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IN-SITU METALLOGRAPHY FOR THE PLANT HEALTH ASSESSMENT STUDY AND FAILURE INVESTIGATION

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ABSTRACT

As an NDE technique, In-situ metallography is considered important for assessing the health of the equipment, which operates under different plant conditions. The acceptance of in-situ microstructure assessment is from the fact that industry needs safe, trouble free and productive operations by adopting to predictive maintenance approach. The in-situ metallography has the strength to meet these requirements. Critical components of Oil and Petrochemical refineries, Power generation units, Fertilizers, Chemical industries are subjected to variety of hostile environments that necessitate microstructure assessment to monitor in-service degradation.

In past, inspection intervals were based on historical experience and engineering judgments. In recent years methods have been developed to set the inspection locations based on risk by understanding the damage mechanisms. Microstructure, if properly analyzed can find out onset of early damage and corrective actions may be initiated well in advance.

Replica test is also known as field metallography where location selection is of paramount importance. Here the knowledge of operation, damage mechanism and metallurgy is essential to undertake in situ metallography for fruitful assessments. Unlike conventional metallography techniques, here the microstructures are prepared in such a way not cause damage to the material, which are analyzed at the same time to derive correct metallurgical information. The process of in-situ metallography involves the surface preparation with mechanical / electrolytic polishing methods to develop the true microstructure by suitable etchant. Then microstructure is transferred to a plastic tape and examined under high-powered optical microscopes / SEM.

Database for microstructure degradation are developed by understanding the failure mechanism and its microstructural signatures. The present paper is aimed at sharing the experience of microstructure degradation of components subjected to high temperature / pressure with failure case studies. It will allow plant manager to understand the insitu metallography technique in detail and assist them in conducting real-time component condition monitoring and health assessment.

INTRODUCTION:

Various NDT tests are carried out on operating plants for various reasons like, condition assessment, life assessment to avoid failure and for safe and reliable operation. Due to critical assessment requirement of plant components, microstructural evaluation of the component has become powerful tool. Earlier metallography was conducted by destructive means where it was required to cut-remove the sample and it was done in laboratory. But with advancement in the technology and development of portable equipment the process of metallography is converted into non-destructive method. Hence, it is possible to monitor periodically the response in degradation vis-à-vis operating conditions.

Pressure and temperature plays an important role in degradation mechanism for alloys like plain carbon, medium carbon and high alloy steels used in different process plant application. Principally, addition of alloying elements increases ability to withstand severe process demands by generating proper metallurgical conditions in an alloy. Microstructure plays an important role in controlling properties like, Mechanical, Metallurgical, Physical and Corrosion of an alloy. Controlled microstructure is a result of the metallurgical process, component had undergone. When microstructure is exposed to high pressure-high temperature it degrades. Microstructure responses to the high temperature-high pressure and various operating abuses like thermal fatigue, process up-sets, high temperature corrosion can be recorded by in-situ metallography. In-situ metallography if properly applied can produce valuable data by which forced outages can be avoided.

Location selection of in-situ metallography is of paramount importance as it decides the condition of the plant components. Location selection is done by understanding criticality of components in operation and failure history of the equipment. It should be representative of the most affected area from the operational condition by anticipated damage mechanisms.

2) IN-SITU METALLOGRAPHY:

The technique of in situ metallography involves location selection, mechanical grinding & polishing / electrolytic polishing, electrolytic etching or chemical etching, replication and microstructural observation.

Equipment and consumables used for in-situ metallography is shown in Photo. 1. The 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 can be listed as self-adhesive polishing papers of different grit size, self-adhesive velvet cloth, solvents, water bottles, diamond paste, and suspended alumina, electrolytes and replica films. Sketch. 1 is the flow chart showing various steps involved in-situ Metallography.

2.1) LOCATION SELECTION:

The location is selected on the basis of a careful analysis of the involved components. Because of the local nature of replica inspection, the selected position must be most critically representative one in the anticipated damage mechanism. There are two types of considerations mentioned hereunder.

a. Mechanical consideration, where parameters like stress, vibrations, bends, weld / HAZ and stress generated from due to self-weight of components in addition to the operating stresses.

b. Process considerations; where parameters like Temperature, Pressure, Flow rate and reaction with the environment are taken in to account.

For instance, in a typical examination of welds normally sets of 5 spots across the weld are usually examined. These 5 spots are positioned in the two base materials; the two heat affected Zones (HAZ) and Weld metal so that all the microstructure regions are covered.

