Analysis for Failure, Damage, and Fracture

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1 Introduction

The field of metallurgical failure analysis encompasses all of metallurgy and materials science and engineering, from raw material production to end product use. There is an extremely wide variety of factors that contribute to damage accumulation and failure in components and systems, including types of parts, alloys, post-manufacture treatments, service conditions, types of loading, applications, environments, and a myriad of combinations of all of these. In addition, there are nearly unlimited questions that can be asked and determinations that can be requested with regard to the failure and/or damage exhibited by components.

2 Scope

This document applies to case working personnel using the associated instrument(s) and supporting equipment in support of metallurgy examinations.

An extremely wide variety of metallurgical factors produce observable and measurable characteristics that can be used to identify and compare evidentiary items. The following procedure outlines the basic analyses most commonly performed to examine an item to ascertain its origin or evaluate an association to other item(s).

3 Principle

Failure analysis is fundamentally an engineering-based investigation of an event or a series of events. By applying engineering principles, an item can be examined to determine if fundamental design problems exist or if a defect may have been artificially introduced. For example, a sharp change in cross-section can result in a stress concentration and crack development under conditions not otherwise expected for the applied stresses. Fracture mechanics permits analysis of such a stress concentration to predict its potential effects on the component's operating life. Often one or more exemplars that share some characteristic(s) with the evidentiary items can provide useful information.

Fractography can be applied to determine the mechanism responsible for crack growth and to determine the crack initiation site. Some fracture mechanism examples include tensile overload, fatigue, stress corrosion cracking, and creep failure. Each fracture surface also provides information on the nature of the applied stress (i.e., torsional, shear, tensile, mixed mode). The metallographic and/or compositional analysis of an object may reveal deficiencies in material or manufacturing process that contributed to failure. Other methodologies, such as magnetic particle testing and radiography, are also available for exposing defects and damage throughout a component.

Items suspected of being from a single original object are compared against each other in their relevant compositional and physical characteristics using appropriate examination techniques. These techniques typically include visual and microscopic examination of manufactured features and damage characteristics, dimensional measurements, and compositional analysis of the items. Two items which are not distinguished from each other on these bases are considered to demonstrate an association and possibly a common origin. For example, a segment fractured from a knife would be expected to have the same alloy composition, edge geometry, spine thickness, and surface finish as the part of the blade from which it fractured. These features alone, though, indicate only that the fragment is possibly from the same product type. Correspondence of the damage characteristics on the separated surfaces is required to conclude that two items were once attached.

4 Specimens

Nearly any metal object and many nonmetallic objects can be examined using the techniques outlined in this procedure. Exemplars for evidentiary items may be obtained and examined to establish the expected variability of manufactured characteristics.

5 Equipment

A list of items commonly used in these examinations follows. Not every item is used for all failure and damage investigations. The instrumentation and equipment used will depend on the nature of the evidence to be examined and compared.

- Photography equipment for macro- and micro-documentation
- Observation enhancing tools, such as:
  - borescope, magnifying glass, jewelers' loupe
  - light microscopes (stereomicroscope, digital microscope, comparison microscope)
  - scanning electron microscope (SEM)
- Digital radiography system*
- Measurement tools, such as:
  - micrometers, calipers, measuring tape
  - optical measuring microscope (e.g., SmartScope FOV*)
  - balances
  - magnet
- Miscellaneous hand tools
- Certified reference materials (CRMs), reference materials, and standardization materials as needed
- Digital multimeter
- Specimen cleaning and protection equipment and materials:
  - compressed air
  - lint free wipes
  - cleaning brushes
  - cellulose acetate replication tape
  - EvapoRust™ rust remover
  - Solvents (such as water, alcohol, petroleum ether, or hexane)
  - ultrasonic cleaner
  - desiccant
  - vacuum chamber
- Compositional analysis instruments:
  - Energy dispersive x-ray fluorescence spectrometer (EDXRF)*
  - Spark discharge-in-argon optical emission spectrometer (SDAR-OES)*

    - Scanning electron microscope with energy dispersive x-ray spectrometer (SEM/EDS)
- Metallographic specimen preparation and examination equipment*
- Non-destructive testing equipment, such as:
    - magnetic particle inspection equipment
    - liquid dye penetrant (LDP) and developer
    - ultrasonic inspection equipment
- Mechanical testing instruments, such as:
    - Hardness* and microhardness* testers
    - Tensile*, torsion, fatigue, impact, and wear testers

* When an instrument marked with an asterisk is used, see the appropriate Chemistry Unit (CU) Metallurgy technical procedure for additional equipment.

