

Use of In-Situ Metallography for Plant Health Assessment Studies and Failure Investigations

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ABSTRACT

As a non-destructive technique, in-situ metallography is considered critical for assessing the integrity health of the equipment, which operates under various plant conditions. The acceptance of in-situ microstructure assessment as a predictive maintenance approach has been dictated by the industry's needs for safe and reliable operations. In-situ metallography can be useful in microstructure assessments for monitoring in-service degradation of critical components of oil and petrochemical refineries, power generation plants, fertilizers and chemical industries. In the past, inspection intervals were based on historical experiences and engineering judgments but the recent development of in-situ metallography could prioritize the inspection locations on a risk base analysis by understanding the damage mechanisms. Microstructure, if properly analyzed, can be utilized to find out the on-set of early damage and corrective actions which may be initiated well in advance. A data-base for microstructural degradation was developed by understanding the failure mechanism and its microstructural signatures. This paper includes case studies of microstructural degradation of components under high temperature and high pressure. The in-situ metallography technique was discussed in detail for monitoring the plant integrity.

1. 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 to ensure safe and reliable operations of critical engineering equipment in power plants, petrochemical industry, fertilizer plants, cement plants, etc. Due to requirements of critical assessment of all those components under demanded working conditions, in-situ microstructural evaluation has become an essential technique for conduction a plant integrity assessment study. Previously, metallography was conducted by destructive means where it was required to cut-remove the sample. With advancement in the technology in development of portable equipments, the conventional metallography has evolved as non destructive 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 steels 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 decode with the help of metallurgical knowledge, past experience and judgment, which are in form of tiny features of micro-constituents called as phases, grain shape etc. It is a result of the metallurgical processes a component

had undergone to attain 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 up-sets, high temperature corrosion and creep, etc. All these damages get recorded 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.

2. IN-SITU METALLOGRAPHY

The technique of in-situ metallography involves location selection, mechanical grinding and polishing /electrolytic polishing, electrolytic etching or chemical etching, replication and microstructure observations.

Equipment and consumables used in in-situ metallography are shown in Figure 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 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 the 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 sylvet 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 process is carried out to develop contrast 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 precipitated at elevated temperatures can be found out. Figure 1 shows the flow chart of replication process.

3. DAMAGE MECHANISMS

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

3.1 Graphitization

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

3.2 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.

3.3 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.

3.4 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.

3.5 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.

3.6 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.

3.7 Decarburization

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

3.8 Grain Coarsening

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

3.9 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.

3.10 Stress Corrosion Cracking

Presence of internal stresses in stainless steel especially at welded joints or at cold worked regions can induce network of fine micro cracks when either the steel is sensitized or surrounding is conducive to

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.

3.11 Sigma Phase

Sigma phase is an inter-metallic compound, non magnetic in nature which normally forms where the steel is exposed 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.

3.12 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.

Figures 1-4 show some of the microstructure damages and normal structures observed with the help of In-Situ Metallography.

4. CASE STUDIES

4.1 Sigma Phase Identification through In-Situ Metallography

Stainless steels are normally used in refineries in the range of service temperature where they are susceptible to sigma phase formation. Normally, the temperature ranges between 580-960°C. Corrosion resistance is drastically reduced and its formation leads to brittle failure of the components during start up and shut down of the plant. In-situ metallography is effectively used to find out presence of sigma phase with electrolytic etching. If it is finely distributed spread uniformly in the microstructure, then it is less harmful. However, if sigma phase is concentrated in form of lumps it is highly detrimental – more so when inter connected. During shut downs, it can trigger onset of corrosion finally leading to leakage. The percentage of sigma phase can be calculated using automatic image analyzer software that aids in taking a decision whether the component is to be replaced or used further. Figures 6 & 7 show the microstructures indicating copious sigma phase formation after 8 years of service of SS 304H pipe in FCC service of refinery. 15-20% of sigma in the weld metal was judged as the criteria that increases the susceptibility towards cracking. However, the decision of replacement depends on the morphology of the sigma phase and cracking tendency of the weld.

4.2 Reformer Tubes

Reformer tubes are used in fertilizer industries, refineries and petrochemical industries either for hydrogen generation or reforming of hydrogen along with the steam. The tubes act as individual reactors for the reforming process. The specific failure mechanism involves creep generated mid wall fissures that progress towards the OD. Mid wall fissures that were noticed under conventional NDT test like UT, Radiography and Creep determination by OD measurements with judicious approach. In-situ metallography was used to determine whether the tubes can be used further or be retired immediately. Figures 8 & 9 are the selected microstructures of in-situ metallographs from a reformer tube of HP modified alloy; comparing control virgin tube microstructure versus the presence of micro cracks in a

used tube having 12 years of service completed. This indicated the final stage of creep life and the tube was retired from the service in order to avoid failures during service.