2.2) VISUAL EXAMINATION AND GENERAL REQUIREMENTS:

Visual examination is done to assess the surface condition and accessibility for the person with equipments. It must be possible to keep the test position clean, dry and free of dust. The temperature of the examined metal must be ambient. When the dimensions and the wall thickness are critical, precautions must be taken to avoid excessive removal of metal surface to the examined component.

2.3) MECHANICAL GRINDING & POLISHING:

Small area of 1 sq. inch is rough ground to remove oxide scale or decarburized layer formed in operation. To keep in-situ metallography investigation non destructive, the total material removed by grinding must not exceed 0.5 to 1 mm.

On a small rotating shaft, abrasive papers at least in three steps with successively finer grits of paper ending with emery paper No 600 are attached.

On each type of emery paper, the grinding time must be three times the time needed to remove the traces from the previous grinding.

If a weld or repair weld to be examined Macro etching must be carried out between grinding and polishing. This makes the weld metal and heat affected zones visible. The polishing is done in one of the following two ways

1. Electro polishing with portable electro polishing equipment
2. Mechanical polishing with polishing disk via 800, 1000 grit finish and with the help of diamond paste to achieve 5 micron and 1 micron finish. Then suspended alumina is used in final polishing.

When macro cracks are detected, mechanical polishing is preferred as it does not affect the crack faces strongly. If electro polishing is carried out it should be preformed after examination of the mechanically polished surface.

2.4) ETCHING:

The prepared surface is etched either chemically or electrolytically. Optimum care is necessary in etching the surface. Over etching or under etching will mislead the result. In chemical etching with the help of the cotton swab, etchant is applied on prepared surface; where as in electrolytic etching etchant is circulated or kept in a soaked cloth and necessary voltage is applied between anode (material to be etched) and auxiliary cathode.

2.5) MICROSTRUCTURE EXAMINATION:

Stage wise examination right from the fine polishing can result into true microstructure development. Final judgment of microstructure is arrived at with the help of portable microscope in which the magnification can be as high as 400 to 500x.

2.6) REPLICATION:

After ensuring the properly developed microstructure, a plastic tape made of Cellulose acetate material is soaked in Acetone and kept on prepared surface. By gentle pressure the microstructure features can be replicated on plastic tape. A tape can be self-refractive or if not it can be painted. There are various methods of replica technique like, Cast resin technique or extraction replica. For further enhancement in the contrast, sputtering with gold is done sometimes to study under the electron microscope for high resolution. With the extraction replica, analysis of carbon precipitated at elevated temperature can be found out. The process of in situ metallography being done in side a boiler in the Photo:2, Where as sketch:2, is the schematic representation of process microstructure development by replication process.

2.7) AFTER CARE OF REPLICA

After the preparation of replicas these may be coated with gold or other light reflecting and conductive material under vacuum to improve the contrast in the light optical microscope. It also makes it possible to use the coated replica in the scanning electron microscope. While being coated and examined in SEM, the replica should be exposed to a minimum of heat.

3) DAMAGE MECHANISMS THAT ARE DETECTED THROUGH MICROSTRUCTURE STUDIES

The principle deterioration mechanisms that could be detected by using in situ metallographic techniques are the microstructural degradation like graphitization, spheroidzation of pearlite, creep, thermal fatigue, hydrogen attack, carburization, grain boundary oxidization and embrittlement of microstructure etc. Aspects of each will be considered in turn. Depending upon the alloys used and microstructure conditions with expertise on interpretations skills many other mechanisms and damages can be predicted.

3.1) GRAPHITISATION:

Graphitization can take place in ferritic steels after exposure to high temperature beyond 400° C for extended time, owing to reversion the cementite in pearlite to a more stable graphite phase. It is a particular form of microstructural degradation that was normally observed frequently in petrochemical components. The mechanical strength is greatly reduced. Steel embrittlement takes place when graphite particles and nodules are formed. The graphitization can take place at the low temperature edge of weld heat affected Zone (HAZ) resulting in a row or band of aligned graphite nodules that may extend across the wall thickness and parallel to weld seam. This form of graphitization can result in significant reduction in loads bearing capacity and thus increase the potential for brittle fracture along this plane.

With the development of more stable chrome -molly steels its tendency is greatly reduced. However, it occurs from time to time both in petrochemical plants and in steam generators in which the temperature is high and the material is not entirely stable.

