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TECH NOTES

Field Metallography

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Introduction

Field metallography is a non-destructive in-situ testing of metallurgical condition of metallic components. The procedure is carried out in the field without the need to remove a component. The component being investigated can be left in service during metallographic replication.

Field metallography is extensively used in high temperature damage assessment of power generating components. The key areas investigated are creep damage assessment as shown in Figure 1, carbide coarsening in low alloy steels and spheroidisation of pearlite in carbon steels.

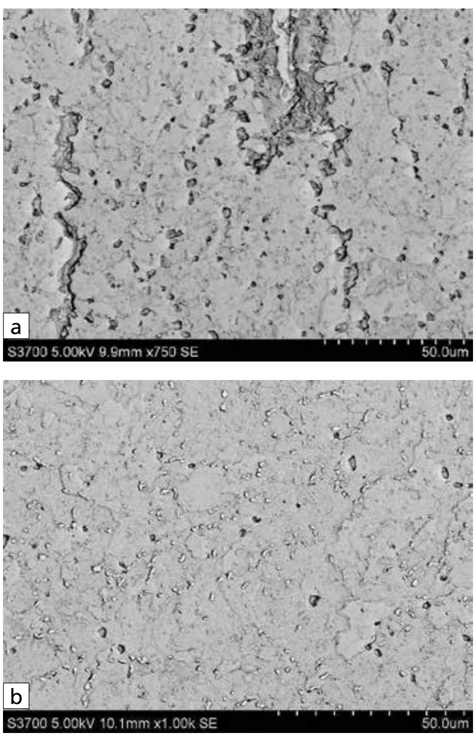

Figure 1 shows SEM micrographs of creep damage assessment with (a) illustrating early stages of creep and (b) showing advanced creep with aligned cavities and cracks. The micrograph is an acetate sheet replica with the details of the microstructure enhanced through sputter coating.

The technique can also be extended to other applications such as grain size measurements, determination of heat treatment of materials, determination of type and grades of different materials such as cast irons and steel. Field metallography is also encountered on tribology to ascertain wear mechanisms to evaluate fretting, burnishing and abrasion mechanisms. The technique is also used for archeological investigations of artefacts and in criminal forensic investigations.

Materials/Equipment/Planning

Pre-Planning

Sectioning's main purpose is to expose the area of interest on the Before carrying out field metallography, planning is critical necessitating certain aspects to be carried out in advance [1, 3]. A metallographer carrying out this analysis should understand the material under investigation with the key parameter being hardness of the material, the type and composition of the alloy and the surface conditions as detailed below.

- The hardness of a material will guide the metallographer on what consumables, i.e. grinding papers, polishing clothes, abrasives will be used,
- The type of alloy will determine what procedures to use and which etchants to carry
- Finally, the surface condition will determine initial grinding process and consumables i.e. whether to use grinding stone, a flapper wheel or a coarse grinding paper.

Equipment

The equipment/accessories needed for the bulk of field metallography are outlined below. This relates to damage assessment of components in power generation. The same equipment's can also be used in other applications

➢ Grinding/polishing
- Portable or motorised handheld grinding/polishing device (ElecterSet)
- Rubber stubs for grinding and polishing papers cloths
- Discs of adhesive backed grinding papers (60, 120, 240, 320, 400, 600 grit in separate containers/plastic bags
- Discs of polishing cloths (VelTex)
- Diamond paste (9, 6, 3, 1µm)
- Plastic bottle containing alcohol/acetone
- Cotton balls for swabbing and cleaning

➢ Etching
- Plastic bottles of premixed etchants (screw caps)
- Cotton balls for swabbing
- Rubber gloves

➢ Replication

- Sheets of cellulose acetate film (20-45microns thick)
- Bottle of acetone or methyl acetate solvent
- Tweezers
- Box of standard glass slides
- Double sided adhesive tape
- Black spray paint

➢ In situ examination of microstructure
- Portable upright microscope
- With camera attachment

➢ Miscellaneous
- Absorbent cloths/paper towels
- Flashlight depending on luminescence of work area
- Safety equipment (Safety goggles, glasses, hardhat)
- Extension cord
- Collapsible stool

The alternative to acetate sheet replication is the use of silicone based, Figure 2, a two-part replication system. The silicone-based system is ideally suited for fracture surfaces of large components or for tribological application where surface roughness measurement is a critical parameter.

