TheNDTTechnician

Vol. 20, No. 4

FOCUS

Nital Etch Technique for the Inspection of High-Strength Low-Alloy Steels

by Joshua Pierre

Nital etch inspection is a systematic process that uses acidic and basic chemicals to help detect localized heat-affected areas in steel alloy products and components. Recently, nital etch inspection has gained a lot of interest in the aerospace industry. This process is categorized as a chemical process utilizing corrosive chemicals and a test technique according to a detailed specification that brings about reliable and repeatable results. The nature of this process makes it "destructive" to the test object. The removal of metal on the component's surface aides in the inspection. However, although considered a destructive test, this does not render the part unusable.

Localized Heat-Affected Area

MIL-STD-867 (US DOD 2019) contains a list of the high-strength low-alloy (HSLA) steels used in the process of nital etch (note that the US military refers to nital etch as "temper etch"). HSLA steels in group A include SAE 52100, SAE 4140, SAE 4330, SAE 4340, 300M, D6AC, 440C, and Aermet 100. There are other listed in this specification; however, for this article, we will focus on the low-alloy steels. The HSLA steels are combined with various other elements during their creation to increase their physical and chemical properties. This is referred to as "alloying." The HSLA steels listed previously contain a low carbon content (0.2% to 2.11%), which enables them to be hardened by heating to a high temperature. This process is called "austenitizing." These HSLA steels undergo a series of thermal treatments, such as austenitizing, quenching, and tempering, to produce a uniform hardness and strength level during their creation. The quenching process rapidly cools the HSLA steel, thus changing its metallic structure to a hardened state. Tempering allows the metallic structure to stabilize and be held at its final state. After heat treating, these HSLA steels may be finished through grinding or

From *NDT Technician Newsletter*, Vol. 20, No. 4, pp: 1-5.

machining. These processes produce a significant amount of heat when performed on hardened and tempered steel surfaces. When these mechanical processes become abusive, certain locations on the surface will undergo a structural change or behave as if the thermal treatment is continual. As a result, the properties of local areas of the high-strength steel have changed on the surface. These areas have become either extremely hard and brittle, which is referred to as being "untempered," or extremely softer than the base material, which is referred to as being "overtempered." The loss of hardness or brittleness gained through this localized heat treatment can severely compromise the integrity of the material. This compromise of integrity can lead to a component fracture, cracking, and catastrophic failure. Figure 1 shows an example of cracks from untempered surfaces under stress. Because the hardness changes in this region, this change can be detected by a Rockwell hardness testing instrument. A Rockwell hardness tester utilizes a pressing indenter of known material and quantifies the hardness of material being tested into a numerical readout.

Nital etch provides an alternate process for detecting hardness changes on the surfaces of HSLA steels without leaving indentations (ASNT 2016).

Nital Etch Process

The nital etch process is a systematic process with a one-directional flow that makes it very user-friendly. A solid understanding of the equipment, chemicals, and techniques used in this process can help a technician perform this process precisely, with little difficulty. One piece of equipment involved in the nital etch process is the corrosion resistant tank. These tanks can be made of stainless-steel materials such as 316L or PVC to contain hazardous chemicals. The corrosion-resistant tanks should be set up in a side-by-side layout with a directional flow to allow processing to occur undisturbed.

Another device used is a calibrated timer; this is used for timing cycles in solutions.

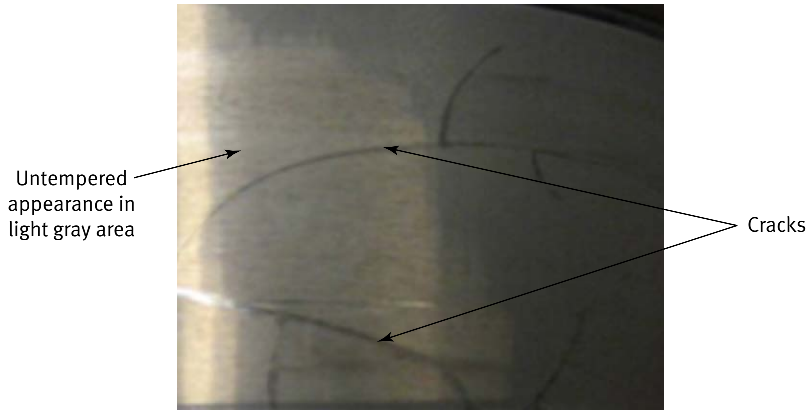

Figure 1. Severe cracking can result when a weakened metallic surface due to heat damage encounters stress.

Temperature-control devices are also used to monitor the activity of the chemicals. If there are any changes in temperature ±5 °F in the acidic bath, the activity can increase or decrease dramatically. This can lead to the over-etching or under-etching of parts. Over-etching of parts results in too much material loss and oversized tolerance holes. Under-etching results in the overshoot of specification-driven etch times and poor uniform appearances.