Photo. 3 and 4 indicates graphite formation in plain carbon steel after a prolong exposure to high temperature under microstructure..

3.2) DEGRADATION OF PEARLITE:

Prolonged high temperature exposure of carbon and low- alloy steels renders pearlite colonies into spheroids. However, addition of alloying elements like Chromium and Molybdenum retards spheroidzation. Photo. 5 & 6 shows globular pearlite due to high temperature exposure of steel material for prolonged use. The spheroidzation of pearlite reduces the mechanical strength of the steel and alloys steels. When complete spheroidzation occurs it needs replacement of the component,

3.3) CREEP DAMAGE:

Creep is one of the most serious high temperature damage mechanisms. It involves time dependent deformation and high temperature creep cracking, develops at grain boundaries in engineering components that fail over an extended time. These include boiler super-heater and other components operating at high temperature, petrochemical furnace and reactor vessel components and gas turbine blades. At higher temperatures, with local overheating, deformation may be localized with large plastic strains and wall thinning. At somewhat lower temperatures and under correspondingly higher stress level. The fracture is eventually inter-granule in nature.

Classification of creep damage in the components exposed to creep range has been made using the largely qualitative approach based on distribution of creep voids and micro cracks observed by in situ metallography. The three stages of creep and associated microstructure degradations are represented in sketch: 3. Basic guidelines for evaluation of creep damage and provide judgment on the inspection intervals are given In Table 1. Photo: 7 & 8 are the optical and SEM micro-photograph of Cr-Mo steel material exposed different creep levels.

In situ metallography used in conjunction with semi quantitative tools like hardness measurements indicating loss of tensile strength would permit remaining life assessment studies on components undergoing creep damage. A point worth noting at this juncture is that the chrome Molly steels are liable to fail by creep in a short time by displaying spheroidzation of carbides but with little void formation.

3.4) HYDROGEN ATTACK:

Hydrogen damage, arising particularly in petrochemical industries, can occur in carbon steels through diffusion of atomic hydrogen in to the metal, where it combines with carbon in Fe3C to form Methane and to eliminate pearlite constituent. This is a special case of microstructural degradation. It is less common today than in past because of the use of low alloy steels containing elements that stabilize carbides. Photo: 9 shows carbon steel in which carbides from the original pearlite has been converted to methane producing voids and loss of carbon form he microstructure. Photo: 10 shows complete cracking of steel microstructure due to methane formation and decarburization. Often recrystallization of ferrite takes place around some of the voids that is produced by combination of deformation under pressure of methane and elevated temperature.

3.5) THERMAL FATIGUE:

Fatigue, involving repeated stresses, can lead to failure at high temperatures as it does at low temperatures. In components operating at high temperatures it often arises through temperature changes that can lead to cyclic thermal stresses. Eventually thermal fatigue cracking would take place in the areas where temperature fluctuation and variations are more. The nature of cracks are trans-granular in nature. Photo: 11 shows crack formation In chrome molly ferritic steel due to thermal fatigue of a tube at the entrance of outlet header of secondary superheater boiler operating at 540° C. Photo. 12 shows development of thermal fatigue crack with SS 316 at the steam inlet of paper digester.

3.6) HIGH TEMPERATURE OXIDATION:

Under highly oxidizing atmosphere grain boundary oxidization takes place that penetrate inside. Thus, the thickening of grain boundaries in carbon steels can be seen with etching response in situ metallography Photo: 13 and 14 shows grain boundary oxidation damage on plain carbon steel surface, necessitated replacement of the component.

3.7) DECARBURISATION:

The carbon in the steel can react with oxygen and in the atmosphere at high temperature to get decarburization of the surface. The loss of carbon in the surface of steel can be found out from microstructure examinations. The absence of pearlite phase due to decarburization in a oxidizing atmosphere at high temperature can be identified in microstructure which is shown in photo:15.

3.8) GRAIN COARSENING:

Grain coarsening takes place with prolonged high temperature exposure which decreases the strength of the steel. This can be easily noticed by microstructure examination.

3.9) Embrittlement and carburization

Embrittlement from precipitations arise in a number of different ways for instance, sigma phase formation in austenitic stainless steels maintained at high temperature or cycled through the critical temperature range of 565 to 989 ° C causes loss of ductility and embrittlement. Ferritic stainless steels may be subjected to embrittlement phenomenon when held at or cooled over the temperature range 550 to 400 °C. If the temperature conditions are likely to lead to such effects, metallographic checks are employed after extended exposure prior to an unexpected rupture developing. Photo: 16 grain boundary sigma formation of SS 316 stainless steel tubes used in the high temperature service.