6 Standards and Controls

The standards and control materials used in this procedure will depend on the specific analytic methods employed and the nature of the item under analysis. Any instrument used in this procedure will employ such standards as required under its specific technical procedure. Exemplars for evidentiary items may be obtained and examined to establish the expected variability of manufactured characteristics.

7 Sampling

Visual examinations are performed on every item examined under this protocol. Further testing is based on the suitability of individual items, or portions of items, for relevant examination techniques. Case notes will describe which examinations were performed on which items. If initial examinations reveal that an analyzed characteristic may vary on a single item, the means of selecting a location to test the characteristic will be noted in the case file.

Samples or sections may be taken from the items for analyses of coating(s), substrate material(s), corrosion product(s), deposits, contaminants, or any other material relevant to the determination(s) requested. Destructive specimen removal must be approved by the contributor prior to modification of the evidence.

If an item contains multiple visually indistinguishable objects that are suitable for one analysis technique, a subset may be selected for testing by non-statistical or statistical means. For failure analysis, sample selection is typically based on observable damage, thus is non-statistical in nature. The manner of specimen selection will be recorded in the case notes.

A. For non-statistical specimen selection, the report will attribute the measured characteristic only to the specimen(s) tested. This can be facilitated by sub-dividing the evidence and reporting the specific analysis results for the sub-divided portion only.

B. If a sampling plan will be used to make an inference about the entire set of visually similar items, then the plan will be based on a statistically valid approach. See METAL-210 (Sampling section.)

8 PROCEDURE

8.1 Analysis

The following analysis sequence was derived from guidelines established by ASM International and augmented for forensic metallurgical applications. Except for initial visual examinations and photography, the following steps and/or tests are not required in every situation and will vary depending on circumstances and the evidence. Additionally, the sequence below serves only as a general guideline. The examinations selected and the sequence in which they are performed should be established by the nature of the evidence and the facts and circumstances of the case. Data gathered during examinations will be included in the case notes.

A. (Required) Perform a preliminary visual and low magnification microscopic evaluation of the item(s) to evaluate the fabrication method(s), coating, marks from service use/abuse, type(s) of failure and damage, possible contamination, and any other characteristics deemed to be of value. Care should be exercised to ensure that mating fracture surfaces are not brought into contact with each other to “see if they fit” to avoid possible destruction of valuable surface information.

B. (Required) Photograph submitted or in situ items in the “as received condition” (ARC). These photographs will record component positions, in situ conditions, fracture and failure orientation relative to its environment and to other components, service conditions, service abuse, and any other characteristic, condition, or information to be considered during the failure/damage analysis. Additional photography should be conducted during the metallurgical examinations to record any features or characteristics upon which a conclusion is likely to be based. Whenever practicable, include a scale in the photograph or apply a verified micron marker to the photograph. See section 8.2.1 Photography.

C. Acquire and analyze documentary information to assist in the reconstruction of the sequence of events leading to the damage and/or failure. While such documentation can help to narrow the focus of an analysis and provide useful guidance, it is not a substitute for the physical evidence generally required to definitively establish a failure mode. Ordinarily, such information is collected by the submitting agency and may include fabrication, manufacturing and processing information; service history; interviews of eyewitness individuals; interviews with individuals whose duties, behavior or failure to act may have induced, or may have affected, the material behavior in question; as received item photos; site/in situ photographs; repair history; environmental details (e.g., temperatures, loading conditions, load magnitude(s), environment chemistry); and similar component history.

D. Prior to any specimen cleaning or specimen removal, perform visual and low power magnification examinations of fracture surfaces, secondary cracks, relevant surface phenomena, gross deformation, thermal damage, and any other metallurgical or environmental characteristic deemed appropriate.

E. Evaluate the physical properties of the items by measuring appropriate features, such as dimensions, mass, and magnetic response.

   1. Use calibrated , traceable measurement tools for significant measurements. See section 8.2.3 Verify Instrument Performance.

   2. See METAL-320 for precision optical measurements using the SmartScope.

F. Collect specimens from the items for chemical analyses of coating(s), substrate material(s), corrosion product(s), deposits, contaminants, or any other material relevant to the determination(s) requested.

