oil quenchant, the quenchant validation procedures shall ensure that the polymer and concentration being substituted achieves cooling characteristics that are similar to the existing oil quenchant and that the properties being produced are equivalent to those for oil quenched parts.

In addition to the specification of proper quenching practices, the vendor’s general material handling and quenching practices can affect component properties. For example,

— minimize the transfer time and the loss of part temperature from the austenitizing furnace to the quench tank;

— parts with blind cavities or long bores should not be quenched with the cavity opening or bore oriented downwards so that steam may become trapped;

— consider the use of a water lance to supplement and enhance the quench in the bore of a long part;

— in some applications where the component is long, heavy walled with a small bore, vertical quenching may need to be used;

— do not delay putting quenched parts into the tempering furnace—spontaneous cracking can occur on highly stressed, quenched parts. All parts should be tempered within 24 hours of the quenching operation.

6.3.10 Hardness Testing Practices

The manufacturer shall inform the heat treater of the maximum or minimum hardness or the hardness range and specify where the hardness tests should be taken as well as the frequency of hardness tests.

Consider giving the heat treater a drawing identifying the most critical areas in the part so the heat treater can try to optimize the heat treatment response of these areas.

Specify the largest possible hardness range allowable on a difficult to heat treat part. This is typically determined by the manufacturer’s material specification.

The manufacturer should not always specify a minimum hardness based upon the minimum hardness required at the most highly stressed portion of the part. If only certain areas of a part require a high hardness, then the manufacturer should consider providing a drawing that gives different acceptable hardness ranges for the different part locations.

6.3.11 Qualification of Heat Treating Suppliers

Heat treatment shall be performed by suppliers who are qualified and approved by the manufacturer through the performance of a technical audit. The technical audit is designed to assess the capability and proficiency of the heat treat provider.

6.4 Recommendations for QTCs

The standard API 6A 5-in. (125-mm) equivalent round QTC with specimens taken at the $T/4$ location may be sufficient to qualify the heat treatment of the material. Where the design requires mechanical properties for cross sections and depths greater than that provided by a 5-in. (125-mm) QTC, a larger size QTC may be required to qualify the heat treatment. The hardenability of the material selected should be sufficient to meet the design requirements. One method of determining the relative hardenability of carbon and low alloy steels is to utilize the ideal diameter (DI) method in ASTM A255.

Prolongations on forgings, bars, tubulars, castings, and other products may be used for the qualification of the heat treatment. The prolongation shall represent the thickness of the manufacturer-defined critical section or the thickest section of the part. The location of the test specimens shall be within the $^1/_4T$ envelope or the manufacturer's specification. Additionally, in some instances, sacrificial production components may need to be used to accurately assess the mechanical properties achieved during heat treatment. Alternatively, comparison of test results from a separate QTC versus the test results from a sacrificial part or prolongation may be performed to justify the use of a separate QTC.

At a minimum, one QTC shall be used for each heat per each heat treat batch.

The separate QTC shall accompany the component it represents through all specified heat treatment and quench cycles. Placement of the QTC in the heat treatment furnace in relation to the materials to be heat treated shall be considered because of the aforementioned difference in thermal response between the QTC and larger cross section material to be heat treated.

7 Design Consideration and Material Selection Requirements

If minimum mechanical properties are required throughout the entire section thickness of the part, the alloy selected shall have the capability to develop the required mechanical properties through the part section thickness.

The selection of the appropriate alloy shall be made on the basis of the geometric configuration of the part and the hardenability of the alloy.

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