

Metallurgy for Industries

Power | Petrochemical | Fertilizer | Chemical | Refinery | Engineering | Automobile

A Monthly News Letter

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In situ Metallography

A Powerful NDT tool for health Assessment

INTRODUCTION

In-situ metallography is one of the most important NDT tools in assessing the condition and life of plant facilities to avoid catastrophic failures and ensure safe and reliable operations of critical engineering equipments in power plants, petrochemical industry, fertilizer plants, cement plants, etc. Due to requirement of critical assessment of all those components under demanding working conditions, in-situ microstructural evaluation has become an essential technique for plant integrity assessment study. Previously, metallography was conducted by destructive means where it was required to cut and remove the sample. With advancement in development of portable equipments, the conventional metallography has evolved as nondestructive method. Now it is possible to monitor periodically the integrity of the facilities through in-situ metallography.

The operating pressure and temperature play an important role for degradation mechanism of metals like carbon and high alloy steels in the process industries. Adding alloying elements increases component's ability to withstand arduous process demands by improving metallurgical condition of an alloy. Microstructure influences and governs properties like, Mechanical, Metallurgical, Physical and Corrosion Resistance of an alloy. It records subtle evidences of manufacturing processes like casting, forging, rolling and fabrication including welding. The evidences require to be decoded with the help of metallurgical knowledge, past experience and judgment, which are in form of tiny features like micro-constituents called as phases, grain shape etc. It is a result of the metallurgical processes a component had attained required properties. When a component is exposed to high temperature and pressure conditions its microstructure degrades. It is also influenced by operational abuses like thermal fatigue, process upsets, high temperature corrosion and creep, etc. All these damages are revealed in in-situ metallography by way of change in microstructure. In-situ metallography, if properly applied can produce valuable data, based on which forced outages can be avoided.

Microstructure of the Month



Magnification: 400X

Etchant: 10% Ammonium Persulphate

MOC: ASTM A 240 Gr 316L

Observation: Microstructure shows fine-grained worked austenitic structure with twins. No carbide precipitation is observed along the grain boundaries. Presence of heavy trans-granular SCC crack is observed.

Cause: The onset of TGSCC seems to be from Chloride attack on account of chloride found in EDS analysis.

IN-SITU METALLOGRAPHY

In-situ metallography involves location selection, mechanical grinding and polishing /electrolytic polishing, electrolytic etching or chemical etching, replication and microstructure observations. The typical kit of in-situ metallography comprises of portable grinder, light grinder with variable speed controller, electrolytic etcher/polisher, microscope and variety of consumables. The consumables are listed as self-adhesive polishing papers of different grit size, self-adhesive sylvet cloth, solvents, water bottles, diamond paste, suspended alumina, electrolytes and replica films.

To get the maximum benefit from this technique, selection of the location to gather microstructure details is of paramount importance. The selection has to be based on the judgment of the experienced metallurgist who has adequate knowledge of degradation mechanism pertaining to the system. Initially, small area is mechanically polished with rough grinding to remove oxide scales from the surface. With the use of self-adhesive papers from course to fine grain size- about 1000 grit finish, progressive mechanical polishing is done. Thereafter, suspended alumina is used with silvet cloth for fine polishing up to 5 micron finish. Finally, diamond paste is used for polishing to attain scratch free mirror like finish. Either mechanical or electrolytic polishing is adopted for the final polishing process. Figure 1 shows photographs of in situ metallography equipment.

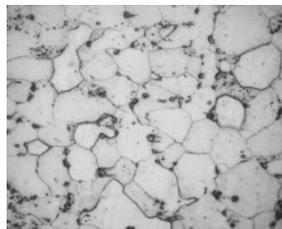
Then the etching is carried out to differentiate the micro-constituents present. Normally, one to two etch-polishing are provided to get good microstructure without any disturbance of the surface. It is essential to view the microstructure with a portable optical microscope having magnification up to 400X prior to replication. Replication is a last stage where wet cellulose acetate tape is pressed on the etched surface. Gentle pressure is applied so that the microstructure diligently gets transferred on the tape. It is then viewed under optical microscope by an experienced metallurgist. The tape can be self-refractive or it can be painted. There are different methods of replica techniques - castrastic techniques or extraction replica is some time used. Further enhancement in the contrast is achieved by gold sputtering, and then replica structure can be viewed under scanning electron microscope for high resolutions. The main advantage of the extraction replica technique is carbide precipitation at elevated temperatures can be found out. Figure 1 shows the flow chart of replication process.

DAMAGE MECHANISMS

Commonly observed damage mechanisms in carbon steels and alloy steels are as follows:

Graphitization

Prolonged exposure of plain carbon steel to a temperature around 400 Celsius decomposes pearlite into iron and graphite which reduces the mechanical strength. It can severely embrittle the steel when the graphite nodules form.

