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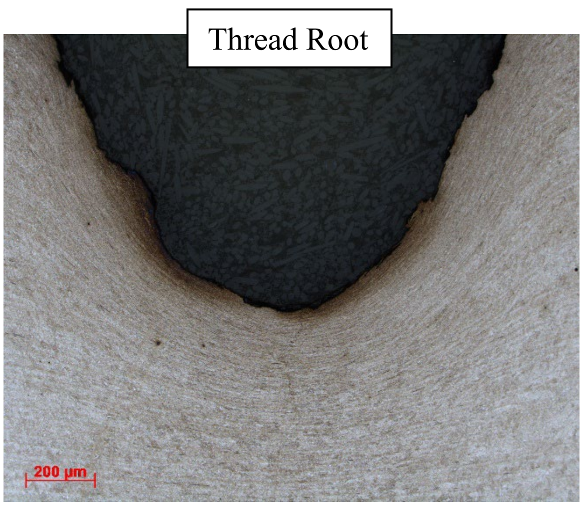
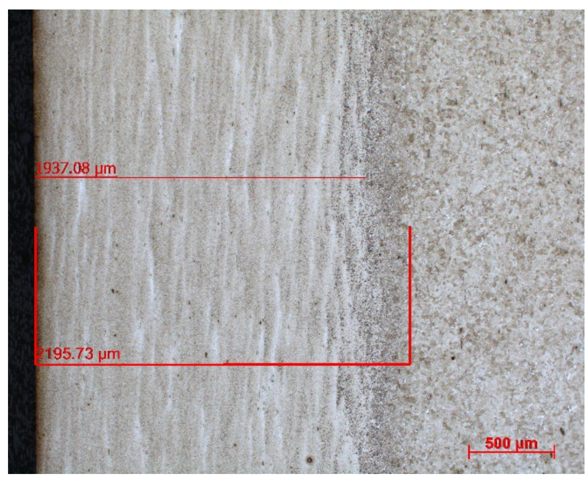
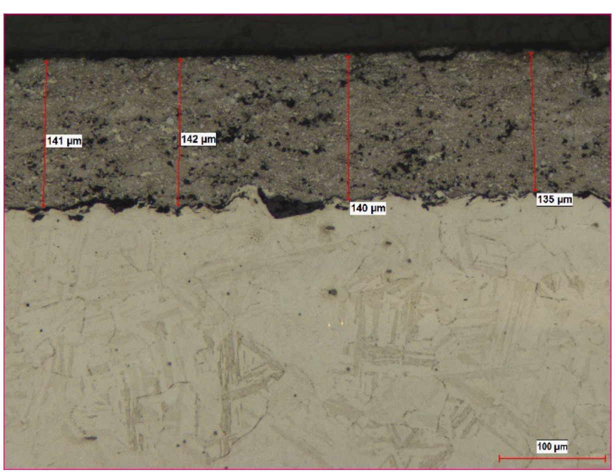
General Laboratory Tools for Metallurgical Failure Analysis

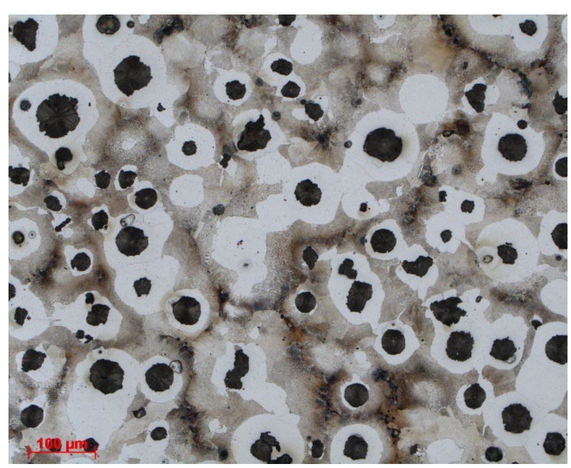
Intro: Each metallurgical failure analysis requires a different approach to finding the mode of failure and its root cause. There are several laboratory tools that can be used to perform the investigation and typically it is the results of these early studies that guide which further investigations are required. The following paragraphs list some of the more common methods, with an understanding that sometimes a need for more specialty tools is required.

Light Microscopy: Studying crack propagation, corrosion progression into the metal, bonding of coatings, etc, can be perfomed using light microscopy. Initial studies are typically made unetched (especially looking for creep voids) and then proceed on to etching to bring out the microstructure or other particular features.

