Quench Oil Cleanliness and Part Quality

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Abstract

Serious problems related to quench oil contamination have always affected the heat treat industry. These problems have led to shortened oil life, variations in the quench curve, loss of production time, unplanned maintenance, sludge accumulation, surface deposits, and massive amounts of rework costs associated with additional part cleaning. This paper will detail the causes for contamination, including; carbon deposits, oxidation of the oil that result in unwanted by-products, ingress of dirt from parts and external sources, and water ingress. We also discuss a way to eliminate the extensive downtime associated with excessive sludge, and cleaning of reservoirs and equipment. The paper will give visual representations of the effects of quenching in dirty oil vs. quenching with clean oil. Additionally, we will look at several case studies that show the relationship of clean oil to clean parts and proper quenching properties. The paper will also detail various methods of particulate and water removal and how effective they are with the quench oil application. This session would be important for the attendees that are utilizing quench oils and are looking to extend their oil life, quality of their parts, and reducing overall costs associated with the process.

Introduction

Throughout the history of metal heat treating, the need for clean and dry quench oil has always been recognized but has been largely ignored by the many heat treat facilities, both commercially and by the Original Equipment Manufacturers.

There are many sources of contamination in quench oils, including soot, scale, dust, water, and oxidation by-products. Staining, especially after heat treatment, is both difficult and expensive to remove. The most common sources of stained parts are contaminated or oxidized quench oil [1][2].

Many of the costs and risks associated with heavily contaminated quench oil can be reduced or eliminated utilizing proper methods of both particulate removal and water removal. Many companies are already cashing in on the benefit and cost savings of clean oil, including: longer life of the oil, increased uptime on equipment, little or no maintenance associated with the contaminated oil, and elimination of rework associated with the cleaning of dirty parts.

Oxidation

The life of quench oil is dependent upon its thermal stability. The thermal stability is a function of the quality of the base oil, the antioxidant package used, and the presence of heat and catalysts [3].

The degradation of quench oil is aggravated by residues on parts, washer residues from oil reclaimed from washers; high energy density heater or radiant tubes, and excessive peak temperatures.

Oxidation of quench oil is caused by exposure to oxygen. As operating temperatures increase, the kinetics of oxidation approximately double with each 10°C. This is especially true with mar-tempering oils because of their elevated temperatures while in use.

Thermal degradation is from exposure to temperatures that cause the base oil and additive package to change. This results in the formation of insoluble products of reaction that can cause deposits on parts and sludge the quench tank.

Contamination can be from many sources. Water, dust, scale, and soot are not the direct result of oil degradation but can contribute to other degradation issues. Soot can act as nucleation sites for thermal degradation products and can mimic oxidized oil.

Oil degradation is manifested by a viscosity increase, acidity increase (as measured by Total Acid Number), varnish and lacquer deposits, sludge, and changes in the quench speed [4]. Examples of staining of parts are shown in Figure 1.

There are many papers covering the mechanism of oxidation of oils and the function of antioxidants [5][6][7][8]. There are three primary steps in the oxidation of oil: Chain Initiation; Chain Propagation and Branching; and Termination.
Carbon Deposits and Sludge

As oils become increasingly oxidized, whether in the quench tank, or in oxidation tests, the viscosity increases. This occurs by condensation reactions that become important as the levels of aldehydes and ketones increase. These reactions are known as Aldol Condensations [9]. It is this reaction that causes varnish on parts and sludge in quench tanks.

The condensation products described in the above section have a limited solubility in the quenchant. These are high molecular weight oligomers. These are molecules that have a few monomer units, in contrast to a polymer that can have an unlimited number of monomers. As oil oxidizes the amount of carboxylic acids will increase. These acids are very effective catalysts for Aldol condensation reactions [9]. These then convert the low molecular weight carbonyl compounds into higher molecular weight oligomers.

As the reactions progress, chemical changes in the oligomers will result in making them insoluble in the quench oil. At this point the insoluble oligomers will precipitate from the quench oil and create sludge on the bottom of the quench tank, and deposits on the hot metal part.

Ingress of dirt from external sources

Not only is the quench oil cleanliness impacted by the severe duty of the quenching process, but also by the surrounding environment as well. Housekeeping in many heat treat facilities is less than desirable, simply because of the nature of the process itself. Subsequently, these quench oils are subjected to a lot of dust and dirt from the environment, along with the dirt and scale that is carried over on the parts as part of the heat-treating process.

Some designs and systems are better than others, but with any non-sealed system, coping with the external ingress just exacerbates the problem that you have with an oil that will become contaminated through the normal process of quenching alone.