4.3 Fire Damage Assessment

With all the precautions and safety measures, accidents do take place in process plants. Accidental fire damage is more common in refineries and petrochemical industries. The carbon and alloy steel components are normally analyzed by replica microstructure analysis. The components having intact pearlite are allowed for further use. Those having complete spheroidization of the pearlite are recommended for replacements. Those having partial spheroidization of the pearlite are recommended for further use with periodic monitoring through in-situ metallography. It requires great depth of metallurgical knowledge to judge the estimation of temperature based on physical metallurgy.

4.4 Remaining Life Assessment through Replica SEM for Boiler Components

Boiler pressure parts like super heater tubes, steam pipes and headers are operating in the creep range and they are designed for certain minimum lifetime. These components deteriorate continuously during the service, as a result of time dependent material degradation process like creep, fatigue, corrosion and oxidation. The limiting factors for the components operating at high temperature and pressure are creep life. Creep life is considered exhausted when it reaches the macro cracks through cavitation stage. The damage takes place in four successive stages formation of cavities, alignment of creep cavities, micro cracks and macro cracks. One can find out the life fraction consumed by identifying the prevailing stage in the microstructure. The successive stages are no creep cavities, isolated creep cavities, oriented creep cavities and the final stage of micro-cracks. Next monitoring interval is based on the level of degradation observed. Observing under SEM after gold sputtering can have most reliable judgments. Figures 10 & 11 are SEM micrographs of the main steam line showing oriented creep cavities with initial stage of micro cracks. It was recommended to monitor the cracks after one year through in-situ metallography and start planning for the replacement in next two years.

4.3 Sensitization in Stainless Steel

Austenitic Stainless steels are extensively used especially for high temperature and corrosion resistance applications. Carbide precipitation is more common while it undergoes processes like hot working and welding and during operation. Alloys are supplied in solution annealed condition where the carbon remains completely dissolved in austenite matrix and there is no carbide precipitation at the grain boundaries for better corrosion resistance. During the course of service or improper manufacturing it may undergo damage by way of precipitation of carbides at the grain boundaries. This can be easily detected by adopting in-situ metallography technique which can also assess the extent of damage. Figures 12 & 13 show presence of chromium carbide precipitations at the grain boundaries and is termed as ditch structure. Such material is susceptible for IGC (Inter-granular corrosion) and IGSCC (Inter-granular stress corrosion cracking) attack under favorable conditions.

5. 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.

6. REFERENCES

- 1) ASM Handbook Vol.: 9: Metallography and Microstructure.
- 2) R. B. Setterlund and W. M. Buehler: Nondestructive Metallography, Metallurgical Consultants, Inc., Houston, Texas.
- 3) R. Viswanathan: Damage Mechanisms and Life Assessment of High-temperature Components. ASM International, 1989.
- 4) J. D. Parker & B. Wilshire: Non-destructive Life assessment of High temperature Components and Weldments, Journal of Pressure Vessel and Piping 50 (1992) 337-347
- 5) B. Neubauer & U. Wedel: NDT Replication Avoids Unnecessary Replacement of Power Plant Components. Power Engineering May 1994.
- 6) J. L. Hau: Assessment of Fire Damage to Pressure Vessels in a Refinery Unit., Corrosion - May 1993.