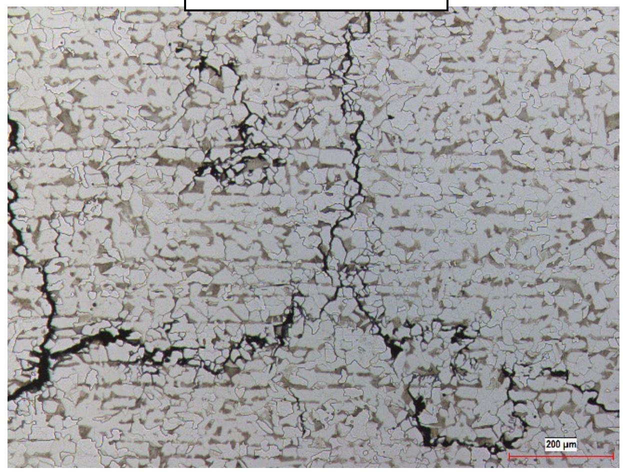






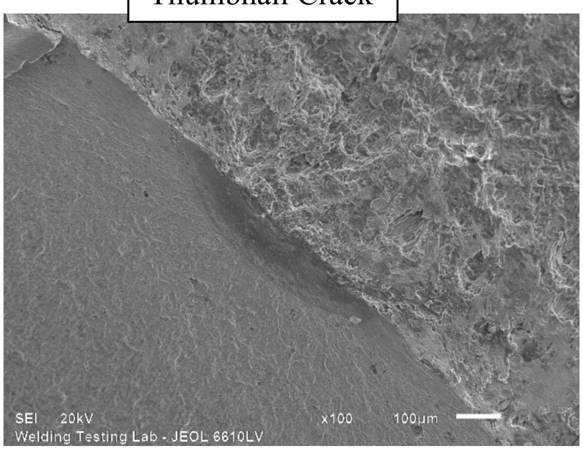


Grey CI

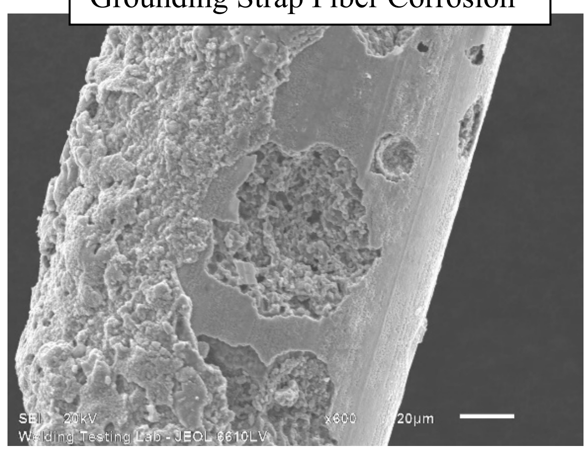


Caustic Cracking

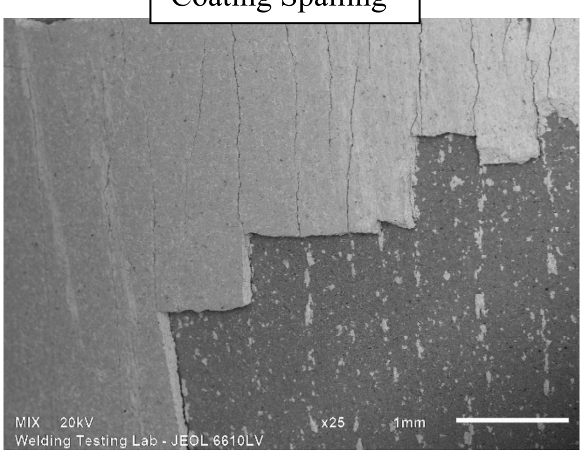
Scanning Electron Imaging: Fracture morphology and defects detection are best analyzed using SEM. Magnification can be varied from low to high, with the former being able to observe defect origination sites and crack growth pattern,s to the latter focusing on fine details such as fatigue striations, micro-void coalescence, intergranular crack growth, etc.