Replication

Surface Preparation *(to mirror finish)*

The preparation procedure selected or used should be tuned to the material type and the microstructural features under investigation. Principles used in standard metallographic preparation are applicable. This involves rough grinding using coarse grit papers and progressively using finer grit paper before polishing stages as shown in Table 1. Application of inter-stage etching during final polishing

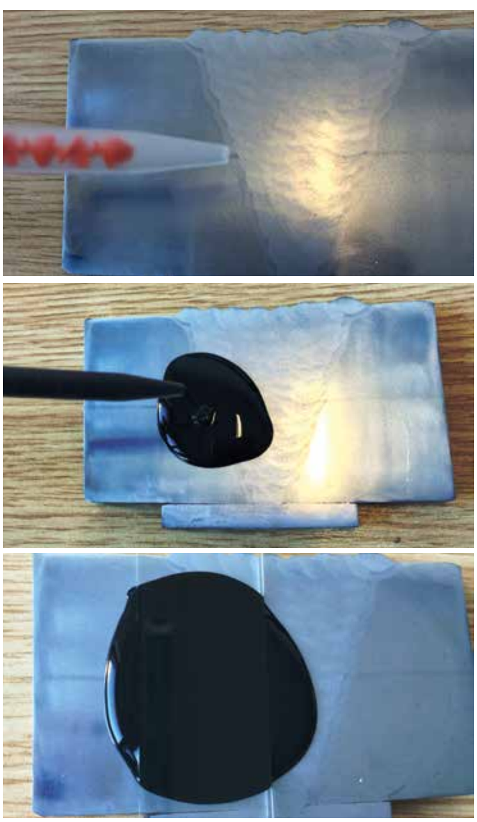

Figure 2 Silicone based two-part replication process

is critical especially for creep damage assessment of components.

Etching

This involves chemical etchants to attack the as-polished surface to reveal the alloy microstructure. Thus, selection of suitable etchant for different alloys is of utmost importance. Common etchant used for Fe, carbon and alloy steels, cast iron is Nital a mixture of nitric acid in methanol or ethanol. The concentration of nitric acid varies from 1-10%. The 2% solution is most common; however, 5-10% is also used for high alloy steels. Application involves swabbing of the polished surface for up to 60 seconds. Villella's reagent is another commonly used etchant for ferrite-carbide structures and will outline constituent phases in stainless steels. It is also ideal for tool steels and martensitic stainless steels. Kalling's No.2 is also used in field metallography to etch austenitic and duplex stainless steels and is applied by swabbing of sample.

NB: Caution should always be used when dealing with etchants and risk assessments and COSHH evaluations should always be carried out.

Table 1. Recommended Procedure Using 30-32mm Discs

Surface	Abrasive Size	Key Parameters
CarbiMet	280grit [P320]	• Electer Set settings for grinding prodedure • Ease of grinding/polishing - Angled handle (45) • Speed: 15,000rpm (Motor load - green Amber) • Depending on area of interest, multiple SiC papers should be used • Precaution: Dust Mask
CarbiMet P600		
CarbiMet P800		
VelTex	9µm MetaDi Supreme Diamond*	• Speed: 6000-7000rpm with 1 cloth per each step • For 1µm step, it should be carried out at least 2 steps with intermediate etching between the stages • Lubricant: **MetaDi fluid
VelTex	3µm MetaDi Supreme Diamond*	
VelTex	1µm MetaDi Supreme Diamond*	

Application and Extraction of Replica

This involves applying the acetate sheet onto the etched surface to create a replica of the surface, Figure 3. Before applying the acetate sheet, it has to be softened with acetone or methyl acetate solvent to adhere to the surface. Once dry, the transparent acetate sheet is sprayed with a black paint to make the film opaque and hence enhance the contrast of the replica before peeling it off with the help of tweezers. The extracted replica is attached on to a glass slide with a double-sided adhesive tape and labelled to show details of the component under investigation, the date and replica number before examination.

Applying the acetate sheet is the most challenging stage during replication process and the difficulty is attributed to the thickness of the film, geometry/configuration of the component under investigation, environmental conditions and the experience of a replicator. Thicker films are not ideal, as they may not fill the contours relating to the etched microstructure with somewhat low spatial resolution.

