

THE CARE AND MAINTENANCE OF POLYMER QUENCHANTS USED IN INDUCTION HARDENING¹

D. Scott MacKenzie²

Abstract

Polymer quenchants are used extensively in induction hardening because of cost, ease of control and effective hardening. However, these quenchants must be controlled and maintained. The basic principles of care and maintenance of polymer quenchants are described.

Keywords: Polymer quenchants; Quenching; Refractometer; Viscosity; Contamination.

¹ *Technical contribution to the 18th IFHTSE Congress - International Federation for Heat Treatment and Surface Engineering, 2010 July 26-30th, Rio de Janeiro, RJ, Brazil.*

² *PhD, FASM. Houghton International, Inc., Valley Forge, PA USA 19482. E-mail: smackenzie@houghtonintl.com.*

INTRODUCTION

Induction hardening is a method of surface hardening parts without changing the surface chemistry. A large variety of parts can be induction hardened as is shown in Figure 1.

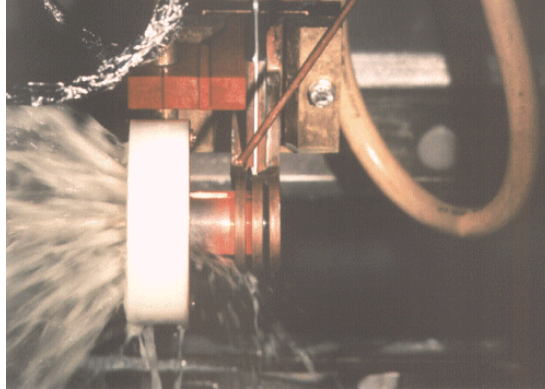


Figure 1. Typical Induction hardening application of a spindle.

The number of quenchant used to quench induction-heated components is almost as numerous as the types of equipment used to heat the parts. No one type of quenchant can be used in every application. The common types in use today include: water, water plus additives (salts, inhibitors, polymers, etc.), normal and accelerated oils, and mar-tempering oils. The choice of quenchant depends on many factors:

- Quench speed
- Hardenability (steel)
- System size
- Case Depth
- Safety
- Shop environment
- Cost
- Compatibility with equipment
- Disposal
- Ease of cleaning
- Contamination
- Distortion susceptibility

Not all of these are considered in every case, as a major concern may take preference over the other considerations. An example of this would be distortion prone bearings that demand the slower transformation of mar-tempering oil, and thus eliminate the possible use of aqueous type quenchant.

Water has many favorable characteristics including cost, ease of disposal, non-flammability, etc. but also has many disadvantages. Water is corrosive to most irons and steels, has an initial prolonged vapor phase that can collapse erratically causing uneven stresses in the quenched part, and remains fast in heat extraction down to the temperature of the bath. The fast heat removal during transformation can crack or distort a part. It is usually restricted to the quenching of plain carbon steels or

massive components with shallow case requirements. Water also is temperature sensitive i.e. the colder the quench water the faster the heat is removed from the part.

Soluble Oil Solutions do overcome the corrosive tendency of water and are relatively cheap. There is a misconception that the additions of soluble oil to water eliminates water's vapor stage problems and gives a less stressed component than plain water. This is not true as the soluble oil solutions still give erratic quench cycles. They also cause problems in waste treatment as the solution is hard to separate and usually requires frequent dumping due to the bacteriological growth. Induction hardening quench tanks are susceptible to bacteriological growth due to normal contamination (from previous metalworking operations), and the stagnant periods between operations. The additions of soluble oil to these tanks shorten the time between disposals of the quenchant. Several of the non-oil inhibitors and newer polymers have replaced soluble oil in induction processes.

Salt / Water Solutions overcome another problem associated with water quenching but have problems of their own. The benefit of salt additions comes from the solids deposited on the hot surface as the water evaporates, causing a faster and more even dissipation of the vapor phase.