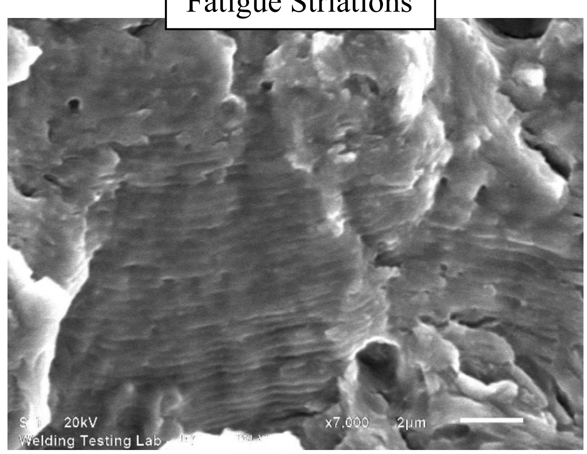
Thumbnail Crack



Grounding Strap Fiber Corrosion

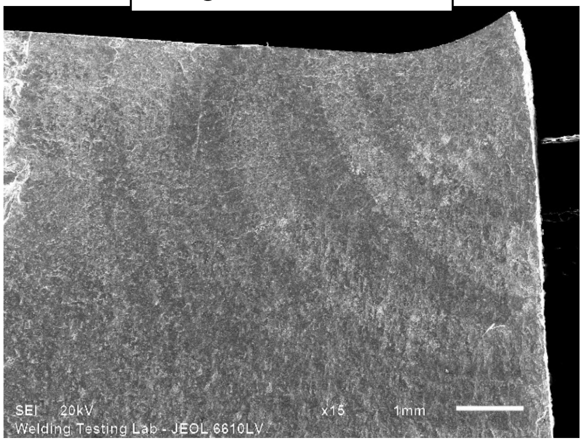


Coating Spalling

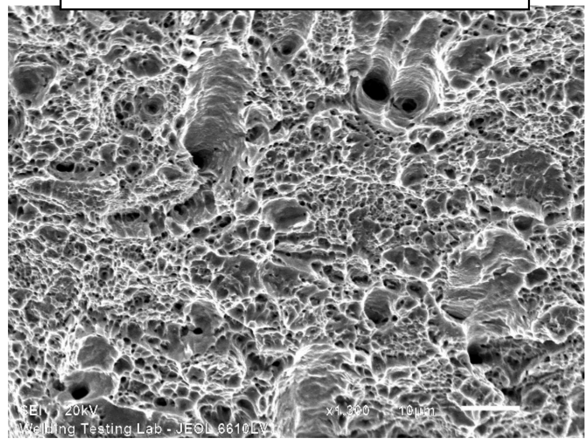


Fatigue Striations

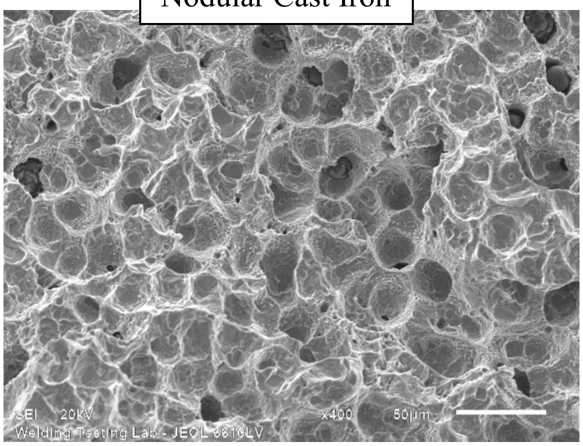
Fatigue Beach Marks



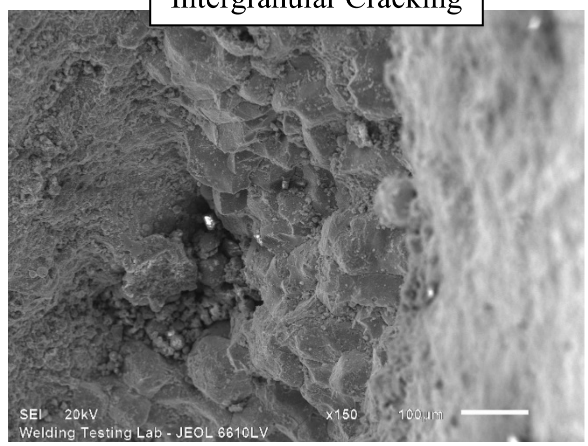
Ductile Micro-Void Coalescence



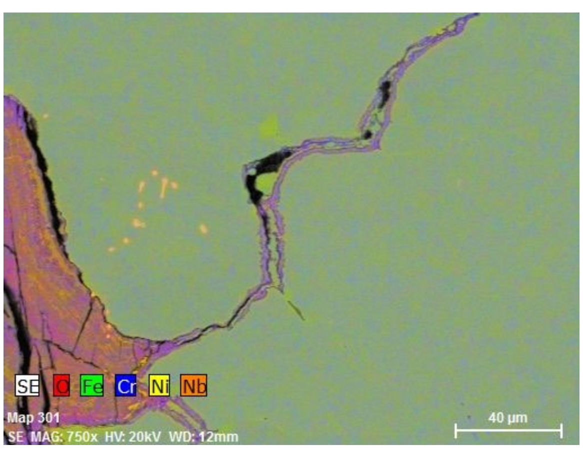
Nodular Cast Iron

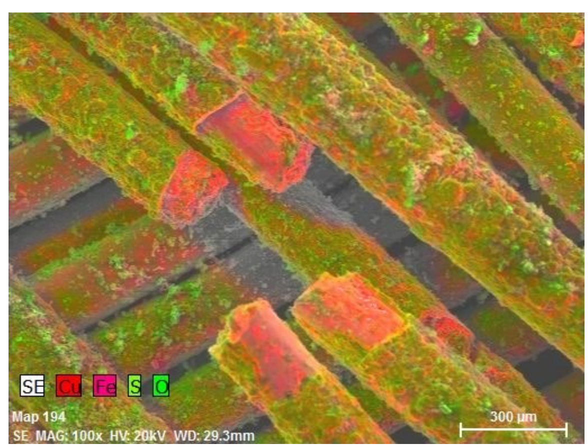