On the other hand, thin films are prone to tearing during replica extraction but have excellent spatial resolution demanding extra care. For metallurgical replication process, silicone based systems are ideally suited for flat surfaces and where extremely low volume replicas are need. For curved surfaces or complex geometry of the

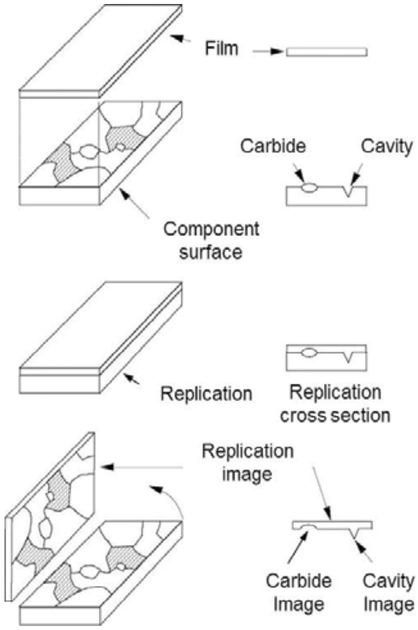

Figure 3 Schematic illustration of replication process

replicated region, acetate sheets replica can be produced with ease and can then be flattened on a glass slide. This is not practically ideally suited for silicone-based system. In addition, the long cure times relating to two-part systems proves prohibitive in this area.

Replica examination

Replica examination is generally carried out using an optical microscope such as Nikon LV 150 upright microscope using bright field illumination. Typical magnifications range used ranges between 50 to 1,000 magnifications at the eyepiece.

Improving contrast: Contrast of the replicas can further be improved by coating the replica with a carbon or metallic coating. This is generally done using a sputter coater by a vacuum deposition of carbon, gold or gold-palladium alloy. This enables replica examination using electron microscopy techniques such as a

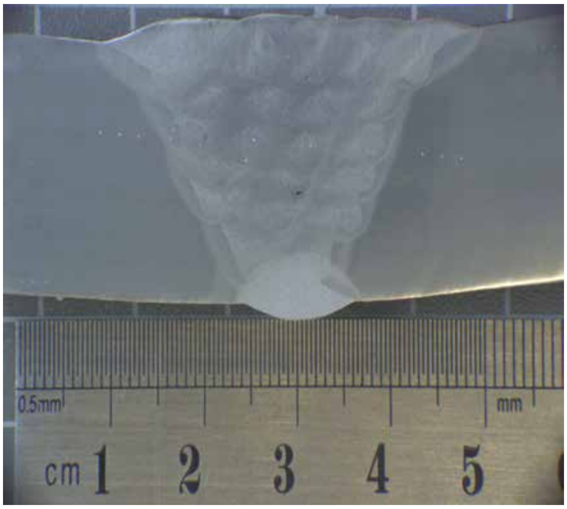

Figure 4 As prepared and etched weld component showing the weld passes, the heat affected zone on either side of the weld and the base metal.

scanning electron microscope (SEM). Metallic coated replicas have been reported to have excellent spatial resolution comparable to the actual microstructure. For silicone-based systems, contrast enhancement is not ideal; however, the resolution is sufficient for general application or low magnification investigations.

Examples Of Metallography Applications
Weld Microstructural Investigations

Figure 4 below shows the weld microstructure with base metal on either side of the weld after etching with 2%nital. Weld microstructural investigations look at the presence of inclusions, reheat cracks at the toe or cap regions due to high residual stresses, blow holes, weld slag among others.

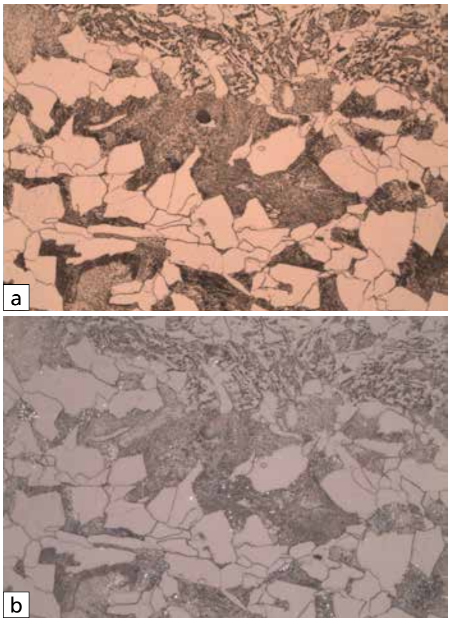

Figure 5 (a) shows the base metal ferrite/ pearlitic microstructure on etched sample and (b) showing the corresponding replica.