Tank agitation is needed to acquire a uniform etch appearance on part surfaces. Also, adequate lighting is needed for inspection purposes. Another important piece of equipment is an industrial-sized oven, which is used after inspections have been performed. These ovens are operated just below low-tempering temperatures to remove hydrogen induced by the chemical process. HSLA steels are very susceptible to hydrogen embrittlement. Hydrogen embrittlement is a condition caused by certain materials absorbing hydrogen. As a result, the absorbed hydrogen in the materials causes components to fail and fracture much below their intended ultimate tensile strength.

It is important to note that most industry specifications list a timeframe that the technician must strictly adhere to in transporting components from the last solution to inspection; then from inspection into the oven to start the stress-relief cycle to remove the hydrogen. Although an in-house laboratory or outside testing facility may not be considered equipment, they are vital for the nital etch process. These chemical laboratories conduct periodic surveys to maintain solution concentration in acceptable limits according to specifications, and often a professional chemist is hired to manage laboratory tests.

Chemistry

Nital etch is based primarily on the sequence of different chemical reactions on surfaces. The sequence of these chemical reactions applied to the components are used to obtain the result of a uniform gray surface. Variations of color other than a uniform gray can be evidence of grinding or other abuse. Chemicals involved in the process are a dilute nitric acid (3% to 5% by volume in water), hydrochloric acid (4% to 6% by volume in water), and sodium hydroxide (2% to 6% by weight in water). When chemicals are being mixed for the process, distilled or deionized water must be used to provide satisfactory etched surfaces. Using tap water containing chemicals will lead to residue and a spotty appearance, which can be very confusing to interpret. Ammonium peroxydisulfate (10% by weight in water) can also be utilized in the chemical process, although it is only used when spot or local inspection is needed. Each of these chemicals is followed by a water immersion rinse to neutralize the process chemicals. The last solution utilized on the process line is a hot water rinse, ranging from 130 to 170 °F (54.5 to 65.5 °C). The purpose of the hot water rinse is to evaporate any remaining liquid left on the component's surface. This can be facilitated by using compressed air.

Techniques

In the nital etch process, there are two ways to accomplish the etching of HSLA steel. Immersion techniques and local (spot) techniques are used to obtain an inspectable surface.

The immersion technique is demonstrated by the component being completely submerged under the surface of the process and rinse solutions for the prescribed time ranges. Understanding the relationship between the chemicals used and their corresponding time ranges will help technicians obtain the best result. The nitric acid initiates the first chemical reaction to the surface of the component while a charcoal gray appearance is obtained and then followed by a rinse (recommended immersion time of 15 to 60 s). During this chemical reaction, a small amount of the alloy material returns to the surface in a form called "smut." Hydrochloric acid removes the smut from the surfaces of the component while providing a lighter, more uniform gray appearance. This is followed by a rinse (recommended immersion time of 30 to 60 s). This chemical also displaces hydrogen into the component, so a stress-relief cycle in an industrial oven will be needed after the inspection to avoid hydrogen embrittlement. The purpose of the rinses is to neutralize the acid reaction on the surfaces. Sodium hydroxide is used to neutralize any remaining acid residue as a final fail-safe (recommended immersion time is 15 to 60 s). Any residue acids left on the surface can lead to localized corrosion pits.

The local (spot) technique is utilized when a small area of a large component requires inspection, local grinding operations have been performed, or when parts cannot be hydrogen embrittlement relief baked. Technicians will have to mask the area of interest with a corrosion-resistant material to preclude the etchant from entering unwanted areas. Industry specifications limit local process area to a 4 in. (10.16 cm) square. Ammonium peroxydisulfate helps accomplish the local etch by becoming etchant (recommended immersion time is 15 to 60 s). A small solution is created to cover the surface of the competent. Technicians must manually swab the area of interest with a cheesecloth for the recommended time range to accomplish a local etch and then rinse. The strength of this chemical is significantly reduced over time and must be discarded after 72 h. It is recommended that new chemicals be mixed before performing the local etch. The benefit of ammonium peroxydisulfate is that the hydrochloric acid does not need to be used, which in return means a stress-relief cycle is not required. Any smut residue produced is easily removed by water.

As stated previously, experience is particularly important in these techniques. When etching components, the best time is just a little over the minimum time given. The reason for this is if a non-satisfactory etch is obtained or indications are confusing to interpret, technicians may start the etching process again, provided they use the remainder of the allotted time to process. For example, if etch range is 10 to 40 s and the technician performs the etch within 12 s and the result is a poor etch surface; then the technician may reprocess the component using the 28 s not used during the first cycle. Using the time ranges listed in MIL-STD-867 for HSLA steel, the etch process will remove just under 0.00005 in. (0.00127 mm) of material from the surface of the component. This is important to take into consideration when re-etching because the component may have close tolerance features such as threaded areas, splines, or holes that will not allow a second etch. Any second etch cycle will enlarge these close tolerance features, rendering the component unusable. Technicians must be attentive to every part of the process to obtain an inspectable surface the first time around. Critical chemicals that need exact time checking are the nitric acid (15 to 60 s) and the hydrochloric acid (30 to 60 s). These two chemicals determine the quality of coloration of the surface. Using a representative sample of a known color variation representing burns (called an etching sample) is a technician's best way of obtaining times that acquire the best results. Figure 2 shows an etching sample with acceptable and unacceptable variations of gray. The time ranges given in MIL-STD-867 for sodium hydroxide and hot water rinses are not as critical as the nitric and hydrochloric acid. These times do not need to be the same for each etch cycle run.

