



# Sample Preservation - The Key to a Successful Failure Analysis

by Marc Pepi

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# SAMPLE PRESERVATION – THE KEY TO A SUCCESSFUL FAILURE ANALYSIS

Marc Pepi

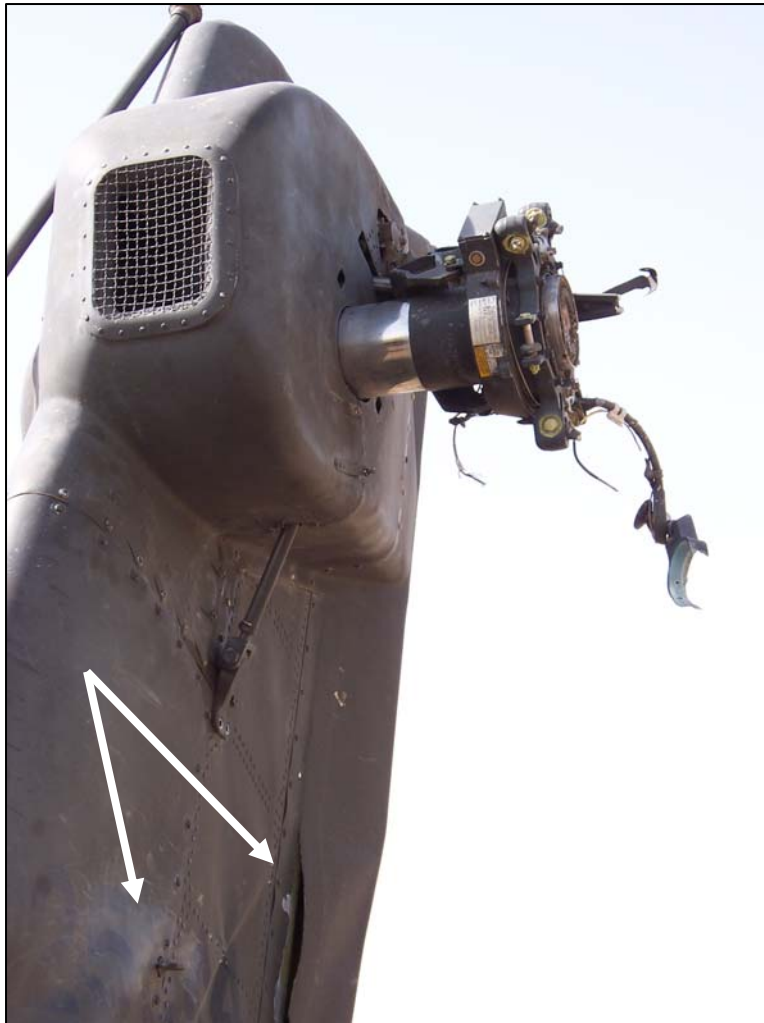
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Abstract: Probably the most important, yet least understood step in failure analysis is sample preservation. The importance of this step cannot be overemphasized. Irreversible damage can occur if the proper steps are not taken to preserve the fractured specimen and associated components. This paper discusses the techniques used to ensure samples are kept in the best possible condition for the failure analyst. Similar to a “crime scene”, every effort should be made by the crash site investigator to prevent post-mortem harm to the failed component. Although not many publications reference this subject, or discuss it in detail, examples of a literature search and practical experience will be highlighted.

Key Words: Sample preservation, failure analysis, crash site investigator, fracture surface

Background: In many instances, the results of a failure analysis are not only utilized to determine what event transpired to cause the failure, but to provide recommendations on how to avoid similar failures in the future. Some failures are of such a magnitude that a date in court is encountered pitting the victims of a catastrophic incident versus those involved in the manufacturing and subsequent processing of the failed component. What happens if the failure is misdiagnosed? Similar to a misdiagnosis given by a medical doctor, severe consequences could result. One of the most important areas in which “evidence” can be inadvertently contaminated is during component handling, especially early on in the analysis. Fracture surfaces must remain untouched so that fractographic investigators can use high magnification images to accurately determine the failure mode [1].

