Replication Microscopy Techniques for NDE

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SURFACE REPLICATION is a well-developed electron microscopy sample preparation technique that can be used to conduct in situ measurements of the microstructure of components. The in situ determination of microstructural deterioration and damage of materials subjected to various environments is an objective of any nondestructive evaluation (NDE) of structural components. The need to assess the condition of power plant and petrochemical metallic components on a large scale recently led to the application of surface replication to the problem of determining remaining life. The usual method of metallographic investigation, which may involve cutting large pieces from the component so that laboratory preparation and examination can be performed, usually renders the component unfit for service or necessitates a costly repair. As a result, metallographic investigations are avoided, and important microstructural information is not available for evaluating the component for satisfactory performance. Therefore, an in situ or field microscopy examination is needed to aid in the proper determination of component life.

The replica technique for the examination of surfaces has been extensively used for studying the structure of polished-and-etched specimens and for electron fractographic examination (see the article “Transmission Electron Microscopy” in Volume 12 of the 9th Edition of Metals Handbook for a discussion of replication techniques in fractography). Surface replication was the predominant technique in electron microscopy prior to being supplemented by thin-film transmission and scanning electron microscopy. Recently, the replication microscopy technique has become an important NDE method for microstructural analysis, and an American Society for Testing and Materials specification has been written for its implementation (Ref 1).

**Mechanical Polishing Methods.** Components in service usually have a well-developed corrosion or oxidation product or a decarburized layer on the surface that must be removed before replication. Coarse-grinding equipment can be used as long as the proper precautions are taken to prevent the introduction of artifacts into the structure due to overheating or plastic deformation. Sandblasting, wire wheels, flap wheels, and abrasive disks have all been used. After the initial preparation steps are completed, standard mechanical polishing techniques can be used. Field equipment is commercially available to help the metallographer reproduce the preparation steps normally followed in the laboratory. Depending on the material, various silicon carbide abrasive disks of different grit size, together with polishing cloth disks with diamond paste or alumina of varying grit size, can be used to prepare for the etching step. Finally, any appropriate etchant for the material being examined can be applied to develop the microstructure. For the proper identification of such microstructural features as creep cavities, a maximum double or triple etch-polish-etch procedure should be used (Ref 2). The etchants used for the various materials investigated by the replication technique are described in Volume 9 of the 9th Edition of Metals Handbook and in Ref 3.

**Electrolytic Preparation Technique.** Although electrolytic polishing and etching techniques have often been employed as the final mechanical polish in sample preparation, inherent problems still exist in this process. The electropolishing technique uses an electrolytic reaction to remove material to produce a scratch-free surface. This is done by making the specimen the anode in an electrolytic cell. The cathode is connected to the anode through the electrolyte in the cell. Specimens can be either polished or etched, depending on the applied voltage and current density, as seen in the fundamental electropolishing curve in Fig. 1. However, the pitting region must be avoided so that artifacts are not introduced into the microstructure. It is virtually impossible to prevent pitting without precise control of the polishing variables, and pits can often be mistakenly identified as creep voids.

Several portable electropolishing units are commercially available. The most important variables (time, bath temperature, electrolyte composition, and the current density-voltage relationship) have been investigated for a selected group of electrolytes (Ref 4). A direct comparison of electropolishing units and the precautions necessary for handling certain electrolytes are given in Ref 5.

It should be noted that there are areas in both fossil and nuclear plants in which neither acid etches nor electropolishing methods and materials are allowed because of the potential for intergranular stress-corrosion cracking. Stainless steel piping in nuclear plants can be replicated to determine defects by manual polishing without etchants. Generator retaining rings have been replicated by manual polishing to resolve NDE indications, because they are extremely sensitive to stress-corrosion cracking and no acids or caustics are allowed to be used (Ref 6).

