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Metallography in failure prediction – An *in-situ* approach

S. DAS
National Metallurgical Laboratory, Jamshedpur - 831 007, India

ABSTRACT

Microstructural study is a routine procedure for investigation of metallurgical failures. Conventional metallographic techniques causes further damage to the component under investigation. In-situ metallography is an alternative technique for studying the microstructure without impairing the integrity of the component. It is mainly used to assess the useful life of a component already under use.

INTRODUCTION

Metallography is the well known technique to reveal the microstructure. The properties of a metal is always dependent on its microstructure. By microstructural examination it is possible to indicate the performance of a metallic component. Microstructural examination is one of the important studies carried out for failure analysis and residual life assessment of metallic components.

Conventional metallography which is carried out in laboratory is destructive in nature. To over come this difficulty in-situ metallography which is non destructive is developed.

Conventional Metallography

Conventional metallography [A] is carried out with the following well laid down procedures.

1. Cutting a sample of suitable size from the component to be examined
2. Grinding one face of the sample successively with coarse to finer grit size emery (SiC) papers.
3. Polishing the ground surface on a rotating wheel cloth impregnated with levigated alumina
4. Etching [B] the polished mirror like surface with suitable etchant and cleaning i

5. Finally observing the microstructure under an optical or electron Microscope.

Conventional Metallography has been used since ages and a lot of developments in its procedures, instruments used, etchant and etching techniques have taken place to make this technique suitable to the needs of the industry and research. The main disadvantage of this technique is that, it is more or less a destructive one.

In-situ Metallography

In-situ metallography technique was developed as an alternative, nondestructive technique to study the microstructure. It could be applied to study the microstructure of a finished component without impairing the integrity of the component. It is also applied to assess the useful remaining life of a component already in use.

Portable, handy and lightweight instruments are available to perform the preliminary grinding and final polishing operations to get a scratch free area. Portable electro polishing and etching instruments are also available to reveal the microstructure. Finally either a portable microscope or the replication technique [E] is used for observing the microstructure.

IN-SITU METALLOGRAPHY PROCEDURES

About 2.5 cm diameter spot on the surface to be examined is ground by fixing self adhesive emery paper discs of same size on the rotating rubber tip of the portable grinder. This operation is performed successively with grit sizes of 80 to 600 in five steps followed by mechanical polishing with diamond paste. After grinding with one grit size the area is washed thoroughly with water. After the grinding operations are over the area is mechanically polished by fixing self adhesive polishing cloth, smeared with diamond paste and recommended lubricant, on the rubber tip of the grinder. The polishing is done normally in three steps with 6μm, 1μm and 0.5μm size diamond paste. Some times 0.1μm and 0.05μm alumina is also used. When the grinding and polishing operations are carried out carefully, a mirror like surface is obtained.

When electro-polishing follows grinding, the grinding operations are carried upto 400 grit size. For electro-polishing the tank of the portable electro-polisher is filled with suitable electrolyte, the polishing voltage and time are set correctly. The electro-polishing instrument is switched on and the specimen surface is anodically connected with clamp. When the probe carrying the cathodic electrolyte is pressed on the polished surface the electro-polishing action starts. If the polishing parameters i.e., electrolyte composition, polishing voltage, time etc., are chosen correctly a mirror finish surface without deformed layer adhering to it is obtained. This process also takes less time as compared to mechanical polishing. Mechanical polishing also gives scratch free surface with a minimum deformed layer which is removed by etching. Etching is done to reveal the structure. The differential electrochemical or, chemical attack on the grain boundary and different phases creates minute contours on the polished flat surface. When observed under optical microscope or scanning electron microscope these are revealed as separate entity resulting into the microstructure of the metal. Chemical etching is done by swabbing the polished surface with a piece of cotton wool wetted with etchant for a fixed time and then washed with water and alcohol.

Electro chemical etching is performed by using the same electro-polishing equipment with correct setting of etching voltage and time.

The etched surface can be examined with a portable microscope [D] with a magnification of upto 400 times. Microstructure can also be recorded if photographic [C] attachment with the microscope is available. When a portable microscope is not available or it is required to examine the microstructure at a higher resolution replication technique [E] is applied.

Replication Technique

Thin sheet (25 to 50 μm thick) replication tape (Cellulose Acetate) softened in acetone or, methyl acetate is carefully spread on the etched surface and uniformly pressed. The solvent evaporates after sometime and the dry replica is peeled off. The contact surface of replica will contain the impression of the minute contours on the etched surface. It is carefully preserved for further processing and observation in the laboratory. Schematic is shown in Fig. 1.