Carburization can produce brittle material when a component is exposed to carburizing atmosphere for extended time at high temperatures. Cracks can initiate from the brittle carburized layer, which has little resistance to bending. This can be detected by metallography.

Normally the damages are compared with the normal microstructures on comparison basis to judge the extent of degradations.

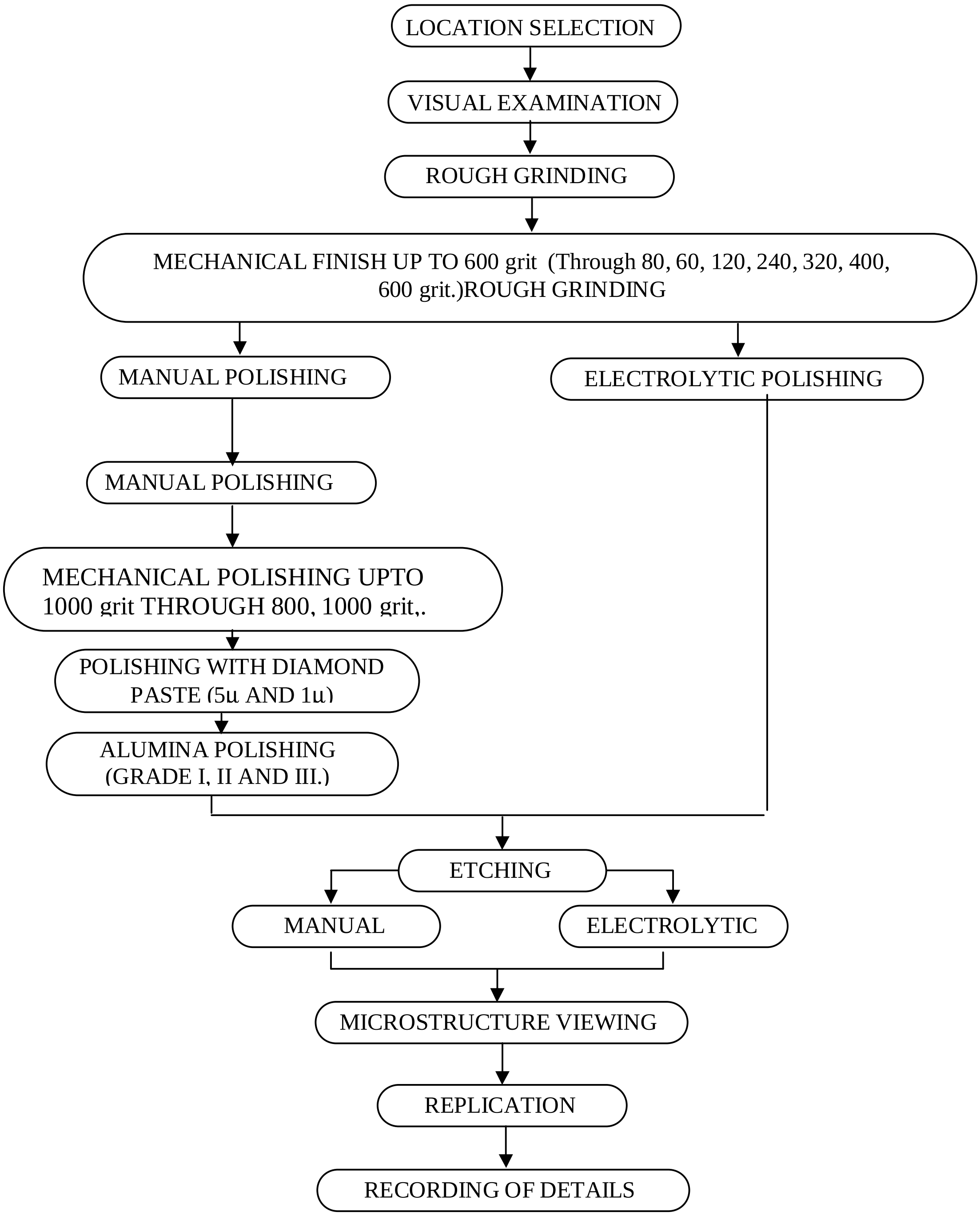
4) SUMMARY:

In situ metallography is a handy NDT tool to assess all those damage mechanisms discussed. It is possible to build a data bank vis-à-vis the performance of the component, which would furnish guidelines for RLA studies/ health assessment studies of process plant components. It is also used as condition monitoring tool by way of periodic checking to safeguard the working and safety of the components.