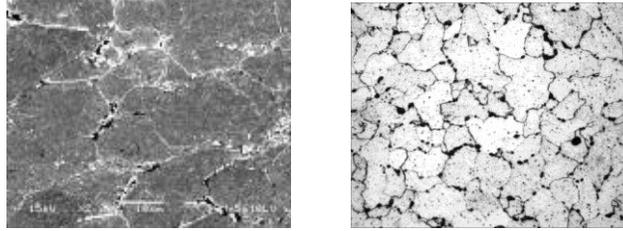


Degradation of Pearlite & Bainite

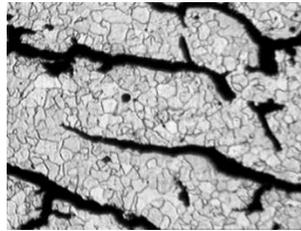
Prolonged high temperature exposure of carbon and low- alloy steel material, result in pearlite colonies getting transformed to spheroids. Chromium retards the process of spheroidization. In some of the power plant components having bainite in the microstructure is essential for the enhancement of creep resistance. Nevertheless, bainite microstructure also degrades into globular carbides in ferrite after prolonged exposure of the component at elevated temperatures.

Creep Damage

Time related, temperature dependent deformation under stress is known as creep. The degradation is stage wise and it can reveal the initiation in the microstructure as creep voids, orientation of creep voids and micro cracks.

**Hydrogen Attack**

Boiler feed water and steam corrosion and hydrogenated atmosphere can lead to formation of nascent hydrogen, some of the hydrogen atoms will then diffuse in the steel, where they react with iron carbide to form ferrite and methane gas. This will lead to micro cracks and lowering of the strength of material.

**Thermal Fatigue**

Thermal cycling having repetitive expansion and contraction develop thermal stresses that may eventually result into fatigue cracks. It is common with rotor shaft and batch type of reactors where there is wide variation of temperature.

High Temperature Oxidation

Oxidation occurs at the grain boundaries and then gradually penetrates inside. The thickening of grain boundaries in plain carbon steel is easily noticed with its differed response to etching.

Decarburization

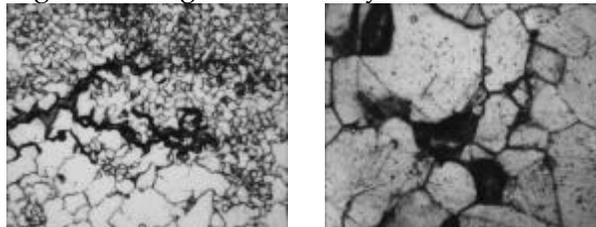
Removal carbon from the surface and edges is normally seen as formation of ferrite at the expense of pearlite.

Grain Coarsening

Prolonged high temperature exposure will result in grain coarsening and thereby the strength of the material is reduced.

Intergranular Corrosion

The weakening of the grain boundary takes place due to preferential corrosion on account of deposition of chromium carbides. Depletion of chromium takes place in the vicinity where the corrosion resistance drops drastically in a highly localized region of the grain boundary.

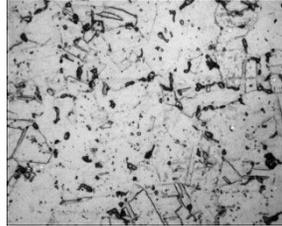
**Stress Corrosion Cracking**

Presence of internal stresses in stainless steel especially at welded joints or cold worked regions can induce network of fine micro cracks when either the steel is sensitized or surrounding is conducive having chloride and other ions. It is one of the most heinous kinds of degradation mechanisms that can lead to catastrophic failures without any warning.



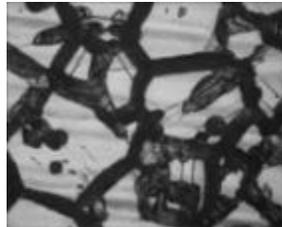
Sigma Phase

Sigma phase is an inter-metallic compound, non-magnetic in nature which normally forms when the steel is exposed to in the temperature range of 580-960°C. The presence of delta ferrite makes it more vulnerable for sigma phase formation. It is brittle in nature and undergoes contraction when it is formed. Thus, micro cracks are likely to develop and steel becomes highly brittle.



Carbide Precipitation

The precipitation of alloy carbide takes place at the grain boundaries making the steel brittle with reduced corrosion resistance.



Degradation by way of second phase formation is also more common in Nickel base and super alloys. The technique of in-situ metallography is also applicable Titanium, Aluminum and copper base alloys. However, bulk usage in industry revolves around steel and stainless steel.

CONCLUSIONS

In situ metallography when properly applied can generate valuable information for safe, reliable operation of process plants. Periodic monitoring of microstructure can generate confidence in operation and provides useful guidelines for timely replacement decisions or helps life extension evaluations.

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For Further details Contact us at testing@tcradvanced.com , Ph: +91-265-2657233