Water Ingress

Water can have a dramatic effect on quenching in oil. The potential for fire, explosion, and injury to people and equipment are ever present and should be taken extremely seriously.

Water can ingress into the oil in various ways, but the most common are condensation, water-cooled heat exchanger leaks, poor maintenance practices, and washing of parts post heat treat. Some of these can be controlled through best practices, but we should all be aware of the dangers associated with water and proper methods of removing that water, down to an acceptable level.

Problems Associated with Contaminated Oil

Shortened oil life

The life of your quench oil will vary depending on the frequency of use, operating temperatures of the oil, and levels of contamination. All these factors affect the quenching properties of the oil and lead to frequent and unnecessary change outs of the quench oil. Companies that see the oil as an investment can put in place good oil maintenance programs, which can easily lead to doubling of the life of the oil, saving both oil costs and the environmental impact of having to dispose of waste oil. It is best to start the program when the oil is new, and the system has been cleaned in order to maximize oil life.

Variations in the Quench Curve

During a recent experiment [3] a sample of Mar-Temper 355 was received from a customer. The oil was badly oxidized (Table 1) and exhibited a cooling curve indicating badly oxidized oil (Figure 3).
Table 1. Table comparing new and as-received oil.

<table>
<thead>
<tr>
<th>Property</th>
<th>New Min.</th>
<th>New Max.</th>
<th>Used Min.</th>
<th>Used Max.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Viscosity (40°C), cSt</td>
<td>69</td>
<td>82</td>
<td>85.6</td>
<td></td>
</tr>
<tr>
<td>Flashpoint, °C</td>
<td>229</td>
<td>252</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Water, %</td>
<td>-</td>
<td>0.10</td>
<td>0.02</td>
<td></td>
</tr>
<tr>
<td>TAN, mg KOH/g</td>
<td>-</td>
<td>0.1</td>
<td>3.0</td>
<td></td>
</tr>
<tr>
<td>Precipitation Number, %</td>
<td>-</td>
<td>0.05</td>
<td>0.4</td>
<td></td>
</tr>
<tr>
<td>Sludge, %</td>
<td>-</td>
<td>0.05</td>
<td>0.5</td>
<td></td>
</tr>
</tbody>
</table>

Figure 3. Severely oxidized oil showing change in cooling curve behavior.

The physical properties of the used oil exhibited severe oxidation. The viscosity had substantially increased. The Precipitation number and Sludge were also nearly an order of magnitude greater than new oil. This shows that the oil is badly oxidized, and contains considerable oxidation by-products, and potentially other contaminants.

In the cooling curve of the oxidized oil, the vapor phase of the oxidized oil is virtually non-existent. Further, the maximum cooling rate has increased substantially. The fine suspended oxidation products are causing the initiation of nucleate boiling at a much earlier temperature. Further, the oxidation products also act as a speed improver, increasing the maximum cooling rate.

During this experiment, a fine filtration system with 3-micron absolute filtration was used to clean the oil. While the badly oxidized oil did not return to new condition, a substantial improvement was obtained. After final filtration, the oil would be acceptable for continued use, with moderate oxidation.

Specific locations on the cooling curve are shown in Table 2. In the oxidized oil, the vapor phase was practically non-existent. The oil showed an improvement in the vapor phase, nearing the value for new oil. The maximum cooling rate was also reduced to within the allowable range for Mar-Temp 355, and the expected variation of the cooling curve test [10]. The temperature at the maximum cooling rate was reduced to nearly the value of new oil.

Figure 4. Cooling curves of new oil compared to the severely oxidized oil. Additional curves showing the improvement after initial pass and final filtration passes are shown.

Table 2. Specific values for the cooling curves shown in Figure 4.

<table>
<thead>
<tr>
<th></th>
<th>New</th>
<th>Used</th>
<th>Initial Pass</th>
<th>Final Pass</th>
</tr>
</thead>
<tbody>
<tr>
<td>Max CR</td>
<td>78.8</td>
<td>91.0</td>
<td>86.8</td>
<td>83.2</td>
</tr>
<tr>
<td>Temp @ CR_{max}</td>
<td>613.2</td>
<td>682.3</td>
<td>682.9</td>
<td>656.8</td>
</tr>
<tr>
<td>Cooling Rate @ 704°C</td>
<td>28.3</td>
<td>89.2</td>
<td>85.5</td>
<td>72.9</td>
</tr>
<tr>
<td>Cooling Rate @ 600°C</td>
<td>76.2</td>
<td>74.1</td>
<td>85.5</td>
<td>71.6</td>
</tr>
<tr>
<td>Cooling Rate @ 500°C</td>
<td>40.3</td>
<td>41.0</td>
<td>37.7</td>
<td>42.5</td>
</tr>
<tr>
<td>Cooling Rate @ 400°C</td>
<td>7.3</td>
<td>13.2</td>
<td>11.8</td>
<td>16.4</td>
</tr>
<tr>
<td>Cooling Rate @ 300°C</td>
<td>4.3</td>
<td>3.9</td>
<td>4.1</td>
<td>4.0</td>
</tr>
</tbody>
</table>
Loss of Production Time and Unplanned Maintenance