Figure 2. An etching master provides a good indicator for the technician to follow as a baseline for the etching process.

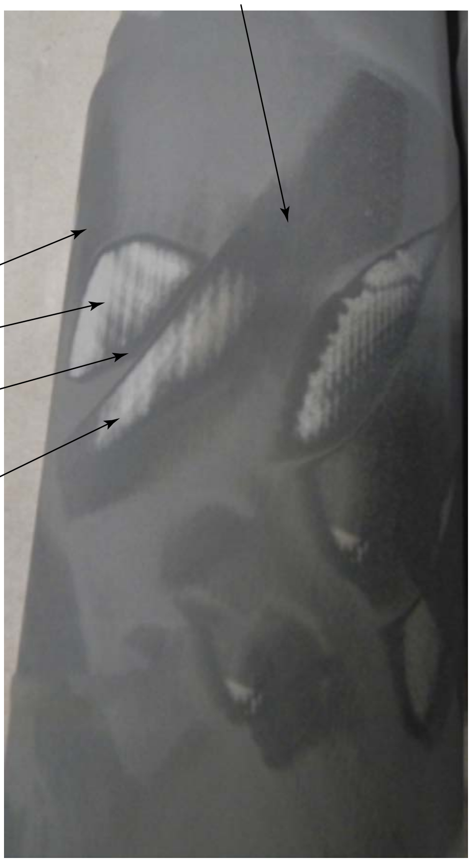

Figure 3. A component surface with varying ranges of hardness resulting from heat damage.

Inspection and Interpretation

Inspection of HSLA steel must start with having the proper lighting at the inspection table to view any indications encountered. The requirement for lighting is a minimum of 200 footcandles at part surfaces as stated in MIL-STD-867. Heat damages are the purpose of inspecting the component. Any surface that has not been damaged will result in a uniform gray appearance.

Indications found in the nital etch process can fall into two categories: overtempered and untempered. These two indications are considered rejectable conditions. Overtempered is characterized as a black indication darker than the surrounding areas. The darker aspect can be attributed to a burn-like appearance. This results in a decrease in hardness in the area of interest. Untempered areas are characterized as a light gray to white color surrounded by dark borders. Untempering mostly always occurs with overtempering at the border of the indication. Figure 3 shows a surface with untempered along with overtempered indications and the difference in hardness values (HRc) associated with the indication within the surrounding area. The higher the Rockwell value, the harder the surface, and vice versa in that lower Rockwell values lead to soft surfaces on the component.

A technician must be attentive to the entire process, especially the cleaning of the high-strength steel. Improper cleaning can lead to unetched areas or spotty appearances. This is mentioned to highlight improper cleaning that can be confused as an indication. The solution to this issue is to clean the component properly and rerun the etch process again if tolerances permit this action. If tolerances do not permit, technician should contact the appropriate engineering authority for direction on how to proceed. Figure 4 shows an example of overtempered indications encountered.

Qualification and Certification

Prior to performing any processing and etch inspection of components, technicians must undergo the qualifications process stated in SAE ARP 1923 (SAE International 2018). Qualifications state that technicians pass a physical, written, and practical examination. Once all

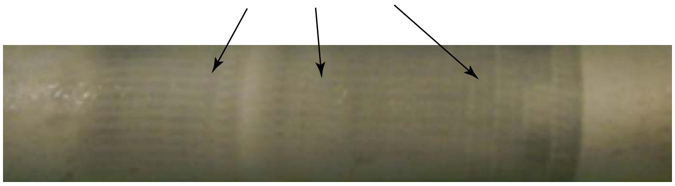

Figure 4. A test piece with rejectable indications of overtempered burns.

sections are completed with a satisfactory result of passing, technicians will be given their certification and will be considered competent to process components. In certain sections of the aerospace industry, levels may be given to technicians to designate the experience a technician has demonstrated.

Conclusions

The principle of nital etch is that untempering and overtempering are visually discernible from the nonaffected surface of the etched part. Utilizing one of the oldest and most common methods of nondestructive testing, which is visual testing, the technician can readily detect and document different variations of color encountered. The integrity of HSLA steels is maintained through a collaboration of industrial equipment, variation of acids and bases, and simplified techniques.

REFERENCES

ASNT, 2016, *Materials and Processes for NDT Technology*, 2nd ed., Chapter 2, pp. 42–47, 75, American Society for Nondestructive Testing, Columbus, OH

SAE International, 2018, ARP 1923: *Qualification and Certification of Etch Inspectors*, SAE International, Warrendale, PA

US DOD, 2019, MIL-STD-867: *Standard Practice for Temper Etch Inspection*, US Department of Defense (DOD), Washington, DC

AUTHOR

Joshua Pierre: ASNT NDT Level III and AESF Certified Aerospace Finisher, Mastic, NY; jpierre791@gmail.com