SALT BATH QUENCHING

The modern molten salt bath is the ideal medium for low-distortion, interrupted quenching processes such as martempering and austempering.

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Molten salts have been used for quenching for more than 50 years. Their wide operating temperature range makes them ideal for many quenching processes aimed at minimizing distortion of iron and steel parts. Their unique characteristics coupled with recent advances in salt quality, pollution abatement, and material handling make salt bath quenching more efficient and economical than ever before.

This article provides an overview of today's salt quenching systems, including their safety and environmental aspects. It also covers the use of salt bath quenching in low-distortion, interrupted quenching processes such as martempering and austempering.

Quenching and distortion
In metal quenching, a part is cooled from the austenitizing temperature fast enough to avoid the pearlite nose of the material's time-temperature-transformation (TTT) diagram and to transform the austenite into martensite to the maximum extent possible. When the quenching medium is water, brine, a polymer solution, or fast oil, it is generally referred to as conventional quenching. Steels with low hardenability can be quenched this way. However, this method can cause distortion and in some cases, nonuniform hardness or even cracking. The tendency to distort increases with increasing steel hardenability.

Causes of distortion usually can be traced to uneven or nonuniform quenching, thermal stresses, and transformational stresses. These factors often can be mitigated by adopting an interrupted quenching technique.

Distortion control: In interrupted quenching, parts are cooled rapidly from the austenitizing temperature to a point above the martensite start temperature ($M_s$), where they are held for a specified time and then cooled to room temperature. Thermal stresses and the potential for distortion are considerably reduced during the hold above $M_s$. Processes that make use of an interrupted quench include martempering, austempering, and variations on them.

For most steels and alloys, the temperature at which the quench is interrupted is usually in the 175 to 370°C

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Reprinted from the December 1999 issue of Advanced Materials & Processes

Fig. 1 — A continuous, mesh-belt-type, salt bath quenching system designed and built by Can-Eng Furnaces Ltd., Niagara Falls, Ontario. It can be used for martempering and austempering.
(350 to 700°F) range. Water, brine, polymer solutions, and most quench oils cannot be used at these temperatures. Attempts at using molten lead and fluidized beds do not appear to have met with much success. Some oils can be used at temperatures up to 230°C (450°F), but for higher temperatures, molten salt is the natural, practical choice. Examples of salt bath quenching systems for interrupted quenching applications are shown in Fig. 1 and Fig. 6.

Advantages of salt over oil

The most distinct advantage of salt over oil is its wide operating temperature range—150 to 595°C (300 to 1100°F) for a typical composition. Thus, salt can be used for any interrupted quenching process. Oil, however, cannot be used above 230°C (450°F), which restricts its use to low-temperature processes.

The quenching mechanism also is considerably different. Most of the heat extracted during salt quenching is by convection (the third stage of liquid cooling), and is therefore at a uniform rate. In oil quenching, heat is extracted during all three stages with varying rates of heat removal. As a result, salt quenching causes less distortion and produces more uniform and consistent hardness.

Other important advantages of salt over oil include:

- Quench severity can be controlled to a greater degree by varying temperature, agitation, and water content of the salt bath.

- Productivity is higher because parts attain temperature equalization faster.

- The excellent thermal and chemical stability of salt means that the only replenishment required is due to dragout losses. A salt bath provides satisfactory quenching performance for many years. In contrast, oil deteriorates with use, requiring closer control and sometimes partial or complete replacement.

- Nonflammable salt poses no fire hazard, whereas oil at a comparable temperature poses a serious hazard.

- Salt can be easily washed off with water and recovered for reuse, if desired. Choosing to recover salt not only eliminates disposal but also reduces operating costs. In contrast, washing of oil requires special cleaners and equipment; and its recovery is not simple.

Limitations: There are relatively few limitations to salt as a quenching medium. It has to be used above its melting point of about 150°C (300°F). And, because it is a strong oxidizer, combustible or incompatible materials should definitely be kept out of the salt bath to avoid the possibility of violent reactions.