Photography: Crash site investigators are responsible for documentation of the event prior to human involvement (unless medical evacuation efforts were needed for personnel). In today’s age of digital photography, the number of photographs taken is no longer an issue. Still, the use of scale and correct lighting is imperative to generating photographs that are useful in telling the “story”, whether in a final report, or in front of a courtroom. Remember that a lot can be revealed by use of oblique lighting, especially when fracture surfaces are documented. Efforts will be made to distinguish the primary fracture from any secondary fractures, and photographs of the crash site will go a long way in helping sort this out. Digital photography allows the burning of the date and time into the photograph, which is recommended. Figure 1 shows a photograph of a helicopter tail rotor section that failed in service. Although no date and time is recorded on the photograph, and there is no scale marker present, the photographer does a good job of framing the tail of the helicopter to show areas of secondary damage.

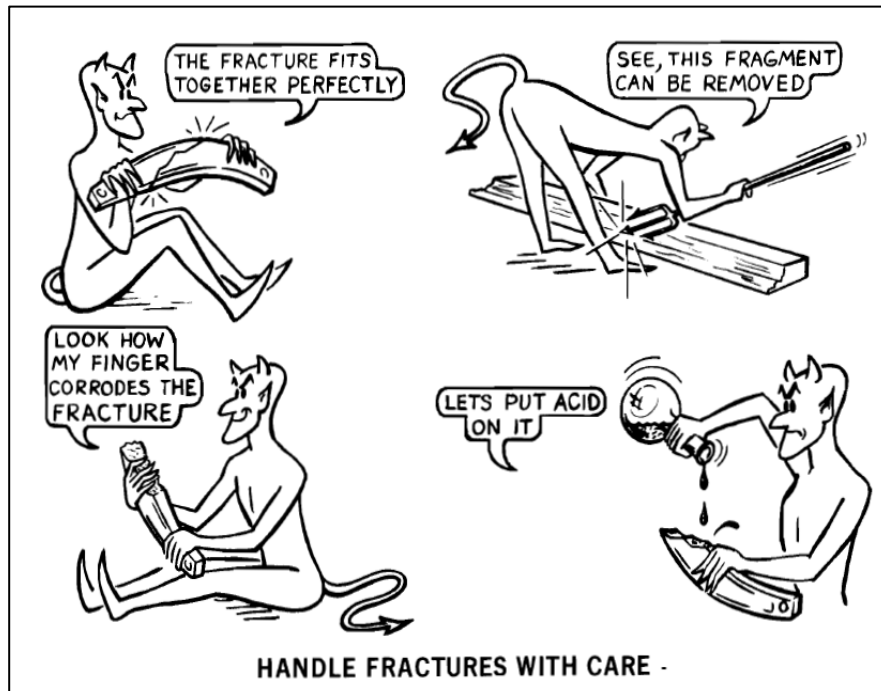


**Figure 1.** Crash site investigator photograph of the failed tail rotor of a helicopter. Note the secondary damage to the tail at the bottom of the photograph (arrows).

Handling of Fracture Surfaces: Fractures, even those of hard or high strength metals, are fragile and subject to mechanical and environmental damage that can destroy important microstructural features [2]. The fracture surface is best considered the smoking gun in any investigation; without this aspect of the failure, it is difficult and sometimes impossible to confirm a failure mode [3]. The bulk of this paper discusses preservation methods with metallic specimens in mind, since these are subject to oxidation. Care must also be taken in handling plastic, composite and ceramic fractures, which may also be sensitive to moisture and easily damaged by contact.

Although it seems like second nature to the failure analyst, it is not always obvious to the layman that metal components (yes – “metal”, not just “steel”) should be handled with caution, preferably while wearing protective gloves (even gloves have been shown to transfer a small amount of foreign material, so handling should be kept to a minimum). Figure 2 shows the types of actions that should not be performed on a failed component [4]. One aspect that is not shown in this figure is that the fracture surfaces should not be cleaned, no matter how greasy or dirty. That type of information could be critical in the future for the failure analyst.