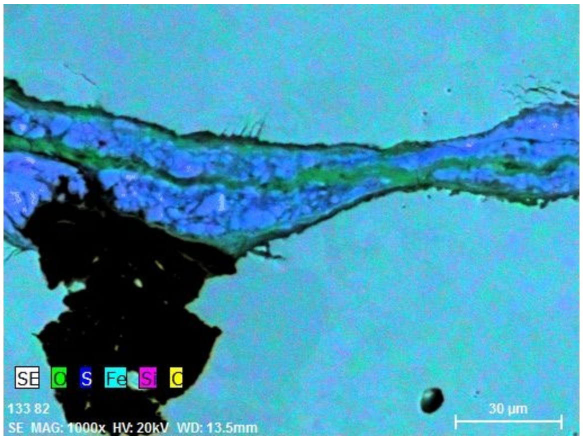
Intergranular Cracking

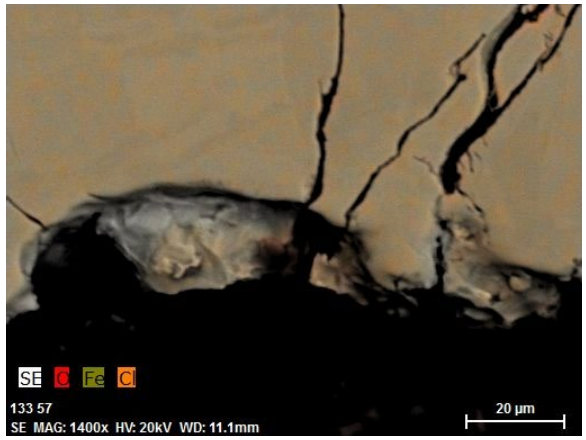


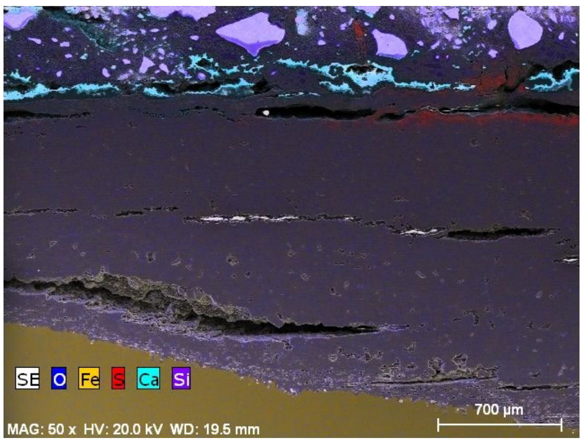
SEM - Electron Mapping: Visual elemental contributions can be examined using SEM Electron Mapping. This technique is useful in exploring grain boundaries for damaging environmental species such as sulfur, chlorine, etc., as well as other effects such as localized element depletion and carbide formations.