Salt may appear to present safety and environmental problems, but the technology for dealing with them is well developed and they are no longer viewed as a deterrent to its use.

Quench severity

Quench severity refers to the ability of a quenchant or quenching system to extract heat from a test specimen, part, or workload. It can be determined by measuring either its hardening power or cooling power. Methods based on hardening power such as the Grossmann technique and Jominy end-quench test are time-consuming. More preferable are methods based on cooling power such as the GM nickel ball, hot wire, and cooling curve tests. Among them, cooling curve (and cooling rate curve) analysis has emerged as the most useful tool for measuring quench severity, as well as for understanding the quenching mechanism and studying the effects of quench variables.

In a salt bath, the cooling rate is nearly constant throughout the temperature range of interest (Fig. 2), confirming that quenching in molten salt occurs at a uniform rate.

Variables in salt quenching

Temperature, agitation, water content, and residence time are the main variables in salt bath quenching.

Temperature: In general, the lower the bath temperature, the faster the cooling rate for any medium. In salt bath quenching, the effect of this variable is generally marginal. However, in some cases, salt bath temperature can be manipulated to achieve remarkable results. For example, low-hardenability steels can be austenitized using two salt baths, each at a different temperature. Similarly, parts having thick sections can be martempered by quenching them in water or brine for a short time and then transferring them to a salt bath. These methods are discussed later.

Agitation: The effect of this variable is greater than that of temperature. Increasing agitation results in a considerable increase in quench severity, Fig. 3(a). Like temperature, agitation can be controlled to advantage. This also is addressed later.

Agitation can be provided by propeller-type agitators or by a pump with a draft tube. Depending on the size of the bath, one, two, or four propellers with single or dual impellers may be required. If a pump is used, salt flow from the bottom to the top of the bath is preferred. Agitation is a must when water additions are to be made.
Water content: A small addition of water to a salt bath increases its quench severity significantly, Fig. 3(b). Water content of the bath typically varies from 0.5 to 2%, usually in the operating temperature range of 150 to 290°C (300 to 550°F). Note that the combination of agitation and a water addition increases quench severity three-fold, compared with an unagitated, "dry" salt bath. Safety considerations dictate that water is always added to a well-agitated bath and never to an unagitated bath.

Instead of fresh water, salt solution from the washing operation can be used. Some heat treaters also use low-pressure steam instead of water. A fringe benefit of a water addition is that it lowers the salt’s melting point, increasing its working range. For example, 1% water lowers the melting point by 11°C (20°F) and 2% lowers it by 19°C (35°F).

Residence time: The time that parts remain in the bath generally depends on steel composition, section thickness, quench severity, and the process being performed. In martempering, a few minutes are enough for temperature equalization. A longer residence time may produce a microstructure other than the desired martensite. In austempering, on the other hand, a few hours may be needed to complete the transformation to bainite. A longer time is not harmful, but it will increase processing costs.

Other factors: Successful salt bath quenching also requires that steel composition, austenitizing temperature, and the section thickness and configuration of parts be considered. Note, however, that these are not strictly quenching variables.

When parts are quenched from an atmosphere furnace into a salt bath, splashing of salt into the furnace is prevented by use of a separating curtain (salt curtain) or an intermediate chamber or vestibule.

Selecting quenching salts
Quenching salt is a eutectic mixture of nitrates and nitrites of sodium and potassium. There are nearly a dozen compositions available, ranging in melting point from 135 to 260°C (275 to 500°F). Selection of a quenching salt primarily depends on the lowest temperature at which it is going to be operated and its melting point. The difference between the two should preferably be 55°C (100°F) or greater. Once molten, these salts all exhibit nearly the same physical properties (table).

Quenching salts used to be available only in granular or crystalline form, which caused dusting when added to a quenching bath, and frothing and scum for a few hours or a day. This adversely affected quenching severity until the bath stabilized. These problems can now be avoided to a great extent by using improved quality salt in briquette or flake form.