As mentioned, even protective gloves can transfer contaminants so handling should be kept to a minimum. But gloves should be worn over the alternative of using bare hands, due to the natural skin acidity of the human hand. Although the pH of human hands varies, the skin averages a pH of 5 [5], and is compared with other items in Table 1.



**Figure 2.** What to avoid with fracture surfaces (see reference [4]).

**Table 1.** Typical pH Values (see reference [5]).

<b>pH</b>	<b>Type</b>	<b>Example</b>
0	Acidic	Battery Acid
1	Acidic	Stomach Acid
2	Acidic	Lemon Juice
3	Acidic	Vinegar
4	Acidic	Tomatoes
5	Acidic	Human Skin
6	Acidic	Milk
7	Neutral	
8	Alkaline	Sea Water
9	Alkaline	Baking Soda
10	Alkaline	Milk of Magnesia
11	Alkaline	Ammonia
12	Alkaline	Soapy Water
13	Alkaline	Bleach
14	Alkaline	Liquid Drano

Other evidence of the corrosive nature of human skin is cheap jewelry, which has been known to tarnish as a result of contact with skin. Another example is the door handle shown in Figure 3. This brass handle has actually been etched over time and contact with acids on the human hand. Compare this etching process to typical brass macroetchants used in the laboratory: a) Nitric acid + water, b) acetic acid + nitric acid, c) hydrochloric acid + ferric chloride + water or methanol, and one gets a feel for the destructive nature of human skin

Another reason to avoid skin contact lies in the fact that most fractures are subsequently examined through EDS to determine the elemental composition of the fracture surface. This becomes important, for example, if the investigator is trying to distinguish a failure mechanism as either environmentally-assisted hydrogen cracking or hydrogen embrittlement. The main difference here is the possible presence of chlorine on the fracture surface, which would be indicative of an environmentally-assisted failure. Unwashed hands in contact with the surface could potentially deposit salts that could alter the results. We always joke not to eat salty chips while performing this type of an investigation! Bottom line: touching fractures with bare hands should be avoided.



**Figure 3.** Macroetched brass door handle showing grain structure.

Visual Examination: The fracture surface should be examined with the unaided eye and at low magnification in order to determine the general location of the fracture origin. This is the most important area for the failure analyst, and will be the focus of the subsequent examination. Confirm the location of the fracture origin with a second opinion, as necessary, prior to sectioning. Based on the location of the fracture origin, areas for sectioning can be identified for subsequent metallography, chemical analysis, and mechanical property determinations [6]. Remember to stay as far away from the fracture origin as possible.


Preservation Techniques: Fractures must be handled with great care from sampling through analysis. The fresh fracture should be protected as soon as practical to prevent the onset of oxidation, which can occur very quickly on newly exposed metal surfaces. However, wrapping them directly into a plastic bag or placing pieces directly in a plastic bottle or container can introduce unwanted hydrocarbon contaminants [7]. Reference [7] suggests wrapping each individual fracture in aluminum foil to prevent such contamination. Loosely covering the fracture in a dry environment versus the alternative of sealing it closed in an air-tight container is also recommended. The latter may act to trap moisture, and actually do more harm than good. After loosely wrapping in foil, bubble wrap can be used for further protection. Woven materials are not as well suited; they provide less cushion, absorb more of the deposits and are likely to transfer fibers onto the failed surfaces [8]. In addition, the failed parts should not be exposed to uncoated brown Kraft paper or cardboard, as the moisture, acid and sulfur that potentially exists within these products can deleteriously affect the fracture surface.