Uptime is critical in most manufacturing environments but can be extremely critical in this industry where the furnaces have limited capacity and are running 24/7 in many cases. An outage to do preventative maintenance and cleaning is oftentimes postponed or cancelled outright in order to keep these workhorses running and producing parts. Cleaning sludge and problems with pumps and heat exchangers can be time killers and can be lessened or prevented with a good oil maintenance program.

Rework Costs

All the problems mentioned above can be very costly and on their own would be reason enough to invest in a good maintenance program. Costs associated with rework can be astronomical and can trump any of the other costs, totaled together.

Also, the costs associated with transportation and returns from the internal or external customer must be considered.

Filtration Methods

Filtration is a common method to remove particulate, including oxidation products from quench oil. There are several methods, and these will be discussed briefly. [3]

The dirt load, including particulate from oxidation products, ranges in size from very small (sub-micron) to very large. The particle size in general is inversely proportional to the amount of particulate present. In other words, there is a greater amount of fine particulate to be removed, than large particulate (Figure 5).

Bag Filters

Bag filtration systems are common in the heat-treating industry. They are an inexpensive method of particulate removal but fall short in the cleaning the oil to a level that would extend the life of the oil. Typically, bag filters are used at 10-20 microns. As can be seen from Figure 6, this size will only remove about 10% of the particulate present. Bag filters have the advantage of being inexpensive but suffer the drawback of very limited particulate retention. This results in replacing the filter element at a very short interval, increasing maintenance costs. Replacement of the filters is a messy job.

Pleated Filter Elements

Pleated filter elements are either cellulose or a micro-fiberglass fiber. These filters have capabilities to some very fine particle removal, down to less than 1 micron. However, the problem with these filters is the amount of dirt that the filter can hold. An industry standard pleated micro-glass filter can only hold around 140 grams of dirt per filter element. With the high number of particles that are inherent in a quenching system, these filters would not last very long and would be a very expensive solution to the problem. In addition, the micro-glass filters would only be able to remove particles and would have no impact on the water, acid, or soft contaminants (that lead to varnish). In the presence of water, the pleated cellulose elements will swell and become clogged very quickly. As an example, a commercial heat treater purchased and installed a pleated filtration system. They stopped using the system shortly after installation due to blinding of the filters, which had resulted in changing the filter elements daily. While the intent was there, and the filters were capable of very tight filtration, the system was just not economically feasible to use due to the low dirt retention of the filter system.

Offline Depth Filtration

The best method for capturing and retaining fine particles, water and varnish, is by installing an offline depth filter. This system would utilize a dense cellulose filter medium capable of filtration down to 3-micron absolute or .8 micron nominal. The system would circulate the oil continuously. The depth filter provides a large surface area and fine capture area for the
capture of particles, soft contaminants and water. This debris would be captured on the surface of the filter and in the interior filter material. This enables the depth filter to have the highest dirt holding capacity of any type filter insert or element. This high dirt holding capacity makes the depth filter the best solution for filtering quench oils with such a high dirt load. Not only can the oil get to the cleanliness level necessary to prevent the sludge and carbon deposits, but this can be done with an insert that can hold several pounds of contaminants. The high dirt load capacity will result in long filter change intervals. Depending on the cleanliness of the oil, and the size of the filtration system, filter change intervals can be extended to months between filter changes.

The typical dirt holding capacity for this type of filter is 4 liters of evenly dispersed solids (depending on size of filter element). The filtration element will retain approximately 4 kg of oxidation products, such as sludge and the oligomers from oxidation. Wood cellulose is most commonly used and can retain up to 50% of the total retention capacity.

Methods of Water Removal

There are six primary methods for the removal of water. [11]

Heating (Boiling off)
Heating the oil is a very effective method to remove water, which over time will evaporate. Some oil filters can be fitted with a heating coil with the purpose to heat the oil to a temperature where water will evaporate. Free water and some dissolved water can be removed with this method.

The drawback of this method is that to be effective the oil must be heated to a high temperature, which will increase oxidation and varnish generation significantly leading to a shorter oil life and part staining. Since water is removed by evaporation the cost of power per amount of water is several times higher compared to other means of water removal.