5) REFERENCES:

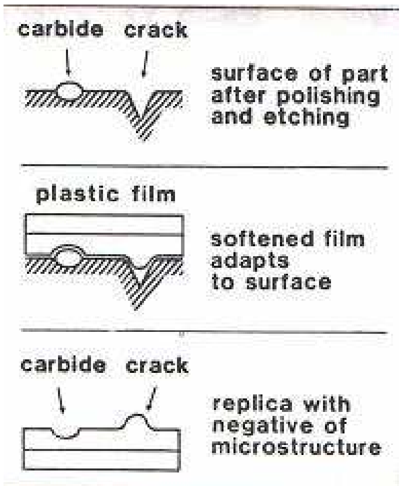
1) ASM Handbook, Vol.: 9, Metallography and microstructure.
2) Damage Mechanisms and Life Assessment of High-temperature Components. R Vishwanathan. ASM Interntional, 1989.
3) Non-destructive Life assessment of High temperature Components and Weldment, J. D. Parker& B. Wilshire Jeournal.of Press vessel and Piping 50 (1992) 337-347
4) NDT Replication avoids unnecessary replacement of power plant components. B. Neubuer & U. Wedel, Power Engineering May1994.
5) Data Bank of microstructure at TCR Engineering service Pvt., Ltd., Mumbai, India. www.tcreng.com

Photo. 1 shows the in situ metallography kit comprises of portable electrolytic ether/polisher with portable grinder and microscope.



Sketch:1 : Schematic representation of flow chart for in-situ metallography process.

Photo: 2 The actual process of in-situ metallography under progress in side Boiler.



Sketch:2 : Schematic representation of replication process.

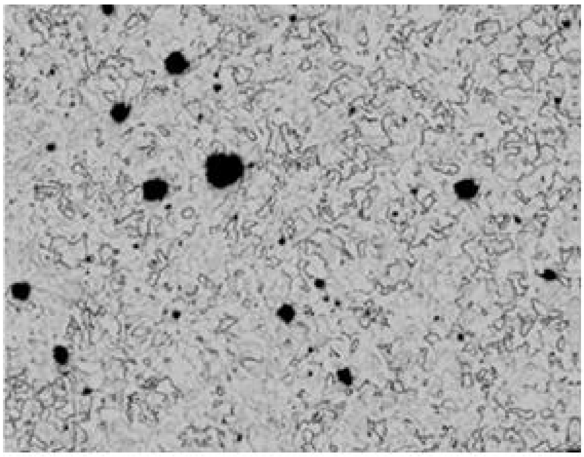


Photo:3 (100X)

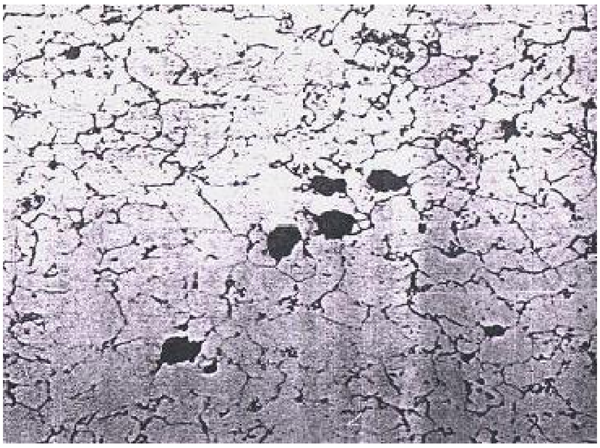
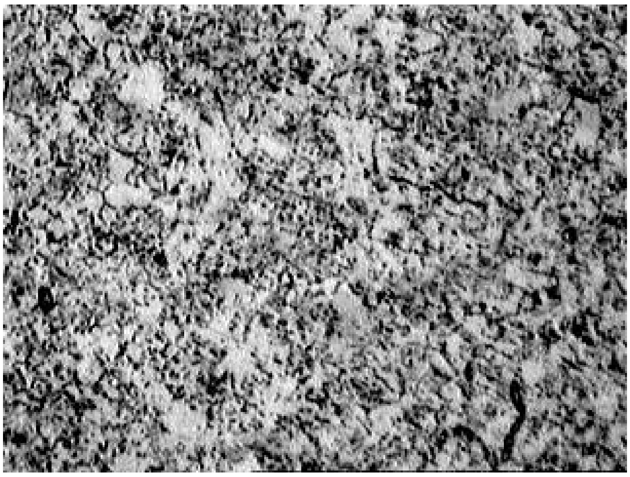


Photo: 4 (500X)

Photo: 3 and 4 indicates graphite formation in plain carbon steel after a prolong exposure to high temperature.



