Application Note

Microscopy in Metal Failure Investigations

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No manufacturing or metal production system is perfect. Even with the most rigorous quality controls and thoroughly validated production methods, a percentage of component failure is always possible during manufacture or service. The consequences of such a failure can be minor; rejection of a small percentage of a batch of aluminum wire due to unexpected breakages or delamination of small regions of coating on a rolled steel. Alternatively, the failure can be catastrophic with long-lasting consequences if the failure occurs in a major engineering application such as an oil rig, airplane, ship, bridge, or other structure.

The purpose of a failure investigation is to determine the primary cause of a failure. Once known, investigators can determine corrective actions to prevent or mitigate future failures, establish liability or simply gain a better understanding of a system under test.

Microscopy is an essential step in this process.

Introduction

There are several important stages in the analysis of a failure. The order and extent will vary depending on the individual case; there may be several branching paths of investigation and the need to rule out several possibilities. Review of similar failures plus expertise on the part of the investigator is also useful. These steps may include:

- Gathering relevant information – service data, mill/casting certificates, material specifications, welding procedures, test data etc.
- Visual examination, photography and non-destructive testing if required.
- Selection, preparation and extraction of specimens.
- Macroscopic and microscopic examination of specimens using light microscopy, scanning electron microscopy (SEM) and X-ray microscopy (XRM).
- Advanced analysis by energy-dispersive X-ray spectroscopy (EDS) and wavelength dispersive spectroscopy (WDS) to determine local composition or electron backscattered diffraction (EBSD) for texture, strain and orientation.
- Specimens may include fracture surfaces, cross-sections, foreign matter, corrosion product or intact reference material for comparison.
- Image processing by segmentation to determine relevant metallurgical information such as grain size, phase fractions, porosity, layer thickness, etc. Standard analysis modules for these parameters and other key features are available in ZEISS ZEN core.
- Mechanical testing and/or simulated service condition testing.
- Analysis of all gathered information, then drawing conclusions based on evidence. This is illustrated in Figure 1.

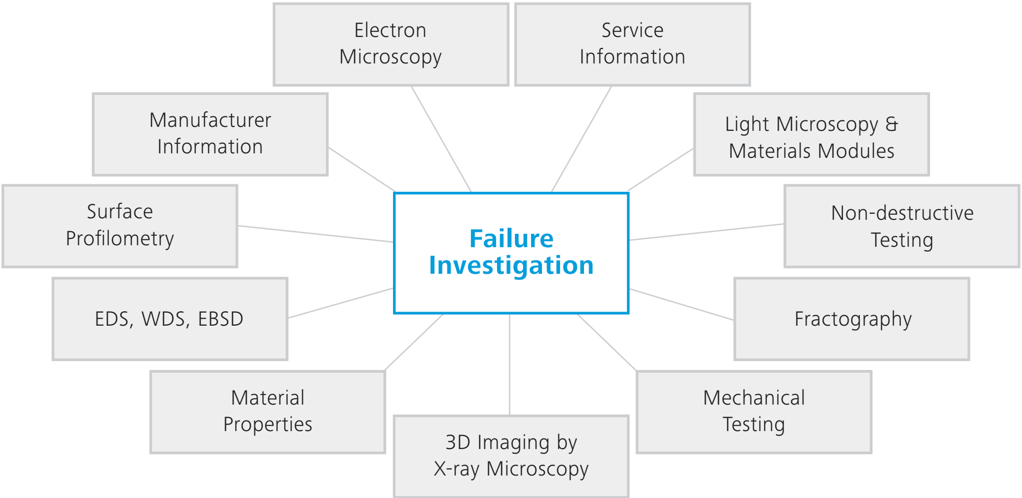

Figure 1 *Investigative techniques for failure investigation*

Microscopy provides many vital tools for failure investigation: the "eyesight of the metallurgist" for identifying features of interest that may have caused or contributed to the failure. Combined with background information and non-destructive and/or mechanical testing, an investigator can build up a complete picture of the system before, during and after failure. Figure 2 shows an example where the presence of necking but a lack of angular distortion can show that the steel probably failed in a ductile manner under simple tensile loading.