**Replication Techniques**

Replication techniques can be classified as either surface replication or extraction replication. Surface replicas provide an image of the surface topography of a speci-
Table 1  Comparison of replica techniques

<table>
<thead>
<tr>
<th>Type</th>
<th>Advantages</th>
<th>Disadvantages</th>
</tr>
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<tbody>
<tr>
<td>Surface replicas</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Acetate</td>
<td>Excellent resolution</td>
<td>Coating required</td>
</tr>
<tr>
<td>Acrylic</td>
<td>Direct viewing</td>
<td>Adhesion</td>
</tr>
<tr>
<td>Rubber</td>
<td>Easy removal</td>
<td>Resolution</td>
</tr>
<tr>
<td>Extraction replicas</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Direct stripped</td>
<td></td>
<td></td>
</tr>
<tr>
<td>plastic</td>
<td>Easy preparation</td>
<td>Particle retention</td>
</tr>
<tr>
<td>Positive carbon</td>
<td>Excellent particle</td>
<td>Coating required</td>
</tr>
<tr>
<td></td>
<td>retention with</td>
<td></td>
</tr>
<tr>
<td></td>
<td>two-stage etching</td>
<td></td>
</tr>
<tr>
<td>Direct carbon</td>
<td>Excellent resolution</td>
<td>Not applicable to</td>
</tr>
<tr>
<td></td>
<td>in situ studies</td>
<td></td>
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</table>

men, while extraction replicas lift particles from the specimen. The advantages and disadvantages of some typical replication techniques are given in Table 1.

Surface Replicas. Replication of a surface can involve either direct or indirect methods. In the direct, or single-stage, method, a replica is made of the specimen surface and subsequently examined in the microscope. While in the indirect method, the final replica is taken from an earlier primary replica of the specimen surface. Only the direct method will be considered in this article because it lends itself more favorably to on-site preparation. The most extensively used direct methods involve plastic, carbon, or oxide replica material. All direct methods except plastic methods are destructive and therefore require further preparation of the specimen before making additional replicas.

Plastic replicas lend themselves to in-plant nondestructive examination because of their relative simplicity and short preparation time. Plastic replicas can be examined with the light optical microscope, the scanning electron microscope, and the transmission electron microscope, depending on the resolution required. As illustrated in Fig. 2, the plastic replica technique involves softening a plastic film in a solvent, applying it to the surface, and then allowing it to harden as the solvent evaporates. After careful removal from the surface, the plastic film contains a negative image, or replica, of the microstructure that can be directly examined in the light microscope or, after some preparation, in the electron microscope. Double-faced tape is used to bond the replica to the glass slide in order to obtain large, flat, undistorted replica surfaces.

There are some significant advantages of the replica technique over the use of portable microscopes in the field (Ref 5):
- A permanent record of the specimen is obtained.
- Better resolution and higher magnification can be used.
- Contamination of the polished surface is minimized.
- Time spent in an unpleasant or hazardous environment is minimized.
- Scanning electron microscopy can be utilized.

Several materials, including acetate, acrylic resin, and rubber, can be used in the surface replica technique (Ref 5). The choice of material depends on the geometry of the component and the microstructural features to be examined.

In the acetate method, an acetate tape is wetted with acetone and applied to the surface; other less volatile solvents, such as methyl acetate, can be used when large areas are replicated. For improved resolution, the back side of the replica can be painted with any fast-drying black paint or ink prior to removal, or for the same effect, evaporated coatings of carbon, aluminum, or gold can be applied at a shadow angle of 45° to the front side of the replica after removal.

In the acrylic casting resin method, dams are required because a powder is mixed with a liquid on the surface to be replicated. After hardening, the replica can be examined directly in an optical microscope without further processing. If adhesion is a problem, a composite replica can be made of an initial layer of Parlodian lacquer before the acrylic layer is applied.

In the dental impression rubber method, uncured liquid rubber material (for example, GE RTV60 silicon rubber compound) is poured onto the surface to be replicated and is contained by a dam. After removal, the replica can be examined directly or can be coated for better resolution.

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Fig. 2  Schematic of the plastic replica technique

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Fig. 3  Positive carbon extraction replication steps. (a) Placement of plastic after the first etch. (b) After the second etch. (c) After the deposition of carbon. (d) The positive replica after the plastic is dissolved.
Extraction Replicas. Several different extraction replica techniques can be used to characterize small particles that are embedded in a matrix, such as small second-phase particles in a steel (see the article “Analytical Transmission Electron Microscopy” in Volume 10 of the 9th Edition of Metals Handbook). More detailed descriptions of the various extraction replica techniques can be found in Ref 7 and 8.