   1. Specimen removal should be conducted causing as little damage as possible. Record with notes and photographs any information derived from the preliminary examinations which may potentially be needed to reach a conclusion but could be considered damaged or eliminated due to specimen removal. Provide adequate protection of all fracture surfaces and damaged regions to prevent contact with each other, with other portions of the same component, or with other objects or items during transport and/or examination. If appropriate and feasible, package with desiccant to reduce degradation by corrosion.

   2. It may be necessary to collect specimens prior to, or between stages of, cleaning. Analyses such as EDXRF and SEM/EDS can often be helpful in identifying the chemical compounds present on a failed component. (See section 8.3 SEM/EDS.)

G. Clean specimen(s) using methods that progress from least to most aggressive (see Table 1 for examples) until contaminant is removed. Preserve any replica(s) or contaminant/debris for appropriate analysis.

Table 1. Specimen Cleaning Procedures

Increasing Aggressiveness	Activity
Less aggressive	Dry air blast
	Soft artist’s brush
	Stiff non-metallic brush
	Aqueous rinse
	Organic solvent (e.g., isopropanol, hexane)
	Chelating Agent (EvapoRust™)
	Ultrasonic cleaning
More aggressive	Plastic replication

H. After cleaning, perform additional visual and low power magnification examinations of fracture surfaces and other relevant metallurgical characteristics. Stereomicroscopic and SEM examinations may be performed to further characterize fracture surface features and any exogenous material present.

I. Perform qualitative or quantitative compositional analysis of any materials which may assist in evaluating damage characteristics or determining cause(s) of failure.

   1. Qualitative compositional analysis includes comparing the spectrum of an item of interest to that of another item, reference material, or specification to identify substrate or coating alloy class or debris composition.

      i. For EDXRF, see METAL-410 and the appropriate associated instrument-specific technical procedure.

      ii. For SEM/EDS, see section 8.3 SEM/EDS.

   2. Quantitative compositional analysis may be used to report specific alloying content (including estimated measurement uncertainty) or to evaluate whether contaminants are present in the substrate material at a deleterious level.

      i. For SDAR-OES, see METAL-410 and the appropriate associated technical procedure for the material and size of the item(s).

      ii. For quantitative application of EDXRF or SEM/EDS, validate an appropriate technique in accordance with FBI Laboratory requirements and CHEM-100 for the specific alloy matrix and analyte(s) of interest.

J. Nondestructive testing (i.e., magnetic particle, LDP, ultrasonic, x ray, and other electromagnetic evaluations) may be performed following the appropriate technical protocol. Non-destructive testing can find unopened cracks. Such cracks can sometimes be used to make inferences about the failure mode when the fracture surfaces are too damaged to allow interpretation. They can also be used to determine how widespread a failure mechanism is in a given system. For example, a fracture in a gas pipeline may be accompanied by other, unopened cracks. If unrepaired, these can potentially lead to additional failures following repair of the known defect. (See METAL-330.)

K. Destructive testing (i.e., hardness, tensile, impact testing) may be conducted to characterize mechanical and material properties. (See METAL-510, METAL-520, and METAL-530.)

L. Metallographic examinations may be performed either before or after mechanical testing for the evaluation of inclusions; microstructural segregation or inhomogeneities; decarburization; carbon pick-up; improper heat treatment;

corrosion; grain size; type, distribution, and morphology of microstructural constituent(s); or other characteristics present of metallurgical interest. (See METAL-450.) Supplementary examinations of metallographic specimens by SEM/EDS can be used to further characterize phase distribution and identify elements present in different microstructural regions of interest. (See section 8.3 SEM/EDS.)

M. Evaluate the data and facts accumulated to determine the fracture mode or cause(s) of the damage exhibited.

N. If appropriate, a mathematical analysis of mechanical factors leading to fracture may be used to:

   1. predict flaw size which caused catastrophic fracture at a load below that expected to cause failure

   2. evaluate manufacturing flaws

   3. establish a quantitative framework for evaluating structural reliability

   4. assist in the design and prediction of service life

O. When the history and service conditions of the evidentiary specimen are known, test exemplar specimens under similar conditions to simulate expected damage.

P. Report findings after evaluation of all gathered data.

Q. Although it is not typical for criminal cases, it may be prudent to suggest corrective measures to prevent future failures.