Metallographic cross-sections can yield a variety of useful data when viewed using a ZEISS light microscope, such as Axio Imager, or a ZEISS scanning electron microscope such as EVO. The general class and condition of the material can be determined, and information gleaned regarding the method of manufacture and any post-processing. Effects on the microstructure arising from service or heat treatment are observable, including sensitization, corrosion or decarburization. More specifically, examination of features such as cracks can give clues to the material history and failure mode, and identify secondary effects/damage that may not be immediately visible at the surface.

Non-destructive testing can also be carried out at high resolution using X-ray microscopy. An investigator is able to build up a full 3D map of a component showing voids, cracking, pores or any inclusions or significant alterations in local chemistry. Depending on the material and thickness, resolutions of up to 0.7 µm are achievable using ZEISS Xradia 520 Versa XRM, with high resolution maintained even on larger (>40 mm) components.

Fractography

Fractography is the examination of fracture surfaces. It typically uses SEM due to the combination of large depth of field, contrast by composition (via backscatter detection) and high resolution (using secondary electron imaging). EDS provides supplementary compositional information. By interpreting the features present on the fracture surface, a skilled investigator can potentially identify the failure mode, the initiation point(s), and any contributing factors to the failure. 3D mapping of bulk deformation can also assist in this assessment.

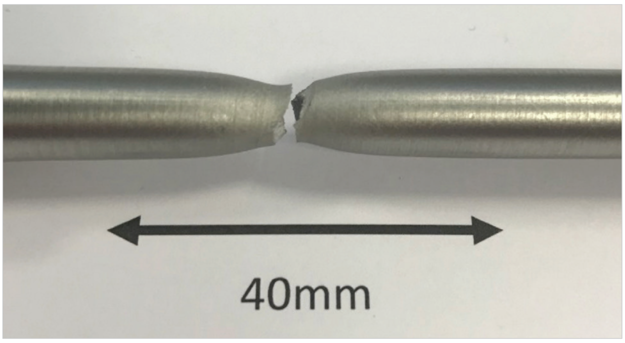

Figure 2 *Steel specimen after failure in simple tension (without torsion or shear). Sample provided by The Test House, Ltd., Cambridge, U.K.*

Figure 3 *Example of failed component: cracked gear from an engine timing component.*

Taking steel as an example, a failure may occur at almost any point after the steel has solidified – during casting, rolling, drawing, welding, coating and subsequent manufacturing steps, or while in service. Figure 3 shows an example of metal failure. Failure modes fall into several broad categories though complex failures may occur by sequential or parallel occurrences of two or more different fracture types that display characteristic fracture surfaces.

Ductile failure can occur due to tension, torsion, bending or more complex strains that may lead to extension, deformation and eventual breakage. The fracture surfaces show characteristic dimples in the surface, with their directions giving an indication of the strain experienced.