Interrupted quenching methods
As previously mentioned, the unique features of a salt bath quenching system make it the most effective and economical method for interrupted quenching processes. These processes not only minimize distortion, but also help achieve certain mechanical properties not easily obtainable using other methods. There are nearly a dozen such processes. Brief descriptions follow.

Martempering and variations
In the martempering interrupted quenching method, Fig. 4(b), the part is immersed in a salt bath maintained at a temperature slightly above Ms and held there long enough for the temperature to equalize throughout its cross section. The part is then removed from the bath and cooled in air to room temperature, allowing austenite to transform to martensite. The thermal gradient between the part's surface and center is much less in martempering than in conventional quenching, Fig. 4(a). This reduces both thermal and transformational stresses, which helps minimize distortion and susceptibility to cracking. As for con-

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### Physical properties of quenching salts

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Specific gravity</td>
<td>1.84–1.92</td>
</tr>
<tr>
<td>Specific heat</td>
<td>0.35–0.40</td>
</tr>
<tr>
<td>Thermal conductivity</td>
<td>0.571 W/m·K (0.33 Btu·ft/h·ft²·°F)</td>
</tr>
<tr>
<td>Heat transfer coefficient</td>
<td>4.5–16.5 kW/m²·K (800–2900 Btu/h·ft²·°F)</td>
</tr>
<tr>
<td>Dragout rate</td>
<td>50–100 g/m² (1–2 lb/100 ft²)</td>
</tr>
</tbody>
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Fig. 3 — Effect of agitation (a) and water content (b) on the cooling rate (quench severity) of a low-melting-point salt at 175°C (350°F). Cooling rate is the average of the rates determined at every 55°C (100°F) interval between 650 and 260°C (1200 and 500°F). This range covers the pearlite nose of the time-temperature-transformation (TTT) diagram of most steels. (Ref. 5)
ventionally quenched parts, martempered parts also must be tempered.

Martempering usually is performed at 175 to 260°C (350 to 500°F), depending on steel composition. Duration of the treatment is just a few minutes, depending on section thickness and the bath’s quench severity. Materials suitable for martempering include carbon steels, low-alloy steels, and gray cast irons.

**Carbomartempering:**
Ferrous metals with low carbon contents also can be martempered, but only after the parts have been carburized or carburized and nitrided. Carbomartempering combines the benefits of both processes. Carbomartempered parts are tougher than conventionally carburized and hardened parts. The greatest advantage, however, is minimum distortion, which means less need to build up and less to grind after hardening. Some components experience so little distortion that they can be assembled without any postheat treat grinding or straightening operation.

If parts are carburized in a gas-fired furnace, they can be quenched directly into a salt bath. If they are carburized in a cyanide salt bath, however, they must first pass through a neutral salt bath to avoid any violent reactions that could result in an explosion.

The carbomartempering temperature is a compromise between the $M_s$ of the case and that of the core, and is chosen so as not to jeopardize case hardness. It is generally in the 175 to 260°C (350 to 500°F) range, and the duration is usually just a few minutes.

**Modified martempering:** This variation, Fig. 4(c), differs in that the temperature of the quench bath is somewhat below $M_s$, usually in the 150 to 175°C (300 to 350°F) range. This increases quench severity. The process can be used for low-hardenability steels, when increasing agitation and water additions are not enough to avoid the pearlite nose of the TTT diagram. It is, therefore, applicable to more steels than conventional martempering.

**Other variations:** Martempering with two baths is used to increase the depth of hardening in thick sections. The part is first quenched in water or brine for a very short time and then transferred into a martempering bath. One application: hardening AISI 52100 steel bearing balls.

Another variation combines martempering with straightening. Some parts require straightening or re-forming after heat treatment, but the operation is sometimes not feasible after conventional quenching and tempering. However, straightening can be performed immediately after the part is removed from the martempering salt bath, while its microstructure is still essentially austenite.

**Austempering and variations**
In conventional austempering, Fig. 5, the part is immersed in a salt bath maintained at a temperature higher than $M_s$ and kept there long enough for all of the austenite to transform to bainite. The part is then removed from the salt bath and air-cooled to room temperature.