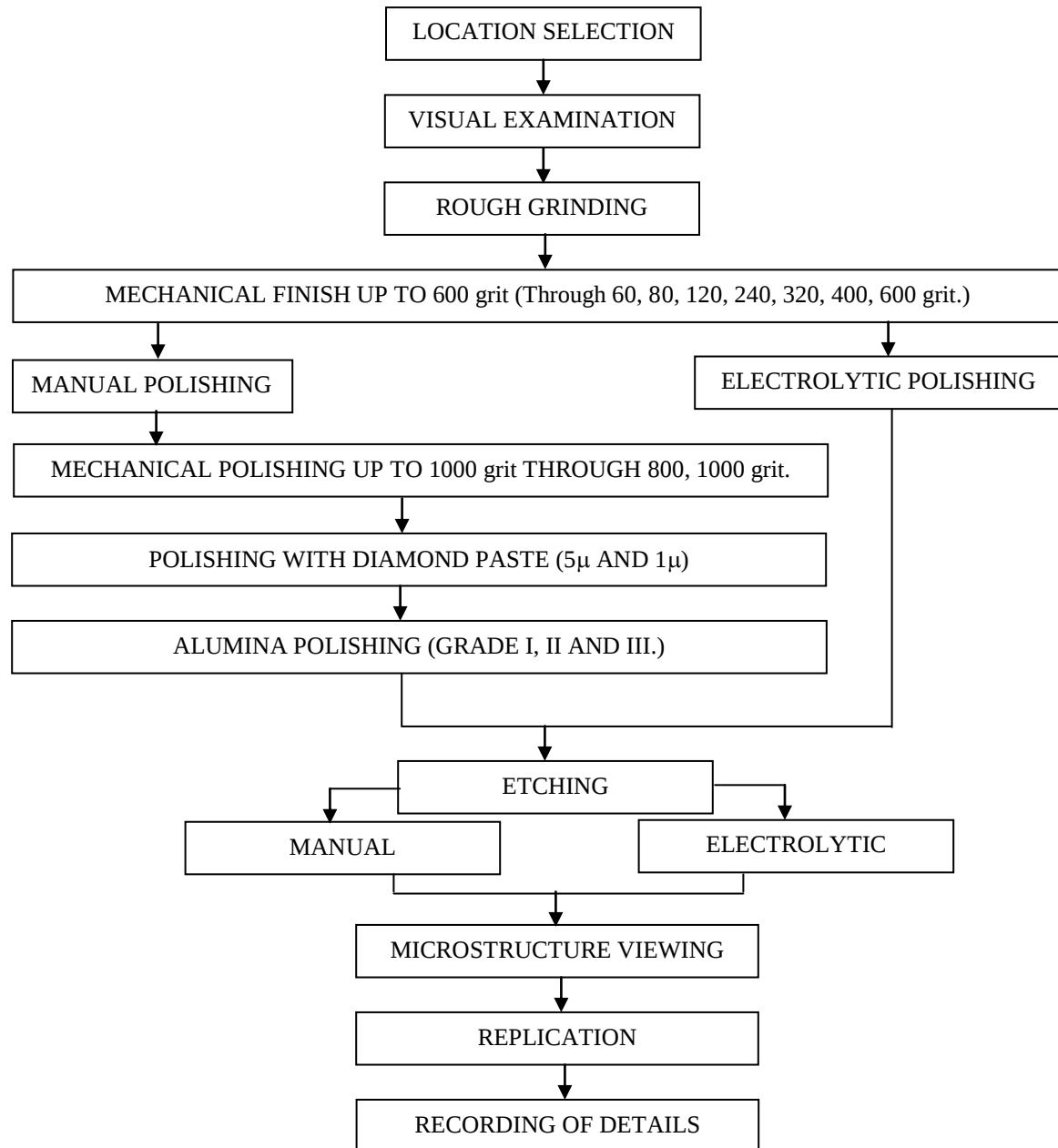


Figure 1 –Shows the Flow Chart of In-Situ Metallography Process



Figure 2 – The Equipment Used for In-Situ Metallography with Electrolytic Etcher/Polisher, Portable Grinder, Microscope, etc.

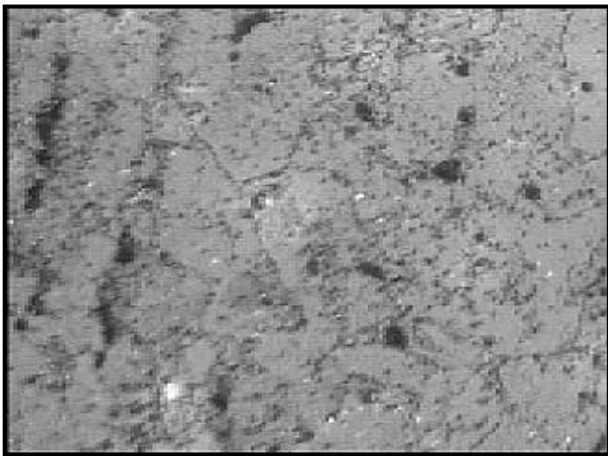


Figure 3 – Shows the Replicated Microstructure of Spheroidized Structure of SA 335 P11 Material, Exposed in the Service for 15 Years (400X)