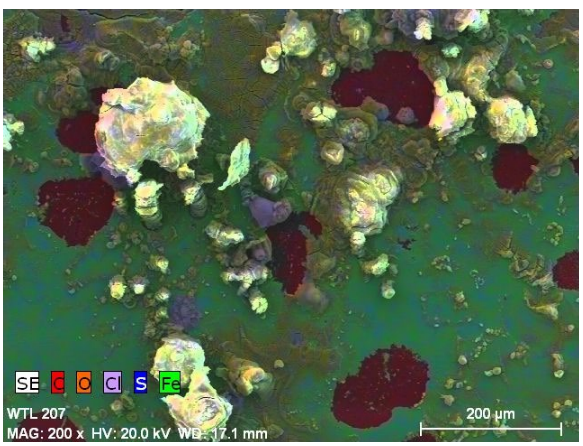


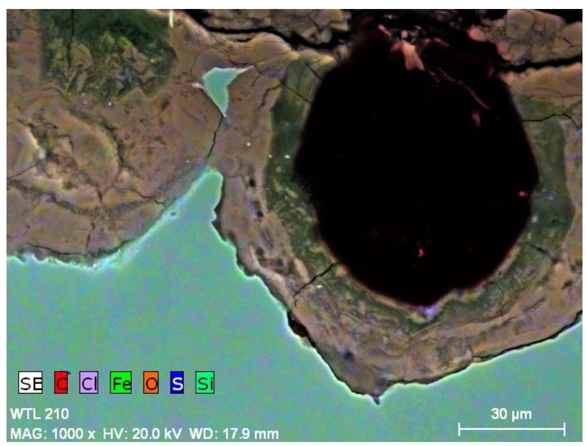

SE O S Fe Si C
133 82
SE MAG: 1000x HV: 20kV WD: 13.5mm
30 µm


SE C Fe Cl
133 57
SE MAG: 1400x HV: 20kV WD: 11.1mm
20 µm

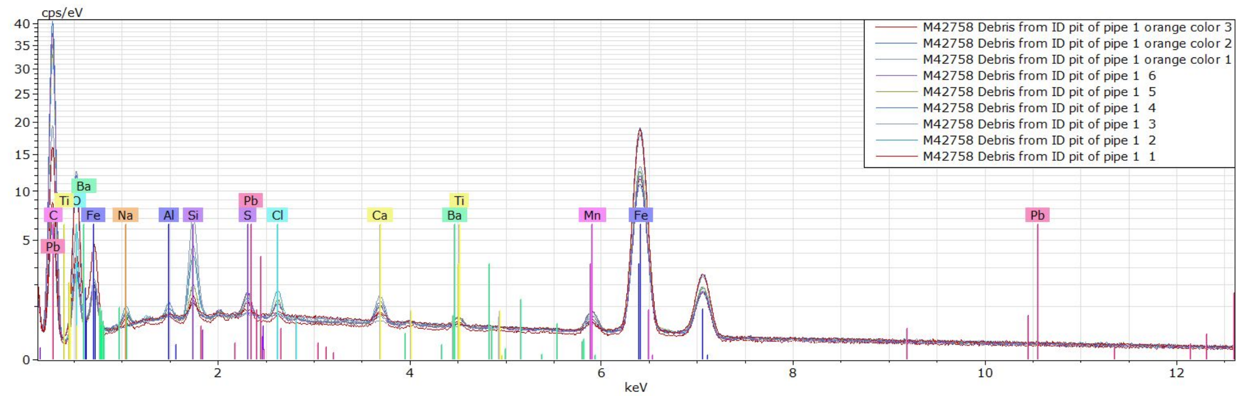

SE O Fe S Ca Si
700 µm
MAG: 50 x HV: 20.0 kV WD: 19.5 mm

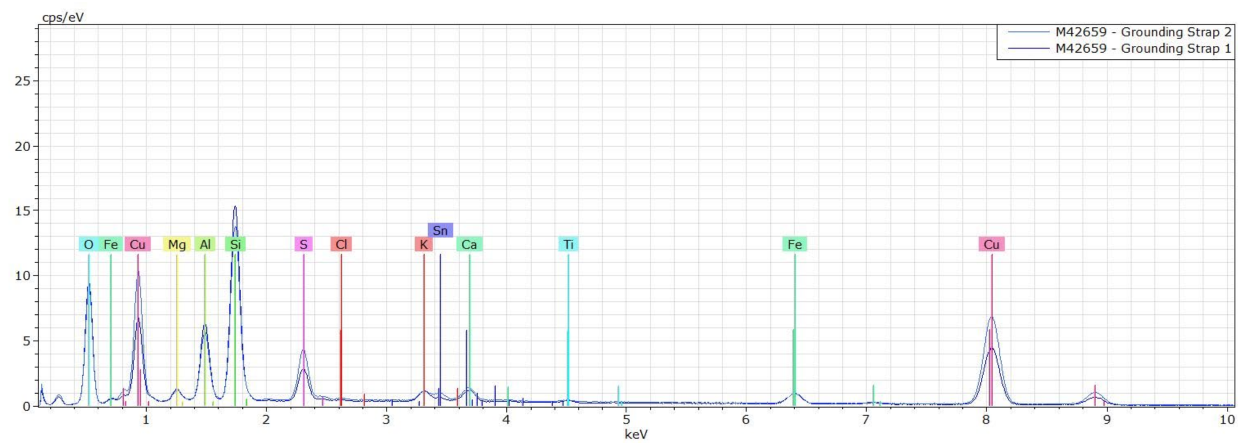

SE C O Cl S Fe
WTL 208
MAG: 650 x HV: 20.0 kV WD: 17.7 mm
50 µm

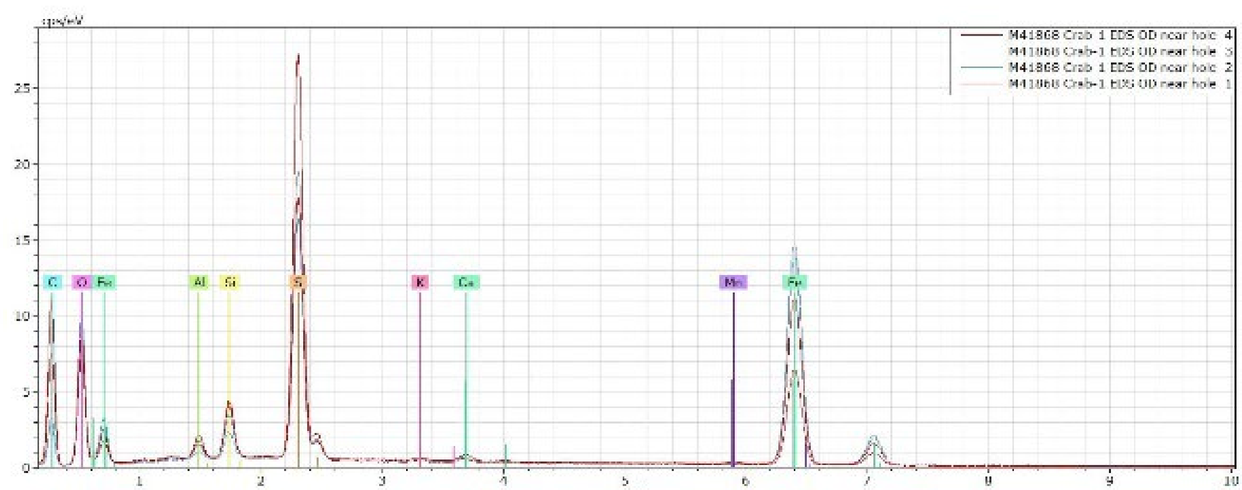

SE C O Cl S Fe
WTL 207
MAG: 200 x HV: 20.0 kV WD: 17.1 mm
200 µm


SE C Cl Fe O S Si
WTL 210
MAG: 1000 x HV: 20.0 kV WD: 17.9 mm
30 µm

Energy Dispersive X-ray Spectroscopy (EDS): Used to determine the elemental composition and quantity by analyzing the x-rays emitted when hit with a beam of electrons. EDS is useful when examining rust or other debris for external contamination such as sulfur, chlorine, bromines, etc. Additionally, spot checks of local micro-structure features can be examined for both major and minor element, but not trace elements. Detection of elements in some machines can be down to atomic number 4, although not all instruments can detect to this low atomic level.