8.2 Supporting Operations

The following additional instrumental conditions will be applied:

8.2.1 Photography

Macro- and micro-photographs will contain a reference scale whenever feasible, however these are included for general reference, and measurements will not be made from the images. Micron markers that are automatically generated by camera or microscope software are to be considered approximate and also will not be used to measure features within the image unless the marker is verified against a calibrated scale.

8.2.2 Sectioning

When possible, cutting and grinding operations will be lubricated to prevent overheating that can change the metallurgical characteristics of the specimen. If lubrication is not possible, the metallurgical changes imparted by the process must be considered during analysis.

8.2.3 *Verify Instrument Performance*

A. The following instruments will be verified according to the appropriate Chemistry Unit technical procedure prior to their first use to acquire significant quantitative data on any given day:

   - traceable micrometers/calipers – these instruments are calibrated annually by a service provider that meets FBI Laboratory requirements. Perform the daily verification on calibrated gauge blocks of appropriate size for the particular instrument, typically 0.05 inch, 0.5 inch, and either 1 inch or 2 inch. Compare the measurements to acceptance criteria posted on the instrument-specific log sheet. Record the verification data and PASS/FAIL status on the instrument log.

   - traceable balances – per CHEM-100

   - SmartScope – per METAL-320

   - Universal mechanical test machine – per METAL-530

   - Hardness tester – per METAL-510

   - Microhardness tester – per Metal-520

   - SDAR-OES – per METAL-400 and the appropriate associated material-specific technical procedure

B. The following instruments will be verified according to the appropriate Chemistry Unit technical procedure prior to their first use any given day. (These instruments are not typically used to report quantitative data for metallurgy examinations. However, should a validated quantitative method be developed, the method will include steps to perform a quantitative verification on CRMs.)

   - XRF – per METAL-410 and the appropriate associated instrument-specific technical procedure

   - SEM/EDS – per section 8.3 SEM/EDS

8.3 SEM/EDS

Compositional analysis by SEM/EDS will be conducted as follows:

A. Prior to the first use to acquire case data on any given day, run the instrument performance verification routine according to IOSS-771. File one copy with the instrument performance records and retain one copy in the case notes.

B. Prepare and insert the specimen(s) ensuring electrical continuity with the specimen stage.

C. Adjust the instrument conditions to image the region of interest for analysis. Backscattered electron imaging can be helpful to locate features that differ in mean atomic number from their surroundings.

D. Acquisition duration will depend on the conditions chosen and the specimen area exposed to the incident beam. The acquisition time can be extended to optimize spectrum clarity or shortened to enhance collection efficiency based on the case requirements.

E. Label the elemental peaks on the acquired spectrum, considering peak shapes and energy positions, the relative heights of adjacent peaks and system-generated peaks. Many SEM/EDS systems have software that can accurately identify the escape and sum peaks in a spectrum. The peak identification system resident in the instrument software can be augmented by analyzing CRMs of similar composition to the specimen of interest.

F. Ensure the instrument identification and the operating parameters are recorded on the printed spectra or elsewhere in the case notes.

8.4 Reporting Results

Report the results of metallurgy analysis for Failure, Damage, and Fracture in accordance with METAL-210 (Reporting Results section.)

9 CALCULATIONS

In many instances, no calculations are required to perform this procedure. However, a wide range of possible calculations can be encountered in a failure analysis. These are case-specific and may include determination of the applied stresses and strains, local stress distribution at a crack tip, and mechanical properties, as well as calculating other material, loading, and environmental conditions. Some engineering resources that contain useful information for calculations based on solid mechanics, fracture mechanics, and mechanical behavior of materials are listed in section 14 References .

Calculations associated with the use of a particular instrument will be found in the appropriate technical procedure.

10 ACCEPTANCE CRITERIA

10.1 Instrument Performance

For instruments that require verification, standardization, or energy adjustment, a copy of the appropriate record(s) will be included in the case notes. For each instrument noted (*) in section 5 Equipment, follow the procedures in the appropriate Chemistry Unit Metallurgy technical document.

10.2 Failure Analysis Conclusions

The conclusions derived from this procedure are based on careful interpretation of all factual information gathered from completed testing and investigation. If a unique scenario does not explain the failure, all possibilities deemed relevant by the examiner will be reported in the conclusion. Conclusions will be expressed in reports and testimony according to current FBI Laboratory requirements.