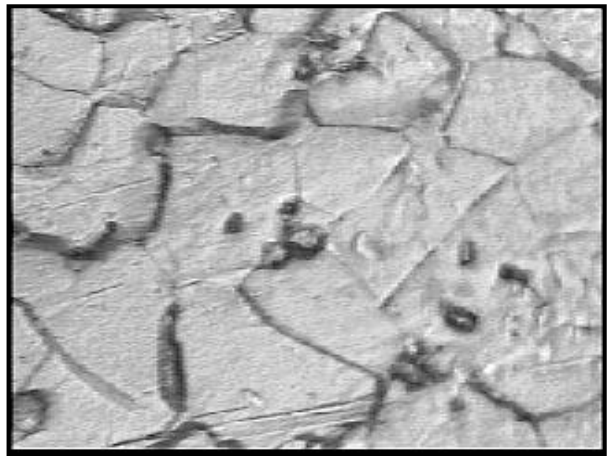


Figure 4 – Shows Microstructure of Plain Carbon Steel Material Showing the Grain Boundary Oxidation, Exposed to an Accidental Fire (400X)

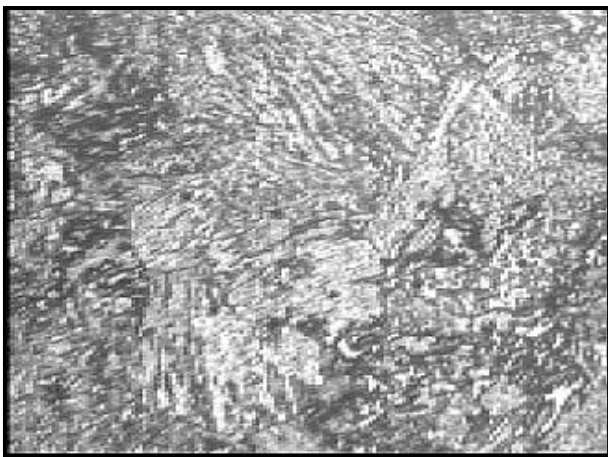


Figure 5 – Replicated Microstructure of Cr-Mo-V, HP Rotor Shaft Showing Bainitic Structure and Prior-Austenitic Grain Boundary (100X)



Figure 6 – Replicated Microstructure of 304 SS Reactor Showing Trans Granular Stress Corrosion Damage at HAZ (400X)

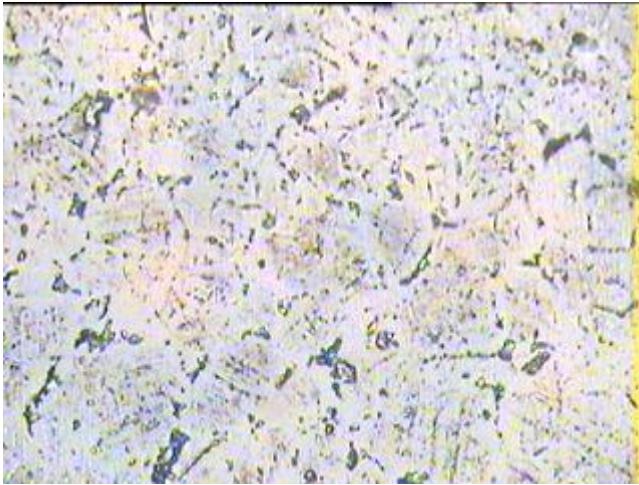


Figure – 6 PM (400X)

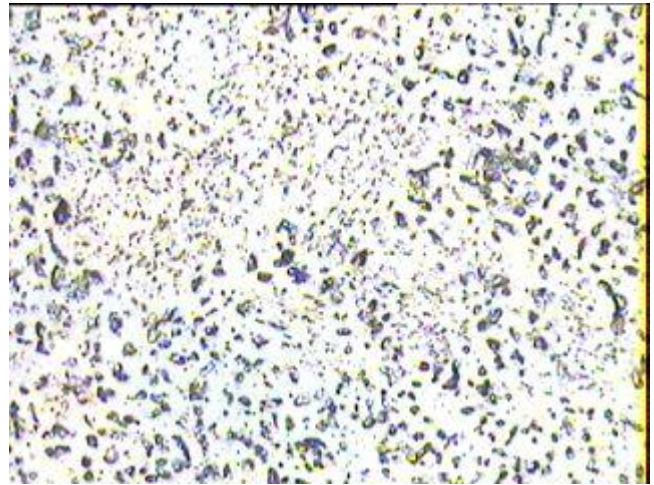


Figure – 7 Weld (400X)

Figures 6 & 7 are microstructures of SS 304H material showing presence of sigma phase formation at the grain boundaries and within the grains of parent metal and at inter-dendrite regions of weld metal.