Austempering offers many of the same advantages of martempering. However, compared with martensite, bainite has somewhat lower hardness, but is much more ductile and does not require tempering. At a comparable hardness, austempered parts are tougher and more wear resistant than martempered parts, and they have higher impact and fatigue strengths. In addition, distortion is less because the expansion due to transformation is less. These advantages account for the continued growth in austempering applications.

Austempering is normally carried out at 260 to 370°C (500 to 700°F), depending on steel composition and the desired mechanical properties. Bainitic transformation is completed within minutes, and the cycle generally does not extend beyond a couple of hours. Steels that can be austempered include carbon steels with 1% manganese, low-alloy steels such as AISI 4150, 4365, and the 5100 series, ductile irons, and gray irons.

**AISI:** Ductile iron is austempered at 230 to 370°C (450 to 700°F). Processing time ranges from 0.5 to 4 hours, depending on steel composition and mechanical property requirements. The microstructure pro-
duced is ausferrite instead of bainite.

Austempered ductile iron (ADI) offers many advantages over both conventionally quenched and tempered ductile iron and steel. It boasts an excellent combination of strength, toughness, and ductility, which enables the design of parts having improved strength-to-weight ratios. As a result, ADI components have replaced steel castings, forgings, and welded fabrications, and even aluminum in both structural and wear applications.3

Attention also is being paid to austempering of gray iron. The process imparts excellent sound and vibration damping in combination with strength and wear resistance.

Carboaustempering: Austempering of low-carbon-content steels after carburizing produces a high-carbon bainitic case and either a martensitic or bainitic core, depending on steel composition and quench severity.

What makes this process unique is that the core becomes hard first, and then hardening progresses to the surface, further minimizing distortion. In addition, while the surface is being austempered, any martensite in the core is being tempered simultaneously. Carboaustempered parts have excellent fatigue strength and wear resistance, and are dimensionally and functionally superior to carburized and conventionally quenched parts.

Modified austempering: In this modification of austempering, also shown in Fig. 5, quench severity is intentionally decreased to force the cooling curve to intersect the pearlite nose of the TTT diagram. Because a mixed microstructure of bainite and pearlite results, hardness is relatively low — usually in the 30 to 42 HRC range — but ductility is extremely high and strength moderately high.

Wire patenting at 510 to 540°C (950 to 1000°F) is a fine example of modified austempering.2 The method also should be considered for carbon steels and heavier sections where ductility greater than that possible by standard austempering is required. Some trial and error will be necessary to develop the optimum cycle for the particular steel, section thickness, and property requirements.

Other variations: A process that combines martempering and austempering enables users to realize the maximum benefits of both martensite’s hardness and bainite’s toughness. Parts are quenched in an austempering bath and held for about half the normal austempering time. They are then taken out of the bath and cooled to room temperature, during which any remaining austenite transforms to martensite. The time-temperature cycle can be tailored to provide a range of hardness/toughness combinations that would be unobtainable with either process alone.

Additional austempering variations involve the use of two quenching baths and manipulation of agitation.

- Low-hardenability steels can be austempered by using two quenching baths instead of one. The temperature of the first bath is maintained at just above $M_s$ to avoid the pearlite nose of the TTT diagram, while the other bath is at normal austempering temperature. For example, parts made of AISI 1080 steel with a section thickness of about 12 mm (0.5 in.) are first quenched for 30 seconds at 260°C (500°F) and then immediately transferred to the second, 315°C (600°F) bath, where they’re held for the normal duration of austempering.

- Austempering of thin-walled parts such as cylinder liners can be enhanced by manipulating agitation. For example, to minimize thermal shock, agitation is not used during the first 15 to 20 seconds of the quench. It is then turned on automatically to complete the standard austempering operation. This procedure helps to minimize distortion without any sacrifice in required mechanical properties.4

Maintaining salt systems

Salt quenching systems are generally maintenance-free due primarily to the salts’ excellent thermal stability and tolerance for contaminants. Salt baths can provide consistently satisfactory performance for many years simply by adding new or recovered salt to replace that dragged out. The amount of dragout depends on the mass and configuration of the parts being quenched and the fixtures or conveying system, but usually is in the range of 30 to 100 g/m² (1 to 2 lb/100 ft²).