This same phenomenon occurs in many accelerated oils as the additives react much in the same way. The detrimental aspects of salt are several:

- High electrical conductivity
- Corrosive to non-ferrous metals
- Very fast quench speeds
- Mixing problems
- Corrosive

Polymer/Water Solutions have seen wide acceptance in induction hardening applications. Many of the benefits of water quenching also pertain to polymer solutions. This includes: non-flammability, ease of part cleaning, cleaner workplace, and minimal disposal problems. In addition controlling the concentration and temperature can change the hardening power, or cooling rate of polymer solution. In many cases, polymer-quenching solutions obtain quenching characteristics similar to quench oil. There are several types of polymer chemistry in use today including:

- PAG - polyalkylene glycol
- ACR - sodium polyacrylate
- PVP - polyvinyl pyrrolidone
- PEO - polyethyl oxazoline

Each polymer has its own physical characteristics. The ACR and PVP products have normal solubility while the PAG and PEO products have inverse solubility (at some elevated temperature below 100°C the polymer will separate from water). The choice of product is normally dictated by the application, the steel hardenability, the compatibility requirements, and in many instances because the user has a familiarity with a given type polymer.

In several applications the type of residue left on the work is important. The PEO, ACR and PVP products give a dry residue while the PAG products leave a tacky residue. This dry residue can be a detriment in induction heated parts that are

press quenched, or special fixtures that can be splashed by a part being transferred to the quench without washing of the fixture. Like water quenchant, the temperature of the polymer solutions is critical and the agitation should be constant.

The other requirement with these quenchant is control of the concentration. Polymers have replaced oils in almost all of the open spray quench applications where oil fumes and frequent fires have been constant problems.

Maintaining Quenchant Media

The user must maintain the quenchant. If a product is rejected due to a heat-treating defect, the quick elimination of the cause of this defect is the only way to continue hardening quality parts. The identification and elimination of the root cause becomes extremely difficult if the controls and records of these controls are shoddy or non-existent. Most manufacturing companies document incoming materials and their manufacturing processes with SQC and SPC but give little thought to the fluid in their quench tank.

Concentration:

Almost all of the polymer quenchant users determine their concentration by use of a refractometer. This is a rapid and easy test done at the side of the quench tank, and is done readily on the shop floor. The refractometer reading (in °Brix) is then multiplied by a concentration factor to obtain the correct concentration. The concentration factor is obtained first measuring the concentration of the quenchant by refractometer (without applying the correction factor). Then the concentration is determined using kinematic viscosity. The correction factor is the concentration by kinematic viscosity, divided by the refractometer reading:

$$CF = \frac{\%C_{viscosity}}{R_{refractometer}}$$

Depending on the use of the polymer, this factor should be determined on an hourly, daily, weekly, monthly, or quarterly basis, depending on the volume of work through the quench tank. For induction applications, it should be done at minimum on a shift basis, because of the large volume of parts that generally heat treated by induction.

There are two general types of refractometers: handheld optical refractometers and digital refractometers. Figure 2 shows typical refractometers. The major problem with the refractometer is that it reads the refractive index of the fluid, and thus includes all dissolved solids including contaminants. Contaminants that darken the quenchant's color cause major problems with this method. This is especially true of handheld optical refractometers.

The range and scale of an optical handheld refractometer is also important to consider. Generally, handheld refractometers have ranges of 0-8°, 0-20° and 0-32°Brix. For the low range refractometers, readings are accurate to approximately ±0.5°Brix. For the larger range refractometers, the readings are accurate to approximately ± 1.0°Brix. In typical induction hardening applications, the concentration generally is in the 3-10% range depending on the concentration and the polymer used. For PAG type polymers, the factor is generally between 2-2.5. For PVP and hybrid type polymers, the factor is generally in the range of 5.6. The use of refractometers is not appropriate for ACR type quenchant. Note that this is only valid for new solutions without any concentration. This would mean for a PAG type polymer that the reading would be accurate to ±1-1.25% PAG. When using a

range of 0-32°Brix, the accuracy of the refractometer would be ± 2.0 -2.5% PAG. For PVP type polymers, with a factor of 5, the accuracy of the polymer would be $\pm 2.5\%$ for low ranges and $\pm 5\%$ for large ranges. This range in concentration control is very large and can contribute to large variations in properties and residual stress or distortion.



Figure 2. Typical Refractometer used to verify polymer quenchant concentration.

Digital refractometers use a monochromatic light source, and are much more accurate instruments. These instruments have an accuracy that is uniform across the entire scale of 0-32°Brix. Generally these instruments are accurate to within $\pm 0.1^\circ$ Brix, and are not prone to operator errors. This means for PAG quenchants, the accuracy is ± 0.2 - 0.25%, and for PVP type quenchants have an accuracy of $\pm 0.5\%$ concentration. Much better concentration control is obtained.