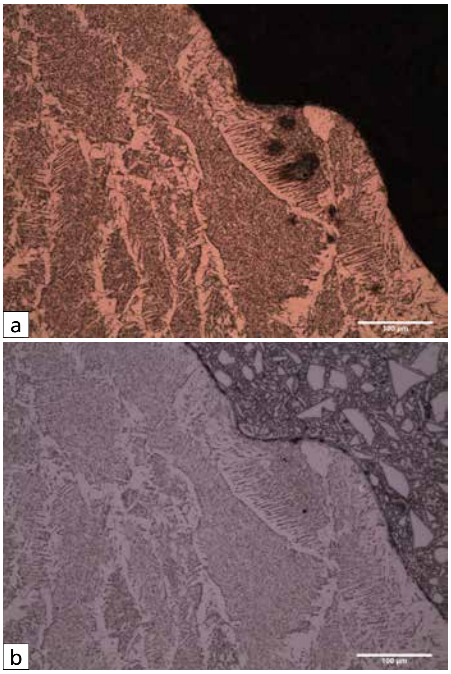

Figure 6 a) shows the weld, weld fusion line and coarse grainede HAZ regions and (b) showing the corresponding replica.

Figure 5a and Figure 6a show the substrate and weld microstructures respectively after final etching. The corresponding replicas, 5b and 6b exhibit a copy of the original microstructure with reduced contrast. All the features are clearly captured exhibiting ferrite grains and pearlitic regions.

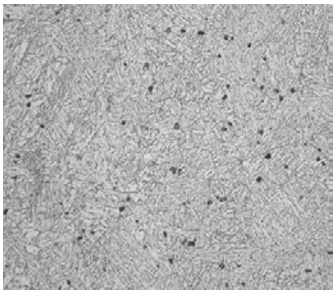

Figure 7 show a 9Cr steel with cavities randomly distributed. Rated as 2a or 2b with the actual number density determining the exact rating

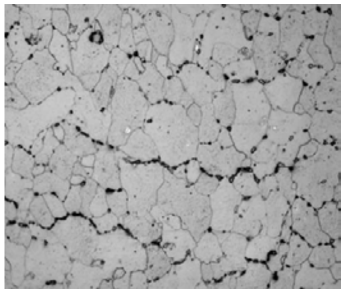

Figure 8 shows a 14MoV6-3 showing aligned cavities along grain boundaries rated as 3b

Creep Damage Assessment

The microstructure rating charts for evaluation of the microstructure and creep damage for high temperature piping and tubing is VGB-TW 507 [4]. The rating is based on the ability to count the number of cavities, cavity orientations, grain boundary separations, micro-cracks and marco cracks as shown in the replica images Figure 7 to Figure 8.

Tribological Investigations

Tribology relates to the study of interactig surfaces relative to motion [4]. Of interest to tribologists is the ability to replicate the associated wear, surface damage or modifications observed on the interacting surfaces. Tribologists often measure the surface texture and roughness parameters before and after the wear process. Examples in this field include metal rolling tribology, tyre tribology, brake tribology, jet engine components among others. The application procedure is shown in Figure 2 above with tribologists preferring flexible to rigid two-part system.

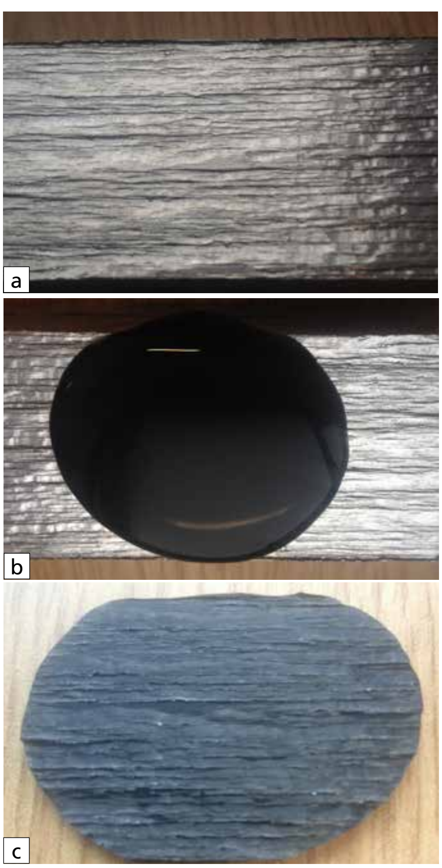

Figure 9 shows an (a) Al rolled surface, (b) replicated region and (c) showing the resultant replica which can be used for surface profilometry measurements

Figure 9 illustrates metal rolling tribology for an aluminum sample Figure 10 shows measurements carried out on a silicone based replica of a stamping tool for aluminum sheet metal. A two-part silicone system with medium to high shore hardness was selected. Surface roughness measurements were carried out on the stamping tool and correlated with the roughness measurements on the replica of the aluminum surface and the test panel. The results showed high spatial resolution on the replica with differences on the measured roughness values of approximately 0.05µm.