Always ensure that fracture surfaces do not come in contact with any other sample while stored, since any mechanical damage to the surface may affect future findings on the scanning electron microscope (SEM). Coating in oil may protect against the onset of corrosion, however, may also affect the results of energy dispersive spectroscopy (EDS) at a later time. If oil is chosen, it must not contain any element that will chemically attack the fracture surface, and should not be used if corrosion is the likely failure mechanism. If it is known initially that EDS will not be performed, a coating such as Krylon Crystal Clear Spray Coating No. 1302 can be applied [4] to protect the fracture surfaces. This coating can be subsequently removed with alcohol when fractography is performed, although problems may exist with rough fracture surfaces. For these surfaces, a cellulose acetate strip (or “replica”) can be applied, which has the advantage of being available in thick cross sections. Reference [9] suggests considering the use of corrosion-inhibiting paper to package samples.

Studies have shown that some solvent-cutback petroleum-based compounds can be utilized that not only prevent chemical attack to the fracture surface, but can be removed completely without leaving any trace [6]. One such compound is Tectyl 506 which, in a study with other coatings, protected steel fracture surfaces after exposure to 100% humidity and 100°F for 14 days. After environmental testing, the compound was removed ultrasonically in a naphtha solution, and subjected to SEM analysis, where the sample was shown to exhibit no attack and contain no residue.

Fracture Surface Cleaning: Before a fracture surface is cleaned, it is important to understand the consequences of such a decision. If future EDS becomes necessary, the surface will not be representative of the conditions leading to the failure. If it is anticipated that failures have occurred as a result of either stress corrosion cracking (SCC) liquid metal embrittlement (LME) or corrosion-related in general, the fracture should not be cleaned before the surface debris is analyzed. If the decision is made to clean the fracture surface, References [6] and [10] list common techniques for fracture surface cleaning in order of increasing aggressiveness (Table 2):

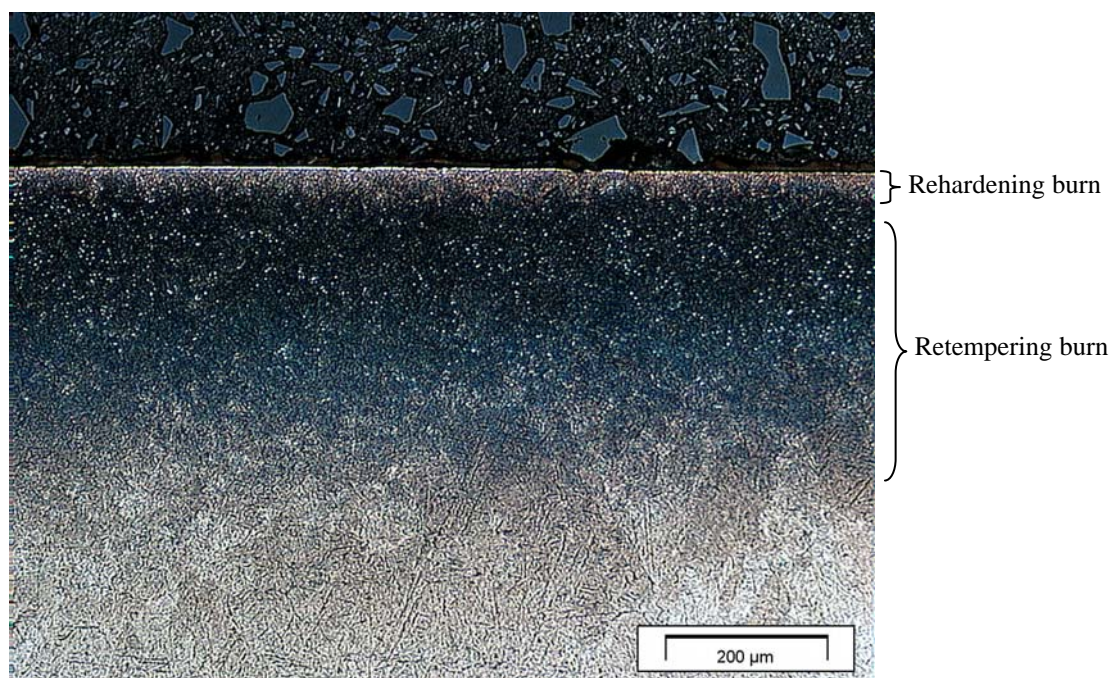
**Table 2** Methods for Cleaning Metallic Fracture Surfaces.