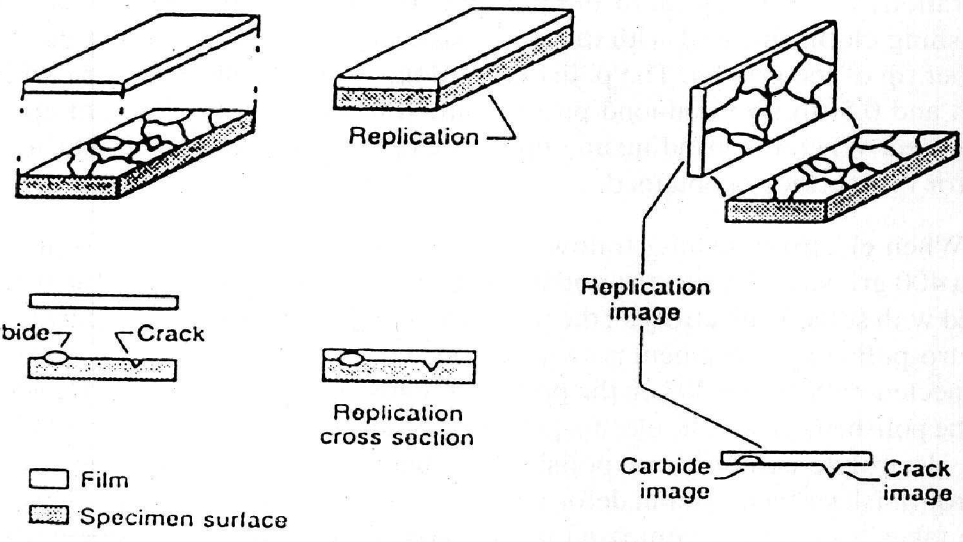

Fig. 1 : Showing schematic replication steps

SINGLE STAGE NEGATIVE REPLICA

The contours on the impression side of the replica are exactly opposite to those on the sample surface i.e., hills appear as valleys and valleys appear as hills. It is called single stage negative replica. Thin layer of metals like, gold, aluminium or chromimum is vapour deposited on the impression side of the replica to achieve a better contrast for optical microscopic observation. The thin metallic layer also makes the replica electrically conductive suitable to observe under scanning electron microscope.

DOUBLE STAGE POSITIVE REPLICA

When replica contains the contours exactly in the same manner as on the etched surface i.e., hills as hills and valleys as valleys, the replica is called a positive replica. It can be prepared by coating thin layer of carbon on the impression side of the replica. The carbon coated replica is then put in actone to dissolve the cellulose acetate leaving carbon replica floating in actone. This carbon replica is picked on grids for further observation in electron microscope. If the carbon replica is shadow coated with heavy metals like platinum, gold palladium the contrast in transmission electron microscopy can be enhanced.

EXTRACTION REPLICA

The precipitate morphology, their composition and identification could be studied by preparing an extraction replica [G] and observing in a Analytical Electron Microscope. For this purpose the polished surface is etched heavily to dissolve a layer of matrix leaving loosely held precipitates on the surface. 1% solution of Formvar (Polyvinyl Chloride) in chloroform is then carefully sprayed on the surface. When dried the thin formvar film is peeled off from the surface by backing it with softened cellulose acetate film. When peeled off from the surface, the loosely held precipitates get stuck to the dry formvar layer. It also represents the surface topography. A double stage carbon extraction replica can be prepared by the same procedure as described earlier, for observation under a transmission electron microscope with analytical facility. The chemical composition can be determined by EDAX, where as the crystal structure can be determined from the electron diffraction pattern.

APPLICATION OF *IN-SITU* METALLOGRAPHY

In-situ metallography have been used to evaluation microstructure where removal of sample for conventional metallography is either difficult or, forbidden to retain the integrity of the component. It could be used in failure analysis and residual life assessment. Damages such as, creep damage [F] due to cavity formation, sensitisation of stainless steel weldments, temper embrittlement [G] an microstructural degradation due to service exposure can be identified by th technique. Examples shown in Fig. 2.