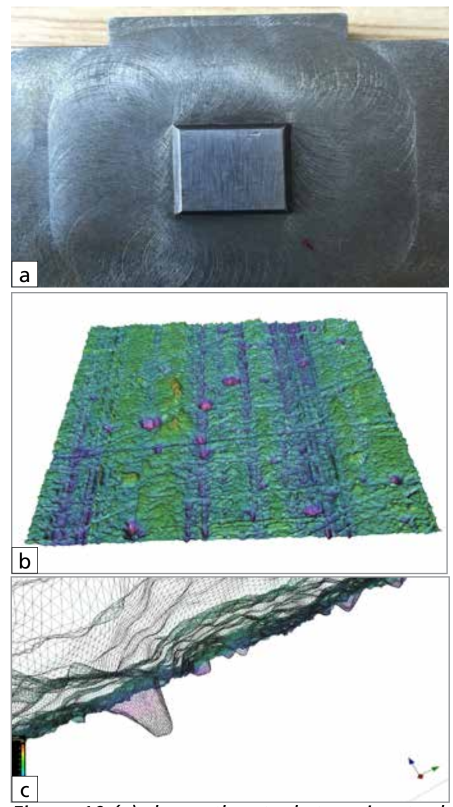

Figure 10 (a) shows the steel stamping tool, (b) illustrates optical surface roughness measurements as a function of depth and (c) is a 3D matrix reconstruction of the surface showing depth of penetration of the surface imperfections

Forensic investigations

Involves use of science through a number of techniques to gather and examine information that is admissible in court [6]. Forensic investigations are quite extensive, examples include, firearm evidence, tool marks evidence, imprint evidence, and tyre marks. An example demonstrated herein, is a tool mark evidence where tools have been used to commit a crime by aiding entry. These tools have unique marks associated with manufacturing process, defects from use which are identifiable on a surface they contact. Law enforcements will often submit tools and items bearing tool marks to be examined. Figure 10, illustrates a screwdriver being used to create tool mark on a lead sample (a) and using silicone based resins, a replica is extracted (b). Figure 10 (c) shows the comparisons of the actual damage (A) and the replica (B) for a match. The characteristics manufacturing striations are evident with defect areas on the tool also discerned.

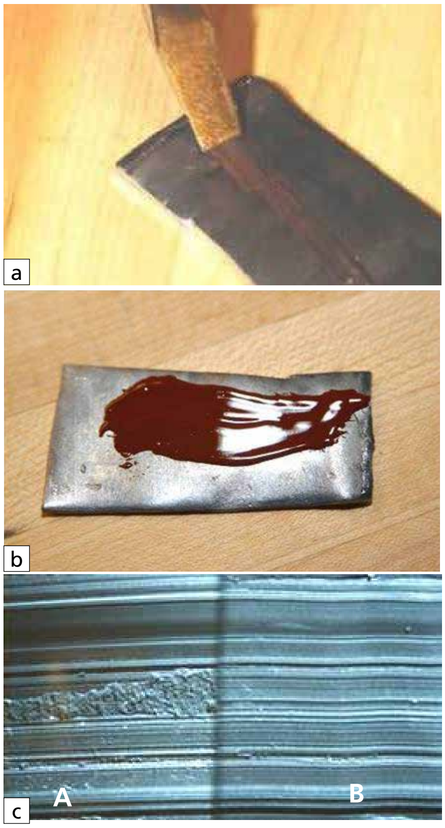

Figure 11 shows an (a) test mark in a lead sample (b) showing two part silicone resin applied to replicate the surface and (c) shows the resultant replica with striations from the test mark (source Minnesota department of public safety)

Conclusions

Field metallography is a non-destructive technique that is essentially an extension of a metallographic laboratory. The principles of laboratory sample preparation are applicable in field metallographic preparation except use of portable tools to extract a record of alloy microstructure. The technique is also used to carry out in-situ investigations of components that are too large to bring to a metallographic laboratory. The technique is primarily used in life assessment of high temperature components found in nuclear and coal fired power plants, petrochemical refineries, and heat recovery steam generator (HRSG's) among others. The report also demonstrated its extensive applications in the fields of tribology and forensic investigations.

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