<b>Method</b>	<b>Removal of...</b>	<b>Aggressiveness</b>
Dry air blast or soft organic-fiber brush cleaning	Loosely adhering debris and dust	Least
Organic-solvent cleaning in ultrasonic bath <ul style="list-style-type: none"> <li>• Toluene or xylene</li> <li>• Ketones</li> <li>• Alcohol</li> </ul>	<ul style="list-style-type: none"> <li>• Oil and grease</li> <li>• Varnish and gum</li> <li>• Dyes and fatty acids</li> </ul>	
Replica stripping	Insoluble debris and oxides	
Detergents (i.e. Alconox®)	Corrosion products and oxides	
Cathodic cleaning	Deposits and oxides	
Corrosion-inhibited acids (i.e. Rodine® 213)	Sulfides and oxides	
Acid etches	Oxides	Most

Always start with the least destructive method, and monitor the results before stepping up to the next method.

**Sectioning:** Sometimes we are faced with the situation of having to section a failed part off of the original equipment for subsequent analysis. Records should be kept, including both notes and photographic documentation, before any sectioning is performed. If dry cutting is not feasible, the fracture surface must be protected from cutting fluids to avoid contamination. Dry cutting is best in avoiding these contaminants, yet could lead to altered microstructures based on the heat generated if the cut is close to the areas to be subsequently examined. Flame cutting is often used for large parts, but again, caution must be used to stay far from the fracture surfaces and areas to be examined. Figure 4 shows the damage that occurred as a result of abusive grinding a carburized tool steel. The outer surface rehardened, transforming into unstable, untempered martensite. The case below this layer was affected; becoming retempered which resulted in a loss in hardness. Although abusive grinding caused this damage, the same could result with abusive abrasive sectioning depending on the heat generated, and proximity to subsequent metallographic samples.

Subsequent sectioning may also be required to produce a sample that can be accommodated by the scanning electron microscope, hardness tester, metallographic mount and for chemical analysis. The same techniques listed above should be employed for secondary sectioning. If using a hacksaw, be sure to feel the sample after short periods of cutting to ensure that heat generation is not a problem. We can comfortably touch something for extended periods of time as hot as 52°C (~125°F) [11], which will not cause any problems with most engineered components.



**Figure 4.** Rehardening and retempering burn in a carburized tool steel subjected to abusive grinding.

**Litigation:** High visibility failures leading to catastrophic damage and/or injury or loss of life will no doubt end up in litigation. The most valuable asset of any fault/failure type forensic investigation is properly preserved physical evidence, whether positive or negative [12]. If samples are not preserved properly during these court proceedings, irreversible damage can occur that may result in undesired decisions. Based on this, it is common practice for lawyers to insist that metallic evidence be stored in a vapor phase corrosion inhibitor (VPCI). This mitigates the risk of continuing oxidation damage and, also as important; it does not alter the chemical composition of the preserved item. One other thing to worry about for a sample subjected to litigation: if the chain of custody on the piece of evidence is broken, it becomes inadmissible in court! In the final analysis, failing to protect the structure or delaying implementation of a protection procedure can have devastating effects [13].

**Informal Survey:** A dozen well-known failure analysis laboratories across the country were polled in order to see which sample preservation methods they used. Some of the most popular responses are listed as follows:

- Take photos with a measurement scale in the as-found condition
- Don't clean fracture surfaces
- Don't introduce contaminants
- Avoid starting the investigation in the field
- Get samples dry – and keep them dry
- Use plastic bags with desiccant for sample preservation

- Wrap samples in clean rags
- Coat fracture with 30 weight oil

It was interesting to see the similarities with the preferred methods described herein, as well as the differences (coating in oil, and the use of rags are not always preferred).

Preservation Checklist: The following checklist was generated as a result of a literature search on this topic. The following references were used to generate this checklist: [1], [2], [14], [15].