The major problem with the refractometer is that it reads the refractive index of the fluid, and thus includes all dissolved solids including contaminants. Contaminants that darken the quenchant's color cause major problems with this method. Kinematic viscosity is a much more accurate method of determining concentration of aqueous quenchants but this apparatus requires specialized equipment and a laboratory environment. This test is not something performed on the shop floor. This method is relatively immune to contamination and accurate concentration can be determined readily using reference control charts of viscosity versus concentration. Typically, the quenchant supplier will provide viscosity measurements on a monthly or quarterly basis. As a general rule, viscosity measurements are accurate to $\pm 1\%$ of reading or better, and $\pm 0.5\%$ polymer concentration.

Contamination:

Contamination is a major problem, especially with induction hardening operations. The contaminants come from:

Liquids from preceding operations not burned off in the heating cycle.

- Hydraulic fluids from the induction equipment
- Oils and greases used as lubricants
- Forging compound additives that are water soluble after heating
- Concentrated elements from the water used in the quenchant

- Insertion of the wrong product into the quenchant
- To minimize or eliminate contamination problems the user can:
- Clean the parts prior to induction hardening
- Use a compatible hydraulic fluid and make sure the seals and other materials are compatible with the quenchant
- Choose lubricants less soluble in the quenchant
- Review the ingredients of forging compounds with the quenchant supplier
- If the water quality is suspect change the water source or install water treatment equipment
- Closely control and monitor all additions to the quenchant.

Without proper control and records, the job of troubleshooting a heat treating problem becomes a game of "Where do I go next?", or "Where are the experts when I need them?"

Probably the largest source of contamination is from the lack of proper cleaning prior to induction hardening. The quench tank captures the coolants from machining operations and possibly hydraulic fluids from the induction hardening equipment used for transfer of parts. Properly designed skimmers and filtration systems will help minimize this source of contamination from the quench tank (Figure 3).



Figure 3. Dirty quench tanks and poorly maintained skimmers contribute to non-uniform quenching and are sources of nutrients for biological growth.

Contamination by cutting fluids, rust preventatives, cleaners or hydraulic fluids can provide nutrients for biological growth. These contaminants may also prolong or stabilize the vapor phase of the quenching process, which can contribute to low hardness and inadequate case depth. Often the use of an air knife prior to the heat treating operation can remove most of the coolant and reduce the contamination. While cleaning the parts prior to heat treatment is always preferred, the use of an air knife is an acceptable alternative.

Sediment and particulate matter such as scale has little effect on the quench rate, but can increase the overall quench rate by providing nucleation sites for bubble formation and destabilize the vapor phase. This is not usually a problem in induction hardening because of the direct impingement of quenchant on the parts. However, this particulate matter can hinder concentration control by making the refractive index difficult to measure. This contamination can also affect the cleanliness of the quenched component. Filtration is always recommended to remove this particulate.

Typically, filtration of the quenchant to approximately 25 microns is adequate to remove most particulates.

Biological Contamination:

Most bacteria contamination is associated with anaerobic bacteria. This is a common problem for all polymer quenchants. This bacterium is not a health hazard, but more of a smell issue. This smell is caused by large numbers of anaerobic bacteria decaying. Both HCl and H₂S are formed by the decay. HCl can some times appear as a green cloud over the quench tank when the equipment is first started. Hydrogen sulfide, H₂S, is the rotten-egg smell. Both of these chemical compounds decrease the pH and contribute to increased corrosion of parts and equipment, and increased bacteria growth. It is this bacterium that contributes to “Monday Morning Smells”. This is typically cured by sump additions of a biocide or dumping the system. This is a costly, and potentially dangerous (with regard to biocides) solution to the problem of bacteria growth.

Anaerobic bacteria thrive in oxygen depleted environments, such as is found at the bottom of a quench tank. Stagnant solutions contribute to localized oxygen depletion, which in turn increases potential for bacteria growth. To prevent stagnant solutions, the quenchant should be kept moving, and the quenchant free from rust and solids. It is these solids that provide much of the food source for anaerobic bacteria.

Bacteria can be readily controlled with out the use of biocides. Cleanliness of the quenchant and quench tank are critical to preventing the growth of bacteria and the subsequent use of biocides. The quenchant should be kept agitated to eliminate stagnant solutions. Once the equipment is shut off for the weekend, the quenchant should be kept agitated. This can be done by keeping the filtration pump “on” over the weekend, or by intermittently initiating the agitation system on a periodic basis. A couple of minutes every hour should be adequate.