After careful preparation of the surface using normal polishing methods, the first step in producing an extraction replica is to etch the alloy heavily to leave the particles of interest in relief. In the positive carbon extraction replica, as shown in Fig. 3, a piece of solvent-softened polymeric film (cellulose acetate tape) is pressed onto the surface exposed by the first etch (Ref 5). Once the solvent has evaporated, one of two steps can be taken. The tape can be carefully pulled from the specimen to produce a negative of the surface, or the specimen can undergo a second etch to free the particles exposed by the first etch (Fig. 3). In the second etch, the specimen can be etched through the plastic; most plastics are quite permeable to etching solutions, and the specimen etches almost as rapidly as without the plastic film (Ref 9). Carbon is then evaporated in a vacuum onto the plastic replica. The carbon and plastic containing the particles now make up the positive replica. The cellulose acetate is then dissolved, and the positive carbon replica is allowed to dry. It should be noted that for the negative carbon extraction replica technique, vacuum deposition of carbon onto the surface of the specimen is required, and therefore this replica method is not applicable to NDE.

Microstructural Analysis

Crack determination is important to help establish the root cause of a potential failure in a component. After a preliminary evaluation of the crack to assess crack shape and length by using magnetic flux or dye penetrant, the replica method is then used on unetched specimens to assist in the crack evaluation. Figure 4 schematically shows the propagation of different types of cracks in a steel structure (Ref 10). Each crack has its own characteristics, and it is often possible to make a correct determination of crack type. It is important to determine whether the crack is the original defect or has been caused by service conditions or damage. Once the crack type is identified, the proper corrective action, such as eliminating a corrosive environment or reducing stress levels, can be attempted. Figure 5 shows the replication of surface cracks in a boiler tube.

Creep Damage. Creep defects cause the majority of failures in power plant components operating under stress and thermal load, and the replica method is especially suitable for the detection of these defects. Therefore, the replica method has become an especially important tool in the determination of remaining life in such components as boiler tubes, steam piping, and turbine components. The replica method reveals defects due to creep at a much earlier stage than other NDE techniques. Creep defects begin as small holes or cavities at grain boundaries or second phases. With time and stress, these holes or cavities can link up and form cracks that eventually lead to failure of the component (Fig. 6). Creep cracks are usually very localized, and they form in welds, bends, or other highly stressed regions. Determining the remaining life of components normally depends on assessments of regular inspections, as indicated in Table 2. Figure 7 shows a comparison of creep voids in a surface replica and the corresponding bulk microstructure.

Precipitate Analysis. The detection of various deleterious precipitates in components subjected to high temperature and stress can lead to improved life assessment
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**Fig. 6** Schematic of creep crack formation. Small cavities (a) link up over time (b) and form intergranular cracks (c) and eventually macrocracks (d).

### Table 2 Creep damage classification

<table>
<thead>
<tr>
<th>Class</th>
<th>Nature</th>
<th>Action</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>No creep defects</td>
<td>None</td>
</tr>
<tr>
<td>2</td>
<td>A few cavities</td>
<td>Reinspection after 20 000 h of service</td>
</tr>
<tr>
<td>3</td>
<td>Coalescent cavities</td>
<td>Reinspection after 15 000 h of service</td>
</tr>
<tr>
<td>4</td>
<td>Microscopic creep cracks</td>
<td>Reinspection after 10 000 h of service</td>
</tr>
<tr>
<td>5</td>
<td>Macroscopic creep cracks</td>
<td>Management must be informed immediately</td>
</tr>
</tbody>
</table>

Source: Ref 11

**Fig. 7** Comparison of creep voids in (a) a replica and (b) the actual microstructure.

**Fig. 8** Comparison of α-phase formation as seen in (a) a replica and (b) the actual microstructure.

Sigma phase is a deleterious FeCr compound that can form in some stainless steels, and its presence can severely limit remaining life. Extraction replicas have been used to determine the amount of α phase in the microstructure (Ref 12), and the amount of α phase has been directly related to the creep rate (Ref 13). Figure 8 shows an example of α phase in an extraction replica.

The composition of carbides, and their stability with time and temperature of exposure, can indicate the remaining life of a component. Extraction replicas have been used to evaluate carbides, and it has been suggested that changes in morphology and chemistry can be used to assist the estimation of effective exposure temperature for use in determining the remaining life of components (Ref 14). Figure 9 shows an example of precipitates extracted from a 200 000-h exposed sample, together with the accompanying chemical analysis.

**ACKNOWLEDGMENT**

The author would like to acknowledge the contributions of his colleagues A.O. Benscoter, S.D. Holt, and T.S. Hahn in the preparation of this article.
Fig. 9 Extraction replica of the microstructure (a) and the precipitate microchemical analysis (b) from an extraction replica

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