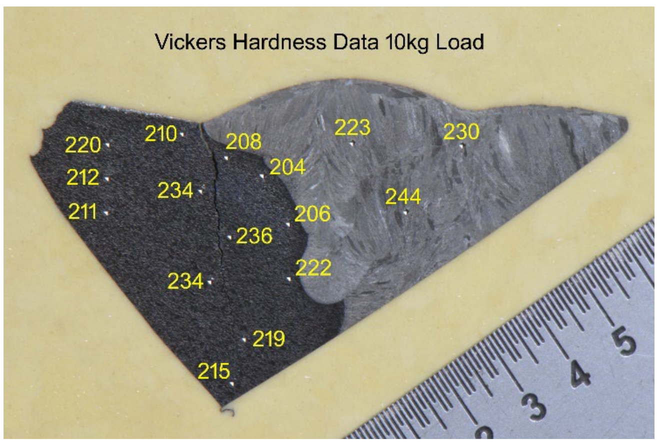
Chemistries: Typically, a quick chemistry check is made when the material is received at the laboratory using x-ray fluorescence (XRF). Subsequently, a more detailed chemistry capture is performed using optical emission spectroscopy (OES). If further refined analysis is made, then a LECO chemical analysis will be performed, which uses a combustion sample. This testing allows for a more precise determination for elements like C, S, H, O, and N.

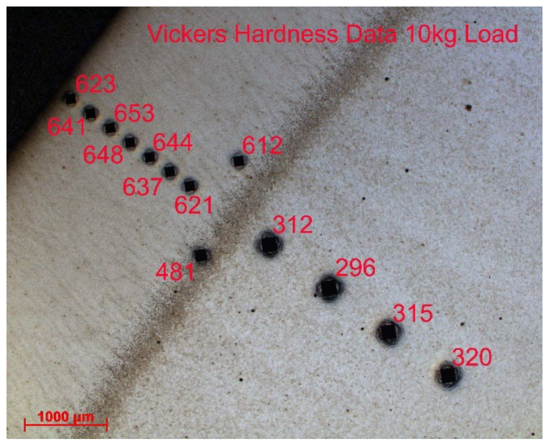
Elements from the chemistry tests are then compared to a standard material specification, such as below:

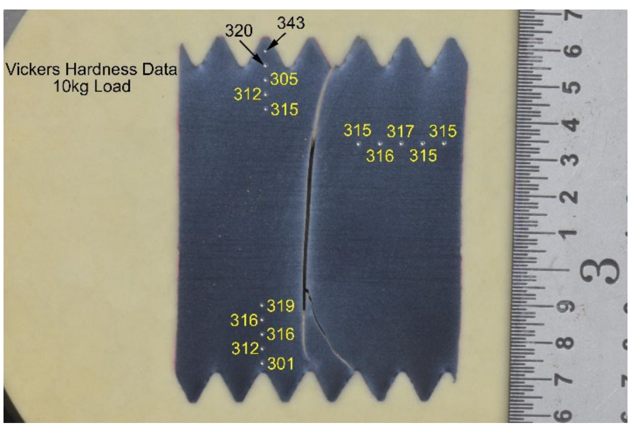
M42985 Chemistry Data		
Elements	Sample (wt%)	ASTM A705 Type XM-12 UNS S15500
C	0.049	0.07 max
Mn	0.48	1.00 max
P	0.022	0.040 max
S	<0.005	0.030 max
Si	0.51	1.00 max
Ni	4.33	3.50-5.50
Cr	15.22	14.00-15.50
Mo	0.22	---
Cu	3.21	2.50-4.50
V	0.07	---
Nb	0.29	***
Ti	0.01	---
Al	0.01	---
Co	0.06	---
W	0.06	---
Fe	Balance	---

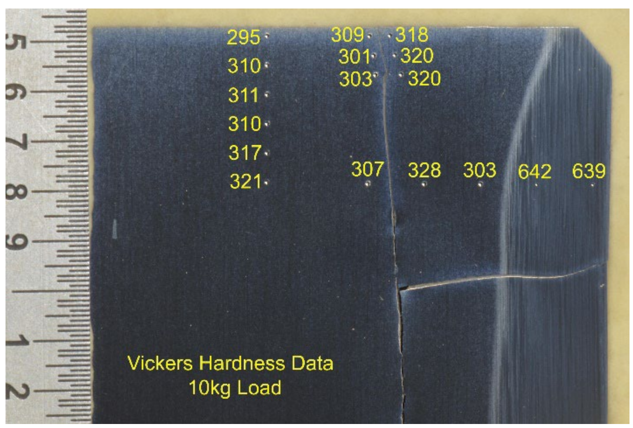
Macro and Micro-Hardness: General strength and surface hardness can be determined using Rockwell, Brinell, or other testing techniques. Testing is beneficial for materials that are to be hardened to a specific degree or welds prior to and/or post stress relief. Macro-hardness readings are beneficial for field activity, such as effects of stress relief, fire damage exposure, sour service criteria, etc. Images are not typically collected for macro-hardness, but instead are reported in table form for the locations tested.

For metallurgical failure analysis, a Vickers micro-hardness test will often be more applicable. Local regions, such as heat affected zones, hardness at crack-tips, surface treatment depths and transitions, etc., allow for more information than a macro-hardness test when investigating various failure mechanisms. Micro-hardness is typically performed on mounts and images are created. Below are some representative micro-hardness mounts and test data points.