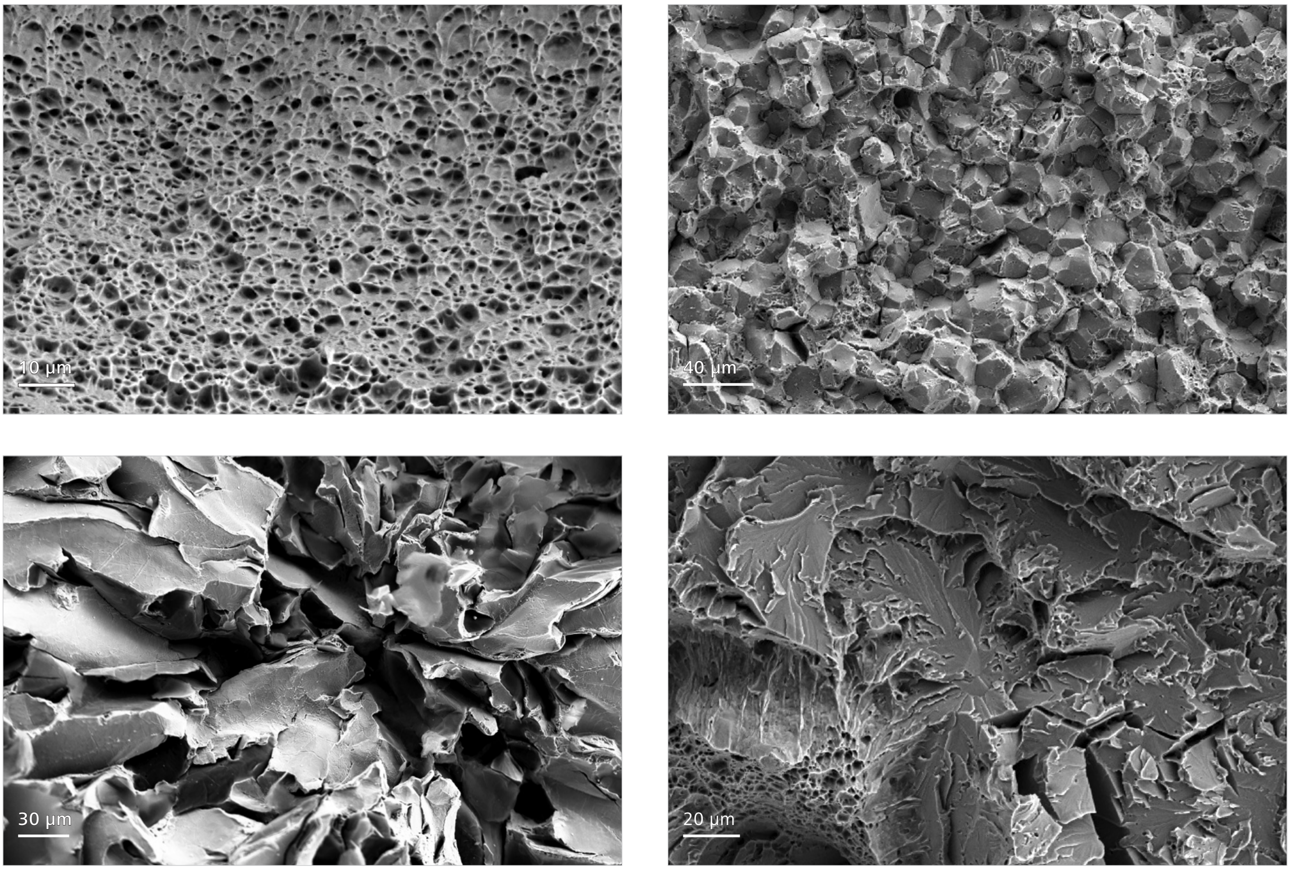

Figure 4 *Fracture surfaces examined using ZEISS Sigma 300 with secondary electron imaging.*
Top left: Ductile failure during bending. Top Right: Intergranular brittle failure during bending.
Bottom left: Brittle failure during tension. Bottom right: Complex fracture surface showing mixed failure modes.
Sample provided by The Test House, Ltd., Cambridge, U.K.

Brittle failure can appear crystalline, with individual facets clearly visible. This is particularly true for intergranular brittle failures, where failure occurred along the grain boundaries. Brittle behavior may be due to the material experiencing temperatures below the ductile-to-brittle transition point, or due to some form of embrittlement.

Fatigue failure arises after repeated sub-yield-point stresses, which may be cyclical. These stresses could be caused by the intended service conditions, e.g., a piston, or they could result from unintended exterior factors, e.g., resonance or vibration of a pipeline. Fatigue can also be assisted or partially caused by thermal effects: repeated expansion and contraction due to changes in temperature as in aerospace or power generation applications.

The features of classic fatigue fracture faces involve progression marks indicating the path of crack growth. Crack branching may be present, and, on a microscopic level, striations often appear on the fracture face. The behavior is complex and dependent on many factors including stress, temperature, geometry, size, material, microstructure and the presence/absence of a stress concentrator or pre-existing crack for initiation.