Boiling off water is accomplished by gradually increasing the oil temperature to a little above the 100°C, with the agitators running. If in an integral quench furnace, the outer door should be opened, and the inner chamber door closed, and at heat. The inner chamber is left up to heat to prevent any moisture from contaminating the refractory. Test samples should be taken at intervals (with the agitators running) to ensure that a representative sample was taken. Initially a crackle test can be run. Once no water is detected using this method, it should be followed-up with a Karl-Fischer titration for accurate water determination.

Coalescing
Coalescing is a very common method of water removal from various types of oils and fuels. Water droplets are separated from the fluid in three steps.

1. The first step is the filter media which removes particles and other contaminants from the oil, increasing the interfacial tension between oil and water leading to an increase in water separation.
2. In the second step the fluid passes a fine mesh of fibers where water droplets are merged together (coalesced), here they will reach a size allowing them to separate in the third step.
3. Separation from the fluid is the third step, where a bulk layer of water is formed in the bottom of the separator, to be discharged from the filter system.

The filter separator is only able to remove free water and to some extend emulsified water, but not dissolved water.

The efficiency of the filter separator drops with higher oil viscosity and water separation strongly depends on the interfacial tension between the oil and water. A water separation test (ASTM D 1401) of the specified oil is required to determine whether the filter separator can remove water from the oil or not.

This method of water removal is very effective, as long as the oil demulsifies. Most quench oils are paraffinic types, which show good demulsification. The ASTM test looks for complete separation of an oil and water mixture (40ml each) in 15 minutes, to be considered a good rating. It is important that when considering this type system, you do this test on an actual oil sample, as oil can lose the ability to shed water over time. For example, a new oil may very well pass the Demulsibility test, but because of age or surfactant contamination, the oil...
would trap or hold on to any water, making it a poor candidate for coalescing. Also, some additive packages may tend to increase emulsification of the quench oil.

A typical coalescing filter is shown in Figure 7.

Absorptive Filtration
Filters containing cellulose, water adsorbing zeolites or other hydrophilic materials, can to some extent absorb free, emulsified and dissolved water from oil. The volume of absorbed water depends strongly on the amount of hydrophilic material, the oil temperature and the water content in the oil. After some time, the filter will reach maximum capacity of absorbed water, and must be replaced.

This method is suitable for all oil types and will also remove particles and varnish from the oil but is most economical for smaller systems containing little amount of water in the oil e.g. condensation accumulated over time.

Figure 7. Typical coalescing filter also useful for removing very fine particulate.

This method is very effective in the day to day cleaning of your quench oils and can be looked at as a maintenance tool, more than a recovery type tool (for waste oils coming from a skimmer on the back end of a water wash application). A typical absorber type filtration system is shown in Figure 8.

Vacuum Distillation
When oil is put under vacuum the moisture will evaporate off at normal operation temperature. This principle is used in vacuum purifiers which can combine filtration of the oil as well.

It will remove dissolved water and some emulsions but does not perform well with free water. Foaming of the oil could occur, and result in an equipment fault.

This method is suitable for most oil types but is most economical for complete closed systems without much water ingestion. High capital cost, difficult settings and operation are some of the drawbacks. Free water can be removed with some vacuum dehydration units, but the cost of power per removed ml. of water is several times higher compared to a filter separator or a centrifugal separator, since the vacuum dehydration unit must evaporate the water before it can be removed. This system also works best when the oil is heated to increase the vapor pressure of the water. Often, an absorption type filter is used prior to vacuum distillation.

Often, when high levels of water are detected, the use of an outside company to vacuum distill the water is called to process the quench oil. They can process the contaminated oil quickly, as well as filter the oil, but the cost is high – typically half or more of price new quench oil per gallon.

Figure 8. Typical absorber type of filtration useful for removing fine particulate and small quantities of water.

Centrifugal Separators
The water is separated from the oil by centrifugal force utilizing the difference in specific gravity between the fluid and the water. Large amounts of free water and some of the emulsions can be removed per hour, but not dissolved water. Excellent water separation is required. The disadvantage is high capital and maintenance costs, as well as high power consumption under continuous operation. The heavy dirt load for a quench
Desorption

Free water, emulsions and some dissolved water can be removed with desorption type systems. This system works by heating the oil and circulates dry air/gas through the oil. The dry air absorbs the moisture from the oil, thereby removing water.

This system is based on very reliable technology which requires very little maintenance and is most economical for large systems or oil types which dissolve large amounts water and create strong emulsions.

The degree to which the oil can be dried depends on the ability of the oil to release water. A water activity test of the quench oil is required to determine whether a desorber can remove water from the oil or not.