Filtration is also very effective in reducing bacteria growth. Filtration serves two purposes. First it eliminates particulate, scale and other debris from the quenchant that can act as a food source. Secondly, it maintains the quenchant cleanliness so proper quenching can occur. Bag and cartridge filters are commonly used in this application. Typically bag sizes down to approximately 15-20 microns are used. However these type of filters are not recommended for this application, as the low fluid flows through the filters and the high concentration of food sources can contribute to bacteria growth. Further, this type of filtration, once contaminated, can spread the contamination to other locations in the quench tank.

Sand filters are very effective filtration systems for polymer quenchants. The nature of sand filtration has very high flow velocities so stagnant solutions do not occur. Further, they are also very effective at filtration, with filtration levels often at 6-8 microns or better. They are also cost-effective, as the filtration media is inexpensive, clean white sand.

Quenchants have been developed that will not sustain bacteria growth – and “will not stink”. These quenchants do not contain any biocides and are specially designed for induction hardening applications with enhanced corrosion inhibitor packages. These are proprietary quenchants designed to have long life and not support bacteria growth. Examples are Houghton’s Aqua-Quench 245 and Aqua-Quench 145 polymer quenchants. These quenchants are PAG based, but should not be mixed with nitrite containing quenchants. These quenchants perform identical to other PAG-type quenchants in similar concentrations.

System Control:

The quenchant user must maintain all of the parameters of the heat-treating operation, including the quenchant. The controls include routine maintenance of the quenchant equipment and the control of the quenchant itself. To install automated temperature, concentration, and flow monitoring equipment does not mean the system is under control. The monitoring equipment must be checked on a routine basis by means verifying the instrumentation. This is done by equipment calibration, independent laboratory analysis, in-house laboratory analysis, quenchant supplier testing, or several of these methods. A typical report for a PAG type polymer quenchant is shown in Figure 4.

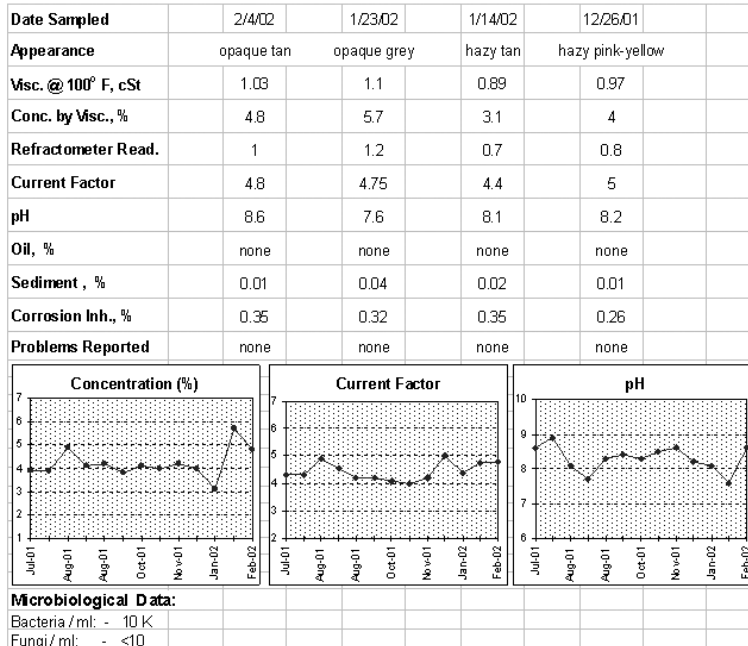


Figure 4. Typical polymer quenchant report.

Quenchant Problems

The first thing usually blamed in any heat-treating operation is the quenchant, and this is especially true in induction hardening operations. There is a good reason for this, as the quenching process creates extremely high stresses in the transformation from one phase to another. This transformation causes dimensional changes and potentially high residual stresses. Many times a quick visual inspection of the operation can indicate a possible cause.

Several common, reoccurring problems in induction systems, which can be ascertained during visual inspection, are:

- **Foam in quenchant.** The existence of foam causes slow and uneven heat extraction
- **Quenchant contamination.** A color change or the presence of floating oil is an indication of a contaminant that could change quenching rates.
- **Agitation problems.** The loss, reduction, or partial restriction of agitation typically causes major quenching problems.

If an aqueous quenchant is being used the concentration should be checked. The next steps in the troubleshooting process are to review all records, such as; charts, quenchant control records, time cycles, current flow, and the material composition. If the cause of the problem cannot be found, a sample of the quenchant

should be sent to an internal, independent, or quenchant supplier laboratory for analysis.