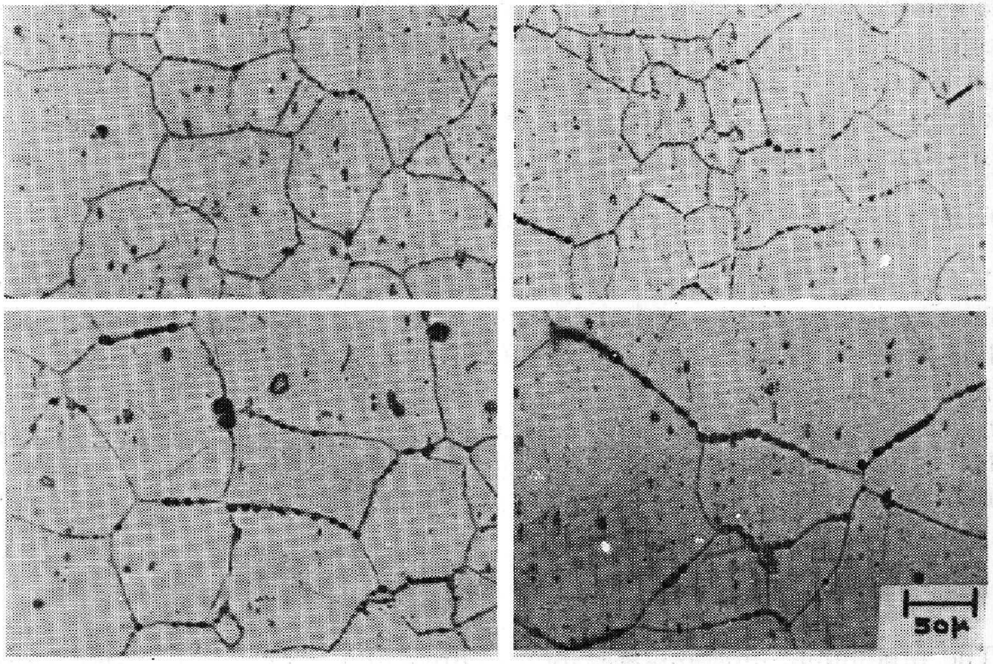

Fig. 2(a) : Creep damage due to void formation

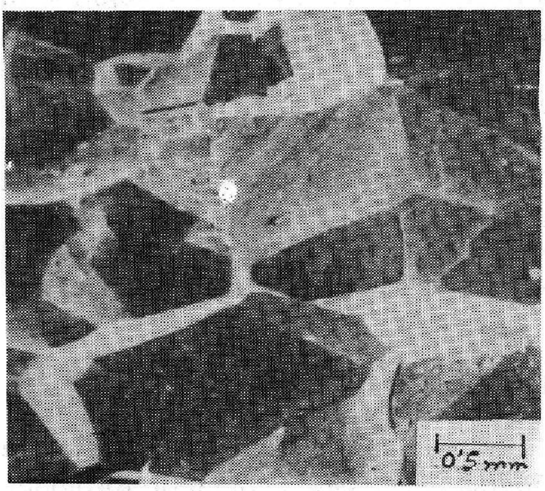

Fig. 2(b) : Temper embrittlement mainfested by intergranular fracture

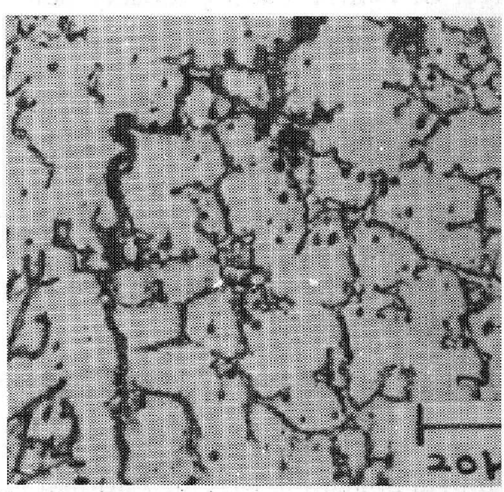

Fig. 2(c) : Crack propagation in stainles steel

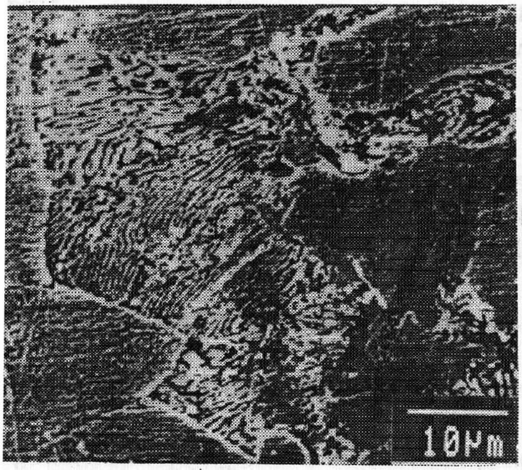

Fig. 2(d) : Lamellar carbides after short service exposure in Cr-Mo steels

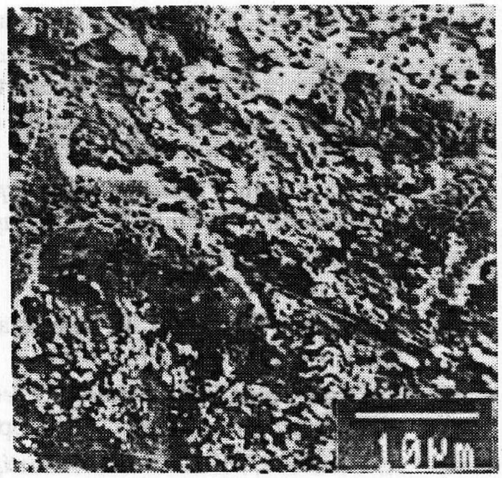

Fig. 2(e) : Spheroidisation of carbide due to long service exposure

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