High capital cost and high-power consumption is required to remove the water due to the necessary heating and cooling of oil and air.

This method is well suited for recovering quench oil from washer solutions, or other badly contaminated solutions. With careful monitoring, the oil can be recovered for further use in a short period of time. This can represent a significant cost savings to the heat treater in further purchases of quench oil, or cost avoidance in not having to dispose of the used quench oil. If a large system is processed, the oil should be tested after recovery to verify that the water has been removed, and that the oil is suitable for continued use.

The effectiveness of a desorber in removing water is shown in Table 3. In this example, several thousand liters were processed, with the water reduced to a safe level (below 1000 ppm) in less than 9 days. An example of a Desorber Filter is shown in Figure 10.

Table 3. Results of water contamination in a quench oil, in recovered washer solution, and resulting reduction of water present using a Desorber filter. Water content determined per ASTM D6304.

<table>
<thead>
<tr>
<th>Time (days)</th>
<th>Initial</th>
<th>5</th>
<th>9</th>
<th>10</th>
<th>11</th>
<th>12</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>4.684</td>
<td>.8215</td>
<td>.0381</td>
<td>.0199</td>
<td>.0228</td>
<td>.0131</td>
</tr>
</tbody>
</table>

A recent case study at supplier to the automotive industry in the Midwest, resulted in water removal from almost 5% to an acceptable level of 131 ppm on their oil (Table 3). The oil performance characteristics were still intact. The financial impact for recovering this waste oil stream was a $192,000 cost savings each year. The payback time period for the equipment to remove water from this waste oil stream was less than two months.

Clean Oil = Clean Parts

So, how does clean oil affect my part quality? Removal of staining on a part can be an expensive process. It requires additional effort such as shot blasting to obtain a clean surface. If the parts are not properly cleaned, then surface appearance is objectionable (Figure 10). Lack of adhesion for subsequent coating process such as painting, nitriding or plating can become an issue.
The photos in Figure 10 above represent two separate case studies. The top photo is from a German company that bought their own heat treat facility to vertically integrate their processes, only to find that when they started processing their own product that the part was very stained and lacked the aesthetic appearance of the parts from the outside processor. They soon learned that the difference was the filtration of the quench oil and put the same filtration in place at their location and, subsequently, the parts returned to their previous, desired appearance.

The bottom photo is from an automotive supplier in Europe that was having a problem with the black deposits on these valve covers. Only 1 ½ weeks after installation of a fine filter system, the surfaces of the quenched parts were significantly cleaner. The deposits had disappeared and rework and sandblasting were discontinued. Additionally, the amount of rejected parts was reduced.

Heavy residue and deposits are shown in the photos below (Figure 11) are from a recent case study at a large Original Equipment Manufacturer in the USA. The OEM implemented a new filtration strategy on a belt furnace, which resulted in an overall cost savings of $150,000 per year in rework costs alone. They also benefited by doubling the life of their oil, stabilizing their quench curve, and maximizing uptime on their equipment due to no further rejects.

In figure 12 above, you can see photos under a microscope, showing the difference in unfiltered dirty oil and the oil after filtration. This was on an open bath quench oil system for large gears in Finland, with a tank volume of over 10,000 gallons.
The manufacturer was having problems with short oil life, sludge formation between the gear teeth on the product, uneven hardening and reduced surface strength. Additionally, the parts had black spots, which led to rework, downtime and frequent cleaning of the oil reservoir.

Upon adding the proper filtration system, the manufacturer has been able to double the life of their oil, reduce the frequency of tank cleaning, and eliminate the need for rework and cleaning of product coming out of the quench tank.

Conclusion

The contamination of quench oil is a given and is inherent in the process of heat treating. From the problems associated with the oxidation of the oil, to the outside contaminants that impact the oil and the water that poses a potential catastrophic threat, there will always be a high level of contamination associated with the heat treating and quenching process.

Even though the contamination exists, the results of that contamination can be mitigated and even eliminated by having the proper oil maintenance program in place. No matter the method you use to clean the oil, the costs associated with doing nothing far outweigh the costs to put that method in place, when you consider all of the factors, including: cost of shortened oil life (resulting in frequent oil changes), rework costs, downtime, environmental costs for disposal, and potential catastrophic failures if you have a water ingress in your system.

However, with a good maintenance program, which includes removing both particulate and water from the oil, you can reduce or eliminate many of the factors that are related to the problems associated with that contamination, increasing uptime on the equipment, assuring clean parts and assuring proper quenching properties. Starting this program from the beginning should be the goal and you will maximize the life of the oil and the overall profitability of your heat treat processes.

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References