Desludging: Contaminants such as metallic debris from the parts and carbonates that form during service may build up over a long period. Agitation keeps fine metallic particles in suspension, and when suspended metallics exceed 0.5%, quench severity is affected. Similarly, if the level of carbonate exceeds its solubility limit, quenching speed will begin to drop.

Both metallics and carbonate can be removed by desludging the bath. The best method is to lower bath temperature as much as possible, turn off agitation and heating, and then allow the contaminants to settle to the bottom of the quench tank, where they can then be scooped out.

Other checks: If addition of water is a common practice, its content should be checked periodically and adjusted to ensure the required quench severity.

The melting point of the bath needs to be checked only occasionally, since it changes very little over time. A significant increase may indicate thermal breakdown of the salt, usually due to accidental overheating. The cause of overheating should be investigated and properly rectified.

Continued
Fig. 6 — Atmosphere Furnace Co., Wixom, Mich., built this austempering facility for AP Southridge Inc., Elizabethtown, Ky. It features an integrated, 0.9 x 1.8 x 0.9 m (36 x 72 x 36 in.) atmosphere furnace and a fully enclosed, 54,500 kg (120,000 lb) salt quench tank.

Required safety precautions

Although quenching salt is non-flammable and relatively nontoxic, concern for personnel safety arises due to the temperature at which it is used. Adequate precautions should therefore be taken to protect operating personnel from accidental burns.

Following these guidelines also will help ensure safe salt bath quenching:

- Although no toxic or hazardous fumes are given off by the quench bath, good exhaust around the bath is highly recommended. This is particularly helpful during charging with fresh salt and during the quenching operation.
- Parts, fixtures, and conveyors entering a quench bath should be absolutely dry and free of any moisture, oil, or other liquid. Otherwise, rapid vaporization of the liquid may cause sudden expulsion of molten salt, which could result in injury or damage.
- Water sprinklers should not be installed in and around any molten salt system. There should be a clearly visible sign saying not to use water or any liquid-type extinguisher in case of fire. Carbon dioxide-type extinguishers and sand are the best means to fight and contain fires surrounding molten salt baths.
- The salt bath should be protected from accidental overheating by instaling audio/visual alarms that go off when bath temperature exceeds a preset limit. If the temperature continues to rise beyond 595°C (1100°F), the salt may break down, and reactions between the products of the breakdown and the bath container could result in leakage of salt.

- Combustible and incompatible materials like cyanide salt should never be introduced into a quench bath to avoid possible violent reactions

Although quenching salt is nonflammable and relatively nontoxic, safety concerns arise due to the temperature at which it is used. Personnel should be protected from burns.

which may result in an explosion.
- Salt should be stored in well-marked, closed containers, which should be kept in a dry location segregated from incompatible materials such as cyanide salts.

Recovery and disposal

After quenching, parts are immersed in an agitated hot water bath, where most of the salt is dissolved, and then rinsed in a hot water spray. Salt from wash water can be recovered by evaporation of its water content. What results is molten salt that is transferred to box-type metal containers, where it is allowed to freeze into blocks.

Following the recovery and reuse route eliminates disposal of wash water. The drawback is that it causes build up of undesirable contaminants. Periodic adjustment of salt chemistry is required to maintain uniform quenching performance. This helps explain why many heat treaters still prefer to dispose of their wash water.

Although neither highly toxic nor flammable, quenching salt is classified as a hazardous material because of its oxidizing nature. When salt is contained in wash water, the hazard is reduced considerably, and many local waste treatment authorities permit discharge of wash water into their drainage systems. If permission cannot be obtained, the handling of wash water can be delegated to a waste disposal company. Sludge can be used as chemical land fill, where permitted. Otherwise, it can be dissolved in water and treated the same way as wash water.

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References