Using a ZEISS scanning electron microscope such as ZEISS Sigma 300, fracture surfaces can be clearly resolved and information gathered about the failure mode, Figure 4.

A failure mode may not necessarily be identifiable by the fracture face alone, e.g., creep, liquid metal embrittlement or stress-corrosion cracking.

Additional data or cross-sectional examination are often required. The root cause will also differ from case to case. A steel component may have failed in a ductile manner due to the presence of a casting solidification crack that reduces the effective cross-section. It may have experienced premature fatigue failure due to the presence of a large inclusion acting as a crack initiator. Alternatively, it may simply have been exposed to a corrosive substance, e.g., rain water, during storage.

Several examples are presented below.

1. Failure in Steel – Location of Initiation Point

Identification of the initiation point of the failure is one of the key steps towards identifying the series of events that led to failure, and the overall root cause. It may correspond to a manufacturing defect, external damage, or the effects of service. Cracking can even initiate from several points, then subsequently coalesce and form one larger crack. This is often observed in fatigue failures.

Figure 5 shows a steel fracture surface. The entire surface was viewed using a combination of light microscopy followed by scanning electron microscopy. It was possible to identify a clear initiation point. The features radiate outward from this point and the general morphology is indicative of rapid failure by means of brittle overload; relatively little distortion was present and the texture is coarse. EDS may be used to search for evidence of corrosion through chemical foot-printing in the failed region. Microscopic examination of cross-sections could help to establish any other factors that could have led to rapid crack propagation.

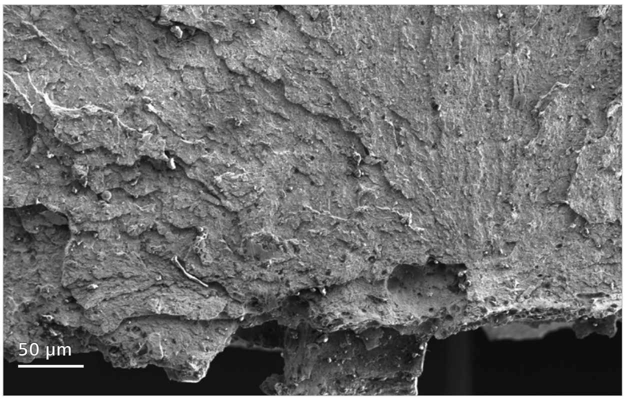

Figure 5 *Steel fracture surface showing location of fracture initiation. Taken using ZEISS Sigma 300 with secondary electron imaging. Sample provided by TWI Ltd, U.K.*

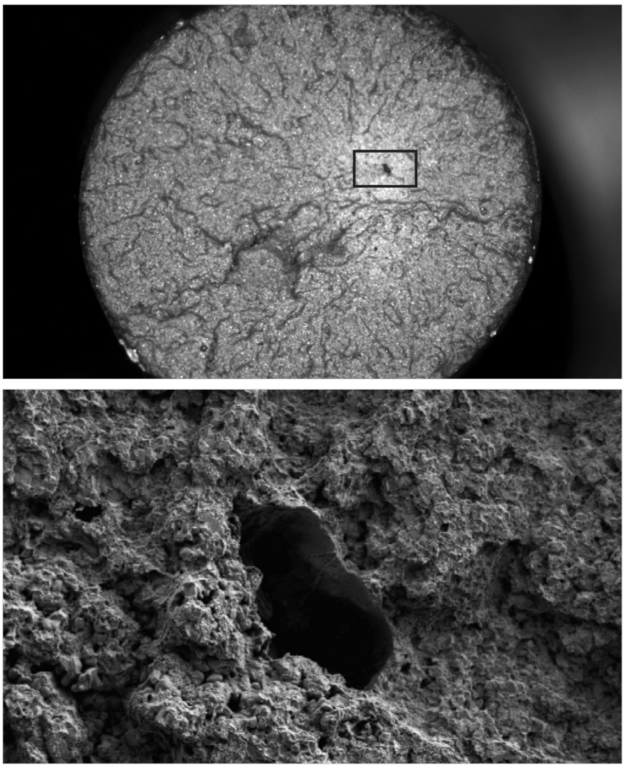

Figure 6 *Top: Typical fracture surface imaged using ZEISS Smartzoom 5. (Bottom) With the help of ZEISS Shuttle & Find, the sample was transferred to the electron microscope to examine the void in detail.*