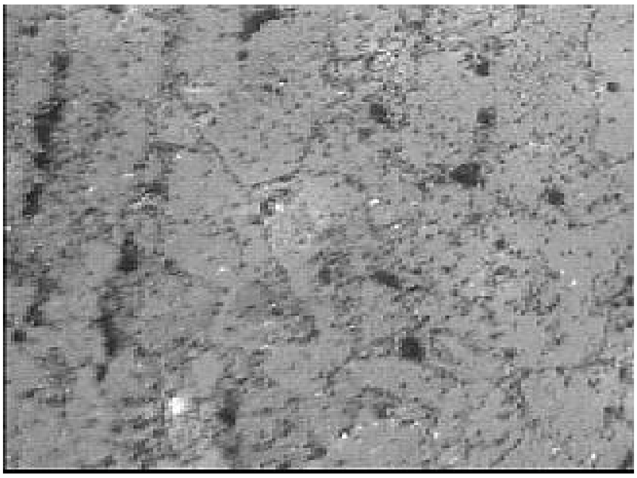
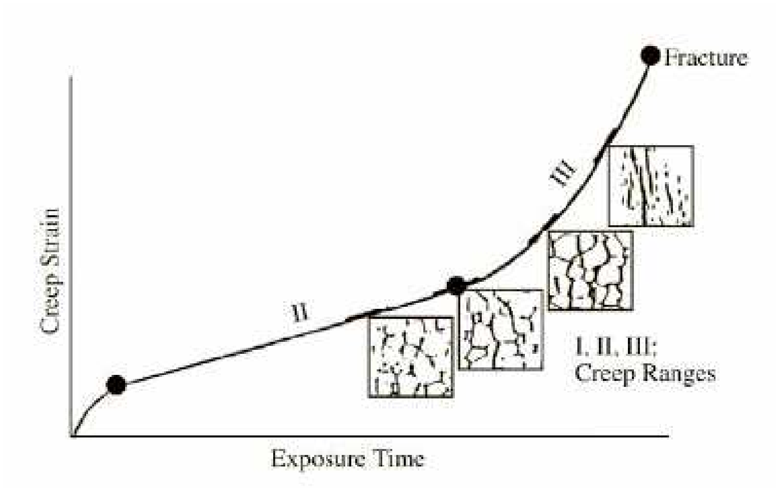


Photo:5 (200X)	Phtoto:6 (1000X)

Photo: 5 and 6, Spheroidzation of pearlite is observed – the upper microstructure shows in situ spheroidzation where as lower structure shows complete spheroidzation.



Sketch:3 Different stage of creep along with microstructure degradation stages

Class	Damage	Recommendations precautions
1	No creep cavities	None
2	Single cavities	Re-Examine after approximate 20000 hours of operation
3	Coherent cavities	Re-Examine after approximate 15000 hours of operation
4	Creep cracks (Micro)	Re-Examine after approximate 10000 hours of operation
5	Creep cracks (Macro)	The plant is contacted immediately

TABLE1: guide lines for inspection intervals and when to replace.

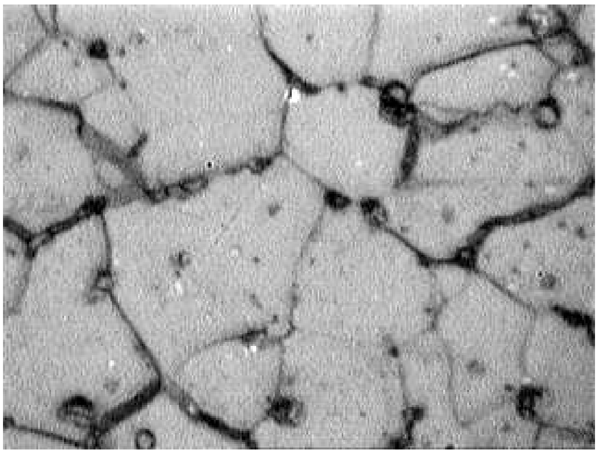


Photo: 7 (1500X)