10.3 Comparison Analysis Conclusions

Analysis for failure, damage, and fracture contributes to evaluating whether two or more items or materials were once part of the same object. 'Fracture fit' is an examiner's conclusion that two or more metallurgy items or materials were once part of the same object. This conclusion is an examiner's decision that two or more metallurgy items or materials show sufficient correspondence between their observed characteristics to indicate that they once comprised a single object and insufficient disagreement between their observed characteristics to conclude that they originated from different objects. This conclusion can only be reached when portions of two or more metallurgy items or materials physically fit together.

11 Measurement Uncertainty

When gathered, quantitative data are generally used for comparative purposes, as detailed in the Calculations section. Expanded measurement uncertainty should not be used for these inter-comparisons because it increases the probability two samples will appear to be analytically indistinguishable and therefore increases the likelihood of type II errors (false inclusion). Instead, the variances associated with the samples and with data acquisition are accommodated by the statistical comparison.

If it is necessary to estimate the measurement uncertainty of an instrumental result, it will be done in accordance with CHEM-100. Instrumental measurement uncertainty is addressed in the individual instrument technical procedures and will be calculated and reported when appropriate.

12 Limitations

The limitations of a particular failure analysis are determined by the type, amount, and condition of objects(s) being analyzed, the available background information, the specific examinations required, and subsequent determinations made, thus cannot be predicted within this protocol. Limitations encountered during the examinations will be recorded in the case notes.

13 Safety

A. Wear an x-ray film badge or dosimeter when operating instruments that generate x-rays. The instruments have protective enclosures and internal safety interlocks to prevent inadvertent x-ray radiation exposure. Never bypass or disable safety interlocks on instruments.

B. Wear personal protective gear and use engineering controls that are appropriate for the task being performed (e.g., safety glasses when cutting and chemical fume hood when etching). Electrical or mechanical hazards may require special precautions (e.g., grounding to prevent electric shock or wearing a face guard to prevent impact from flying debris.) Review instrument technical procedures and pertinent Safety Data Sheets (SDS) prior to conducting examinations.

14 References

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- METAL-210, Examinations for Association and Origin, Chemistry Unit, latest revision

- METAL-221, Functionality Examinations, Chemistry Unit, latest revision

- METAL-222, Examinations of Timing Mechanisms, Chemistry Unit, latest revision

- METAL-223, Lamp Bulb Examinations, Chemistry Unit, latest revision

- METAL-320, Operation of the SmartScope FOV Video Measurement System, Chemistry Unit, latest revision

- METAL-330, Digital Radiography, Chemistry Unit, latest revision

- METAL-400, Compositional Analysis by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES), Chemistry Unit, latest revision

- METAL-401, Analysis of Carbon and Low Alloy Steel Samples by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES), Chemistry Unit, latest revision

- METAL-402, Analysis of Small Carbon and Low Alloy Steel Specimens by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES), Chemistry Unit, latest revision

- METAL-403, Analysis of Copper by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES), Chemistry Unit, latest revision

- METAL-410, Compositional Analysis by Energy Dispersive X-Ray Fluorescence Spectrometry (EDXRF), Chemistry Unit, latest revision

- METAL-411, Operation of the Thermo QUANT'X X-Ray Fluorescence Spectrometer, Chemistry Unit, latest revision.

- METAL-412, Operation of the Bruker M4 Tornado X-Ray Fluorescence Spectrometer, Chemistry Unit, latest revision.

- METAL-413, Operation of the Olympus Delta Handheld X-Ray Fluorescence Spectrometer, Chemistry Unit, latest revision.

- METAL-450, Metallography, Chemistry Unit, latest revision

- METAL-510, Rockwell Hardness Testing, Chemistry Unit, latest revision

- METAL-520, Microhardness Testing, Chemistry Unit, latest revision

- METAL-530, Operation of the Instron Model 3382 Universal Testing Machine, Chemistry Unit, latest revision

- IOSS-771, Performance Monitoring Protocol (QA/QC) for the Scanning Electron Microscope (SEM) / Energy Dispersive X-ray Spectrometer (EDS), Chemistry Unit, latest revision

15 Revision History

Revision	Issued	Changes
03	09/30/2022	Revised to comply with new formatting requirements. Removed expository information to retain as training material. Relocated information among Introduction, Scope, and Principle sections. Added compositional analysis details. Added Reporting Results section. Removed informational references. Added references to technical procedures.