The cavity at the initiation point is >200 µm wide and may be indicative of a manufacturing defect or corrosion pitting, and thus future investigation would focus on this area (plus the other corresponding fracture face) in detail. Some foreign material is visible as darker speckles near the initiation point. The investigator must establish whether these contributed to the failure or are simply a result of handling or environmental exposure post-failure.

Using ZEISS Shuttle & Find, a combined hardware/software solution, data can be quickly gathered over multiple length scales. A fracture surface can be imaged in a light microscope or confocal scanning microscope such as ZEISS LSM 800. Any regions of interest requiring further investigation can be marked. Larger areas can be scanned using ZEISS Smartzoom 5. The sample is then moved into a scanning electron microscope and Shuttle & Find software can rapidly relocate and focus on these marked regions, Figure 6.

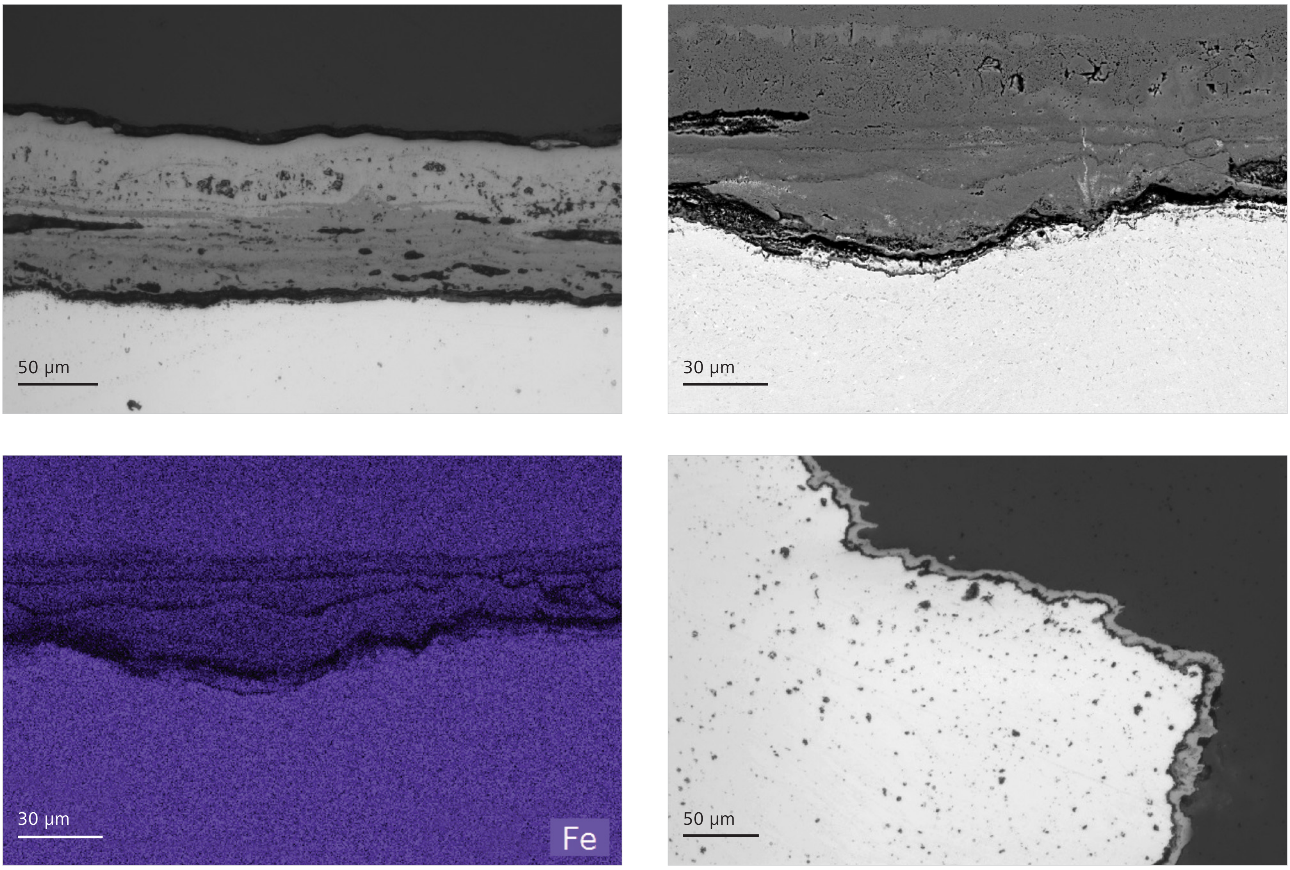

Figure 7 *Cross-sections of 9% Cr steel after failure at high temperature.*
Top left: Oxide scale on intact region, taken using ZEISS Axio Imager Z2.m. Top right: Oxide scale on an intact region, taken using ZEISS Sigma 300 in backscattered electron mode. Bottom left: EDS map of iron content in scale in intact region. Bottom right: Fracture surface, taken using ZEISS Axio Imager. Z2m. Sample provided by TWI Ltd, U.K.

2. High Temperature Failure in 9% Cr Steel