The problems encountered with quenchant suppliers can be separated into several categories, which include:

- Distortion & cracking
- Low hardness or physicals
- High drag-out
- Fires (oil only)
- Odor or smoke
- Corrosion (aqueous only)
- Foaming
- Biological (aqueous only)

A fault-tree showing possible reasons for problems in induction hardening using polymer quenchant suppliers is shown in Figure 5.

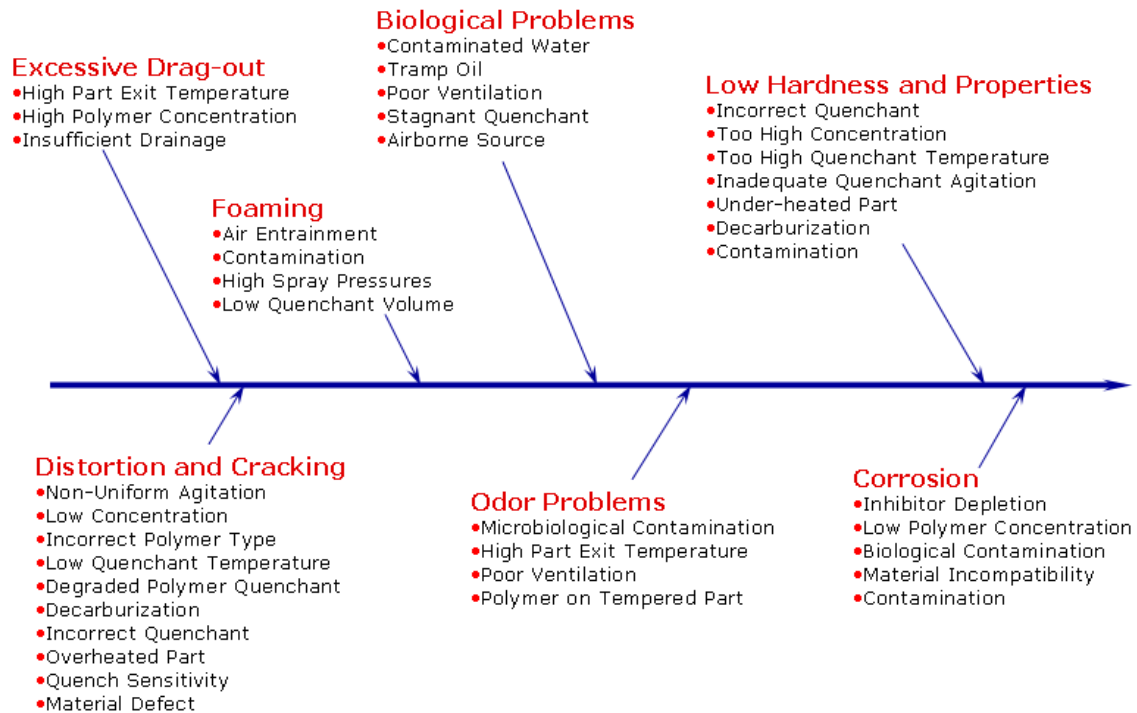


Figure 5. Fault tree for troubleshooting induction hardening problems when associated with polymer quenchant suppliers.

Distortion and Cracking. The heat-treating operation receives the blame for all cracked or distorted components found after the heat-treating operation. In many cases the blame is placed on the quenchant. There are generally two groups of problems that can cause cracking. The first group is a list of reasons attributed to things prior to the quenchant, while the second group lists what can cause cracking during quenching. Neither list is complete, nor is the major post-quench reason of delayed (or eliminated) tempering included.

Usually when cracking or distortion is attributed to the wrong quenchant it is because the quenchant is too fast. This could be due to the concentration of a

polymer being too low, the inability of a given polymer to harden a component, an oil being too fast, or water being used as the quenchant without additives. Low concentrations of polymer quenchants can cause high stresses that can lead to cracking and or distortion.

Uneven or lack of agitation can cause cracking and distortion. Plugged sprays are common in induction hardening (Figure 6). The agitation should be even throughout the quench cycle to give as even a stress pattern as possible.



Figure 6. Non-uniform quenching due to plugged nozzles.

Excessive Drag-Out. In most cases this is due to excessive exit temperatures, high concentration or insufficient drainage from the parts. Excessive exit temperatures cause high drag-out of the polymer because the exit temperature exceeds the separation temperature for PAG quenchants. For most applications, excessive drag-out can be minimized by reducing the concentration; longer quench dwell times (to allow the polymer to go back into solution), and by changing the orientation of the part if the configuration of the part would tend to trap quenchant. The use of an air knife to blow off excess quenchant after quenching can reduce the amount of quenchant consumed.