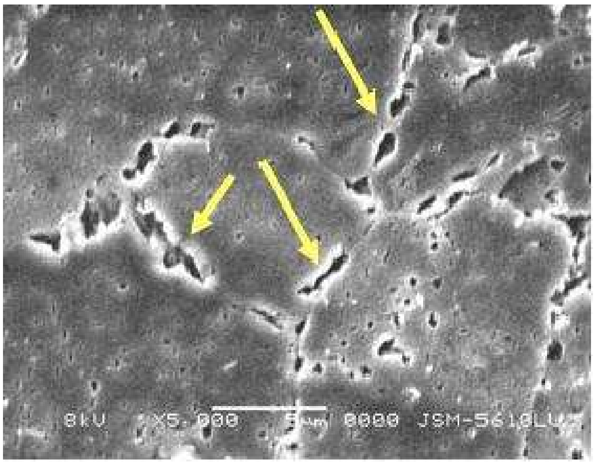


Photo:8 (5000X)

Photo. 7 creep voids and micro cracks observed by in situ metallographs under optical microscope. Photo. 8 shows SEM view of replicated microstructure of Cr-Mo steel showing clear presence of oriented creep cavities and micro cracks.

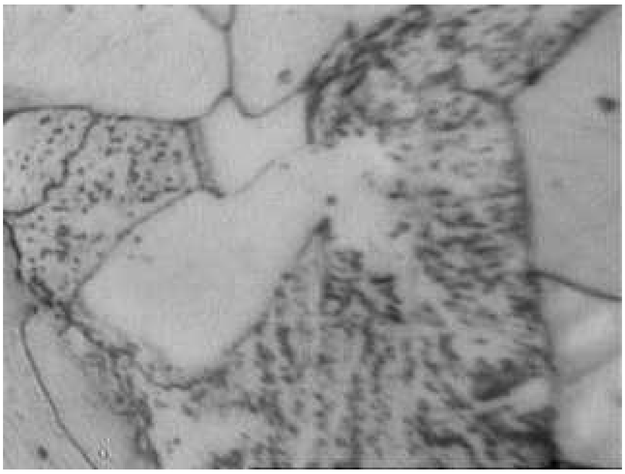


Photo.9 (1000X)

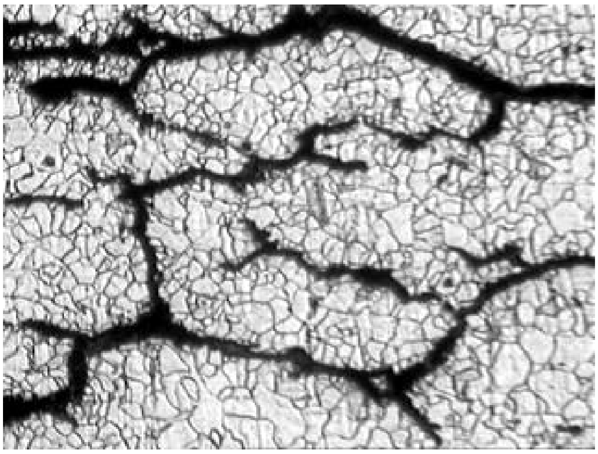


Photo.10 (100X)

Photo. 9 carbon steel in which carbides from the original pearlite has been converted to methane producing voids. Photo. 10 shows complete cracking due to methane formation of use of C-1/2Mo steel material under hygrogram service at elevated temperature.