A broken 9% chromium steel component was examined in cross-section through the failed region. On the surface of the metal, remote from the fracture face (Figure 7, top left), a multi-layered ~100 µm thick patchy scale was observed that did not adhere strongly to the surface. Several regions had no scale.

Backscattered electron imaging in a ZEISS Sigma 300 equipped with a 30 mm EDS detector (Figure 7, top right and bottom left) confirmed that the scale consisted of a series of oxide layers, each with different compositions, but all based around iron oxides (Fe_2O_3 and Fe_3O_4) or chromium oxide (Cr_2O_3). The different contrast modes possible and available in bright field light microscopy using ZEISS Axio Imager.Z2m, plus backscattered electron imaging in the SEM, elucidate different features in the structure. The scale had several voids and did not appear to have any significant mechanical strength or protective character.

The thickness of the scale on material remote from the fracture face indicated that there was not a high rate of gas (or particulate) flow past the surface, otherwise it would probably have spalled away by this point. The oxide scale on the fracture surface (Figure 7, bottom right) consisted of a single layer and was significantly thinner but covered the entire fracture face.

Based on the structure of this scale, it can be determined that the steel experienced elevated temperature in an oxidizing environment for a prolonged duration, probably air. Failure of the component had occurred while hot, hence scale on the fracture face. However, the steel had not been hot for long after the failure. It can therefore be derived that the most probable failure mechanism was high temperature creep.