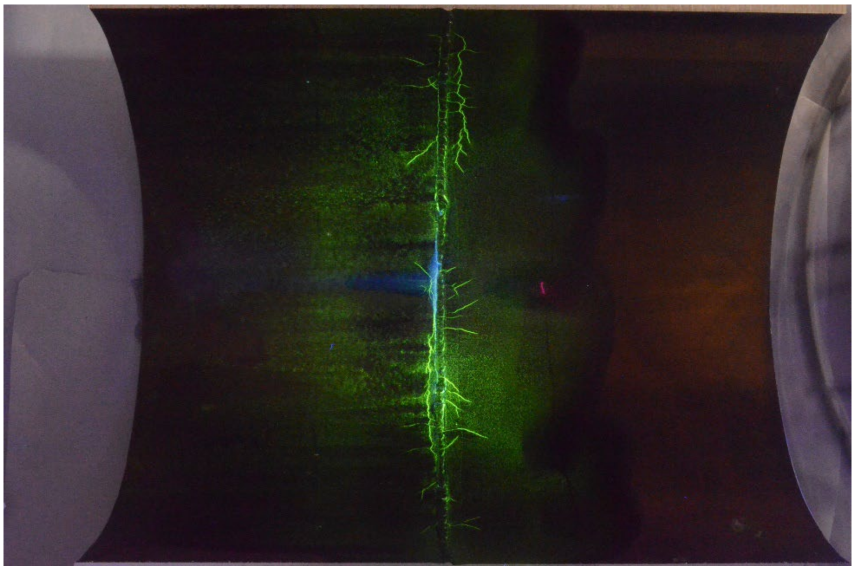


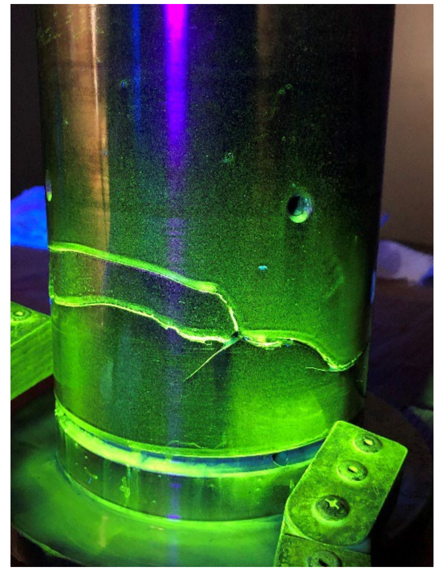


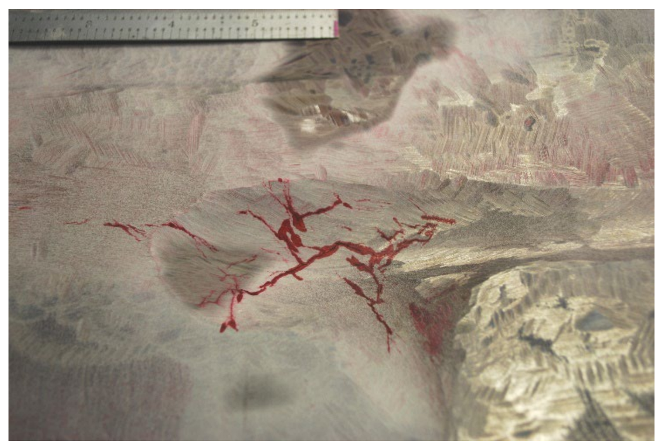


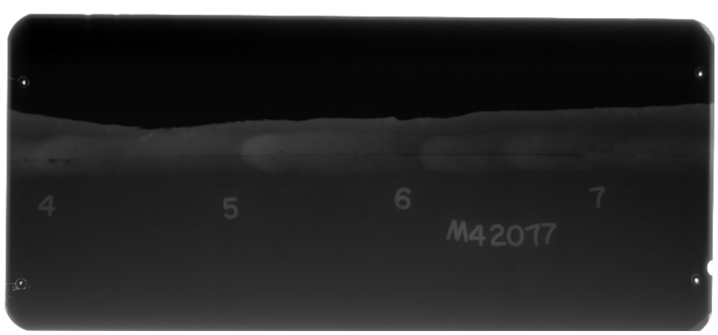


Non-Destructive Testing: Often prior to detailed examination and testing, the received failed components are examined for locations of defects, using non-destructive methods such as dye-penetrant testing (PT), magnetic particle testing (MT) and radiography (RT)