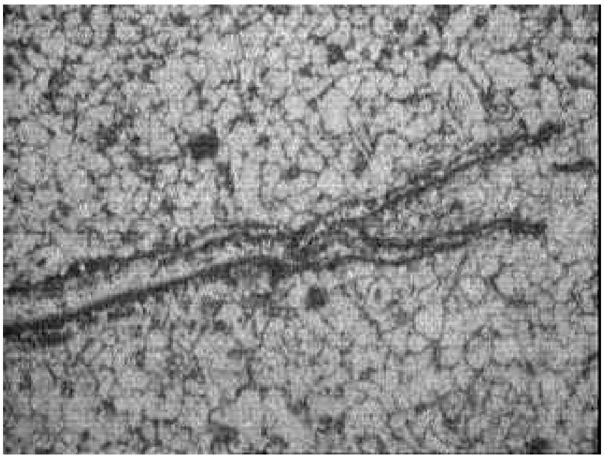
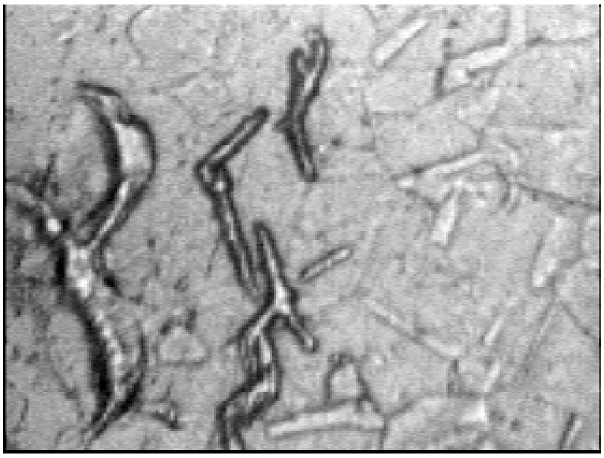


Photo: 11 (100X)



Phtoto: 12 (200X)

Photo: 11 crack initiation In chrome molly ferritic steel due to thermal fatigue of a tube at the entrance of outlet header of secondary superheater boiler operating at 540°
Photo: 12 shows development of thermal fatigue crack with SS 316 at the steam inlet of paper digester.

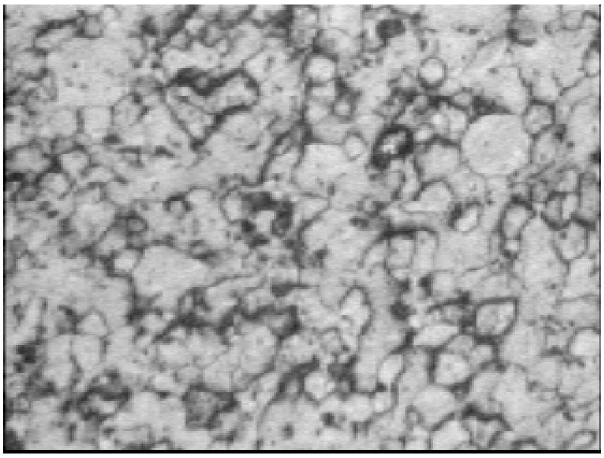


Photo:13 (100X)

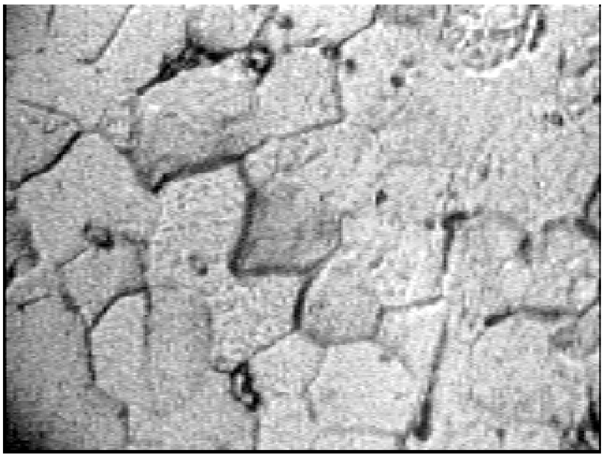


Photo:14 (1000X)

Photo: 13& 14 shows plain carbon steel of a cement kiln shell. The temperature rise occurred due to breakage of internal refractory and external surface got several oxidized.

Photo:15 (100X)

Photo. -15 essentially ferritic structure showing removal of carbon form the surface of the steel.

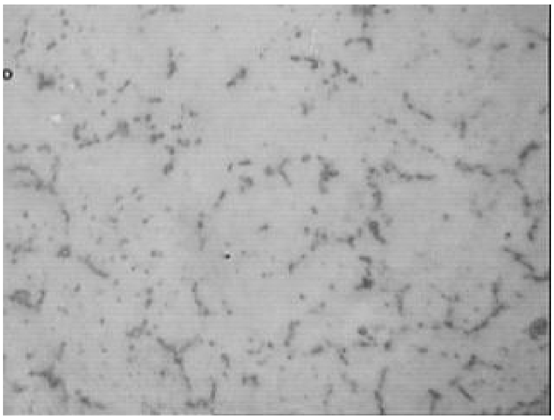


Photo: 16 (500X)

Photo: 16 Sigma phase formation of SS 316 material operating at 600ºC temperature for long time.