Foaming. Foaming problems in a quenchant are almost always related to mechanical problems. The most common source is inadequate fluid levels in the quench tank. This can contribute to vortexes at the inlet to the pump. Excessive agitation from high spray pressures can also cause foaming. Contamination from soaps, or coolants can also cause issues with foaming. The first thing to check is the level, and spray pressures. Pump seals can also cause aspiration, and air entrapment. Application of commercial defoamers, recommended by your quenchant supplier can help resolve this issues after the mechanical issues have been examined.

Low Hardness and Low Properties. While most problems due to low surface hardness can be related to either the incorrect quenchant; too high a quenchant concentration or excessive quenchant temperature, there are other things that can cause low properties. Inadequate heating dwell times or inadequate part temperature can mimic a poor quench. The part temperature should always be verified to rule out this from the “usual suspects”. Elevated quenchant temperatures and high quenchant concentration can both slow the cooling curve of the quenchant. Improper agitation can cause soft spots and distortion, as well as “barber pole” response (Figure 7).

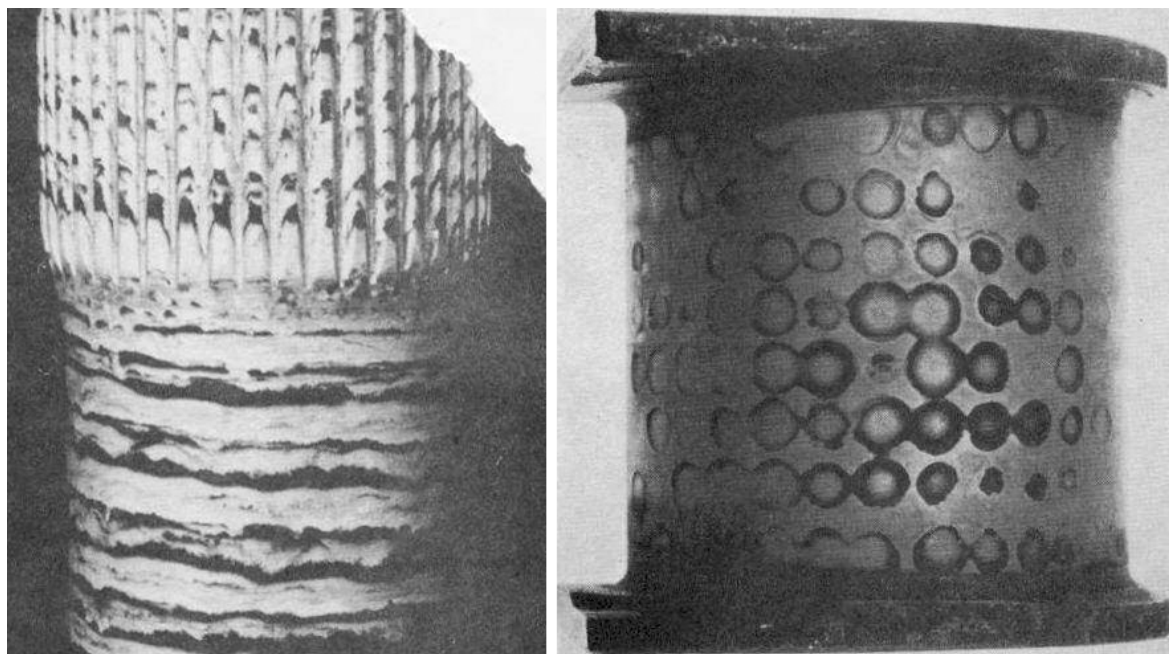


Figure 7. Soft spots and distortion can occur when the free exit of water stream or vapor is impeded. With scan hardening, "barber Pole" patterns can develop when the quench stream is erratic or displaced.

CONCLUSIONS

In order to harden components successfully, the entire heat-treating process must be controlled. Consistent heat treating means the control of the components coming into the hardening operation; control of the heat treating equipment; control of the quenchant; and control of the heat treating process parameters. The only way to control all of aspects of heat-treating is to understand all parts of the operation, including the principles of quenching. The more that is known about an operation, and the equipment and chemicals used in that operation, the easier it is to eliminate potential problems or to solve problems that did occur.