1. Photograph: Document the failure scene and failed component(s) with photographs, including wide angle, and zoom shots; remember you can never have enough photographs. If the failed part requires disassembly, capture this process through photography.
2. Preserve: Remember - Don't touch anything. Avoid touching the sample or area of interest with bare hands. If you must use your hands, wear gloves and keep handling to a minimum. Look for secondary damage caused by the failure and document it.
3. Do not clean the failed component.
4. Do not try to fit mating fracture surfaces together.
5. Choose samples that are representative of the failed component.
6. Preserve the sample integrity; cutting fluids will contaminate a fracture surface and abusive sectioning will alter the prior heat treatment.
7. Preserve the fracture surface; if two mating surfaces are in your possession, sectioning should only be performed through one of them, and only if necessary. Store these samples in clean containers.
8. Avoid tape, as the adhesive may leave a film on the surfaces of the samples in contact with it.
9. Clearly identify the containers with the part number, or other description of the component under investigation.
10. Prepare: A listing of operating conditions and the manufacturing process background of the failed component should be made available to the failure analyst.

Conclusion: Sample preservation is one of the most important aspects of a failure investigation, and care should be taken to provide the failure analyst with a sample in the best possible condition for accurate assessment of the failure scenario.

References:

- [1] "Strategies for Failure Analysis", R. Graham, ASM Advanced Materials & Processes, August 2004.
- [2] [http://www.kaker.com/defects\\_on\\_rolls/testing/sample\\_preservation\\_handling.html](http://www.kaker.com/defects_on_rolls/testing/sample_preservation_handling.html).
- [3] Plumbing Engineer, August 2002, "Discovering Why Pipes Fail", Steve Ferry.
- [4] Electron Fractography Handbook, MCIC, Battelle, June 1976, p. 1-4.
- [5] <http://www.celibre.com/sksacid.aspx>.
- [6] "Preparation and Preservation of Fracture Specimens", R.D. Zipp and E.P. Dahlberg, ASM Handbook Volume 12, Fractography, 1992, p. 72.
- [7] Outlook, Vol. 20 No. 3, "In the Unlikely Event of a Failure", By Ron Graham, [http://www.wahchang.com/pages/outlook/html/bkissues/20\\_03.htm](http://www.wahchang.com/pages/outlook/html/bkissues/20_03.htm)
- [8] Guidelines for Forensic Analysis of Failed Parts, R.A. Page, December 2000, p. 11.
- [9] "Practices in Failure Analysis", ASM Handbook Volume 11, Failure Analysis and Prevention, 2002, p. 397.
- [10] Metallurgical Failure Analysis, C.R. Brooks and A. Choudhury, McGraw Hill, Inc., 1993, p. 58.
- [11] "Process Control of Temperature", NDT Resource Center, <http://www.ndt-ed.org/EducationResources/CommunityCollege/PenetrantTest/QualityProcess/TemperatureQC.htm>
- [12] Evidence Storage V. Evidence Preservation, [www.EvidenceMaster.com](http://www.EvidenceMaster.com).
- [13] Forensic Engineering, ISI Publications, Inc., 1995, p. 18.
- [14] Field Guide for Failure Analysis, Gas Machinery Research Council, [www.gmrc.org](http://www.gmrc.org).
- [15] "NTSB Aviation Investigation Manual, Major Team Investigations", Appendix J, November 2002.

The following references, although not used herein, may also be helpful to the reader:

- a. ASTM E860, "Standard Practice for Examining and testing Items That Are or May Become Involved in Litigation", 1997.
- b. ASTM E1188, "Standard Practice for Collection and Preservation of Information and Physical items by a Technical Investigator", 2005.
- c. ASTM E1459, "Standard Guide for Physical Evidence Labeling and Related Documentation", 1998.
- d. ASTM E1492, "Standard Practice for Receiving, Documenting, Storing and Retrieving Evidence in a Forensic Science Laboratory", 1999.
- e. ASTM E2332, "Standard Practice for Investigation and Analysis of Physical Component Failures", 2004.

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