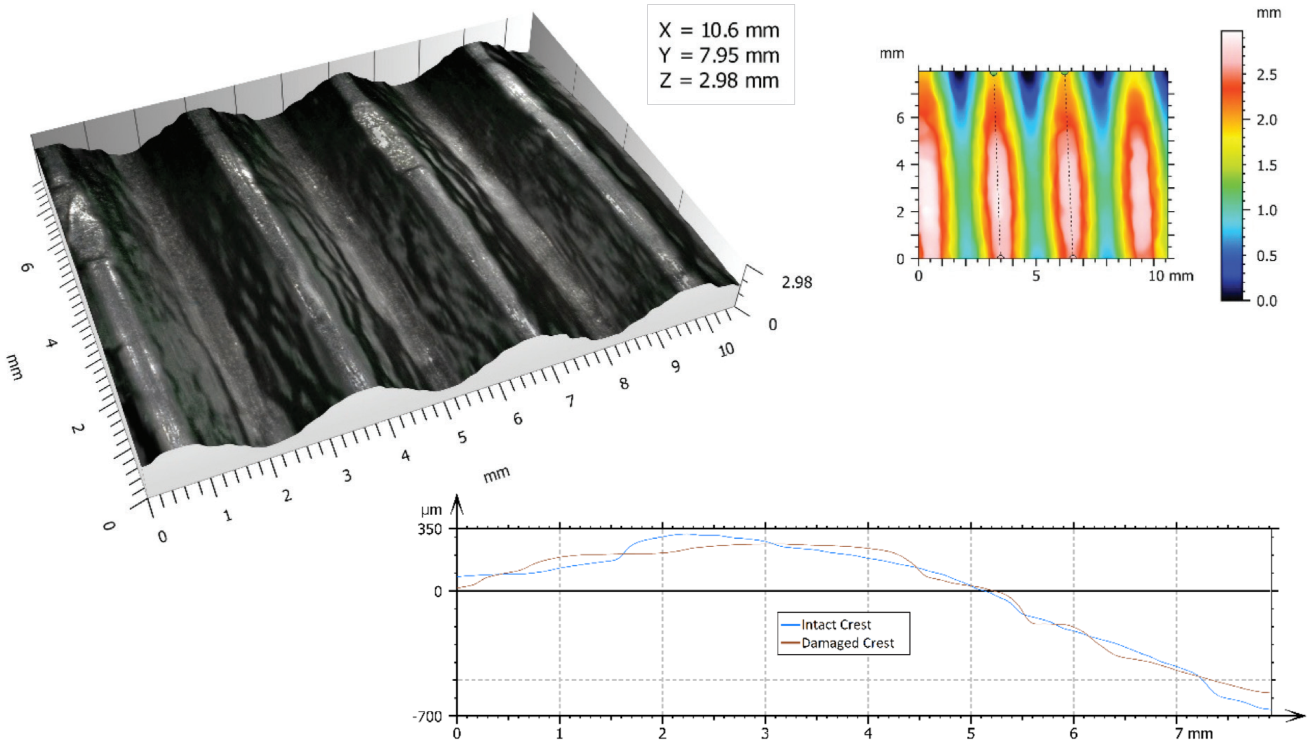

Figure 8 *3D map of damaged 25 mm screw thread with height profile and the positions of line profiles indicated on this height profile. Map captured using ZEISS Smartzoom 5 and analyzed using the Confomap software.*

3. Examination of Damaged Screw Thread

Several techniques used in investigation of full failures can be applied equally well to components that have only partially failed or experienced damage. In our final example, a 25 mm diameter screw thread was examined. It had experienced mechanical damage visible to the naked eye. To assess the severity of the damage, a non-destructive 3D map of the surface of the screw thread was obtained using ZEISS Smartzoom 5.

By drawing line profiles across a damaged crest and an undamaged crest (Figure 8), we could see that the damaged crest was rougher with a greater variation in height profile along the damaged region. The presence of three troughs, roughly equidistant from each other, indicates that the damage likely resulted from another rough part scraping along the surface but this is not conclusive.

There was no significant difference in the overall form, indicating that the damage was mostly cosmetic and the integrity of the component was probably unaffected. Non-destructive testing can establish whether cracking is present with X-ray microscopy providing sub-micron resolution scans of the interior of some components. If a component can be cut, cross-sections can give information on cracks as well as any local surface effects such as hardening or sensitization.

Summary

Once all the available and relevant data have been gathered through multimodal microscopy and other techniques, the investigator can compile a comprehensive failure analysis report to determine the root cause (if possible), determine corrective actions, assign liability/responsibility, identify any related areas or components that may need further investigation, and provide supporting evidence for any of these points.

Light microscopy, electron microscopy, EDS and X-ray microscopy provide an investigator with an invaluable and versatile tool set for any failure investigation. They have proven their worth over many decades and are used frequently in commercial failure analysis in a variety of industries.

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