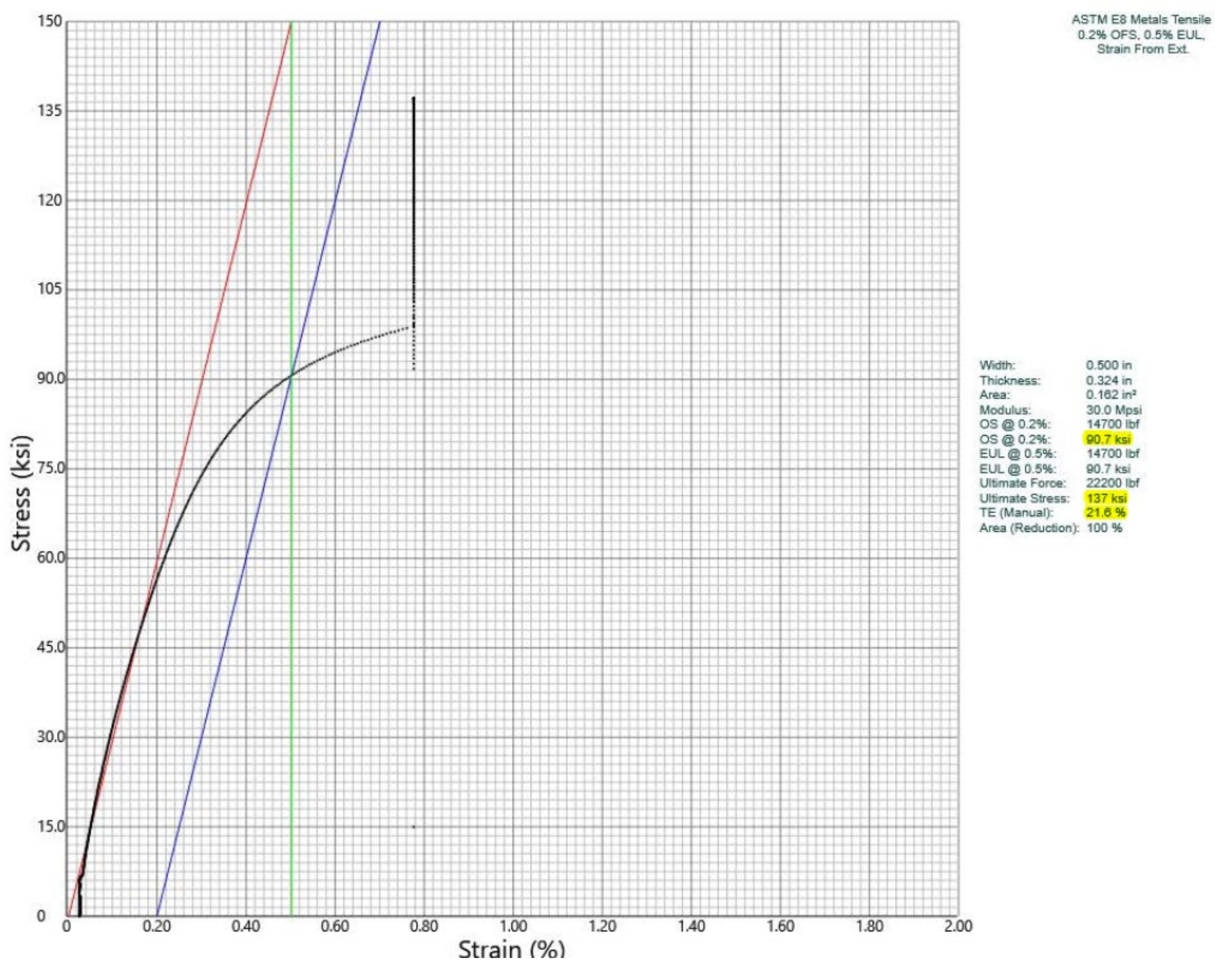








Tensile Tests: Simple tensile tests can be performed to capture ultimate tensile strengths. Such tests can be performed to identify out-of-spec materials or materials subjected to strength-degradation mechanisms like spheriodization. A more detailed stress-strain tensile test can be performed which can identify strain hardening effects (a high yield strength with little additional strain hardening to the ultimate tensile sterngth). Such cases would identify the occurrence of one or more high application loads greater than the material's lower-bound yield strength.



Additional Supportive Test: As the failure analysis proceeds, additional test may be required. Below lists some of these:

- Corrosion-associated micro-organisms by quantitative PCR (qPCR) [NACE Standards TM0106-2006 and TM0212-2018]
  - Total Bacteria
  - Sulfate-Reducing Bacteria (SRB)
  - Sulfate-Reducing Archaea (SRA)
  - Acid Producing Bacteria (APB)
  - Acetic Acid-Producing Bacteria
  - Butyric Acid-Producing Bacteria
  - Iron-Oxidizing Bacteria (IOB)
  - Leptothrix & Sphaerotilus Species
  - Gallionella Species
  - Iron-Reducing Bacteria (IRB)
  - Geobacter Species
  - Shewanella Species
  - Denitrifying Bacteria (DNB)
  - Methanogens

- X-Ray Diffraction (XRD)
  - Differentiate between phases of the same chemical composition
  - Can provide information about a materials crystal structure, crystalline phases, and other structural parameters.
  - Can be used to identify the crystal structure of an oxide.

- Electron Backscatter Diffraction (EBSD)
  - Identification of unknown phases at each analysis point.
  - Map the distribution and measure the area fraction of the phases
  - Distinguish between phases with similar chemical compositions but different crystallographic structures.

- X-Ray Photoelectron Spectroscopy (XPS)
  - Used to identify phases in materials by analyzing the core levels of their electron orbitals.
  - Monochromatic x-rays are directed to a material, which causes electrons to be ejected from their atomic orbitals.
  - The emitted electrons are then analyzed by a detector to determine their kinetic energy.
  - The energy spectrum then provided details of their chemical state, elemental composition and electronic structure.
  - Often used in conjunction with XRD.

- X-Ray Fluorescence (XRF) Spectroscopy
  - For analyzing the type of oxide present in a sample.
  - Provides both qualitative and quantitative information about the elemental composition.

- Fracture Mechanics Testing
    - CTOD
    - J-Integral
    - Basic Charpy

- Plastic Testing