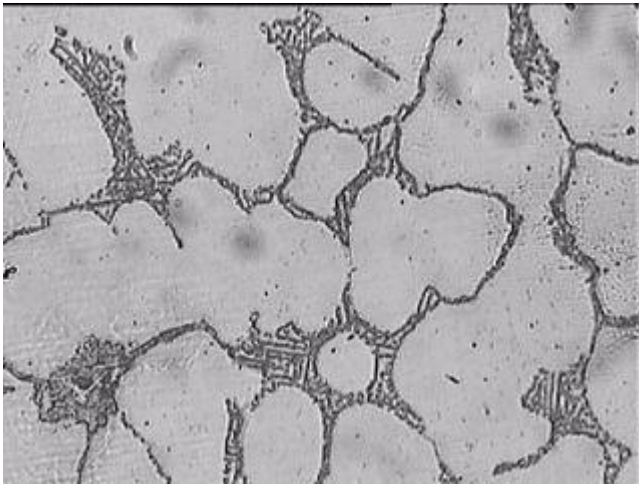


Figure 8 – Microstructure of HP Modified Alloy
Shows Inter-Dendritic Network of Primary Carbides in the Matrix of Austenite. Presence or Secondary Precipitations including Carbides are Observed within the Matrix (400X).

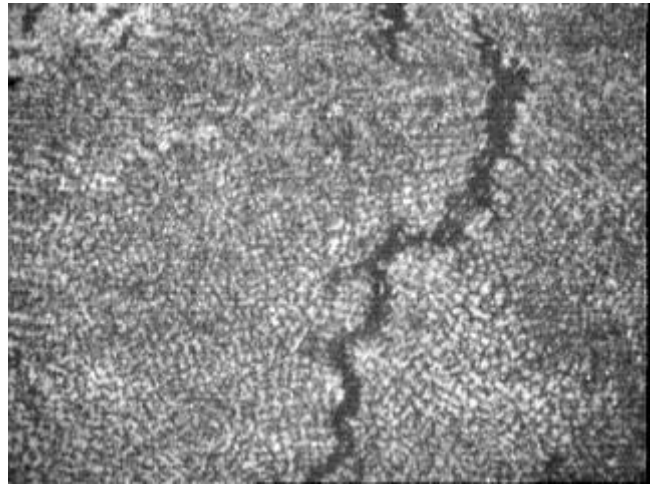


Figure 9 – Microstructure of HP Modified Alloy
Shows Primary and Secondary Precipitates including Carbides. Coarsening of Secondary Precipitation Observed within the Austenitic Matrix. Micro Cracks are Observed. Carbides Present at Dendritic Regions (100X).

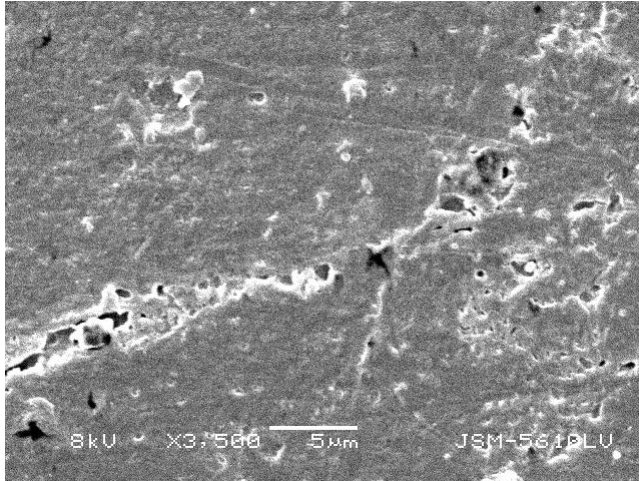


Figure 10 – SEM Picture of Parent Metal of Microstructure of P11 Material Showing Oriented Creep Cavities at Grain Boundaries (X3500)

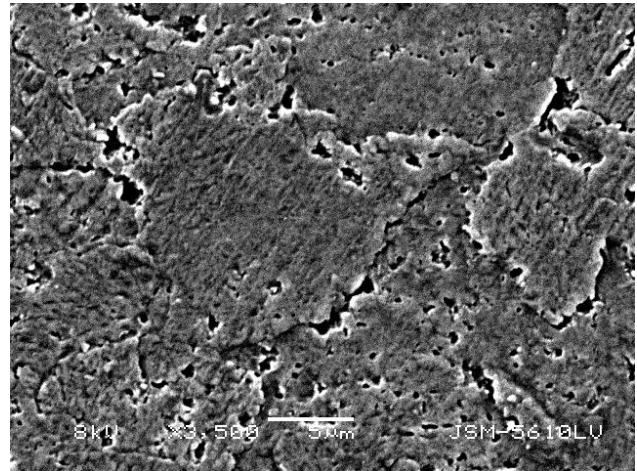


Figure11 – Microstructure of P11 Material Shows Initial Stage of Crack Formation at the Grain Boundaries (3500X)

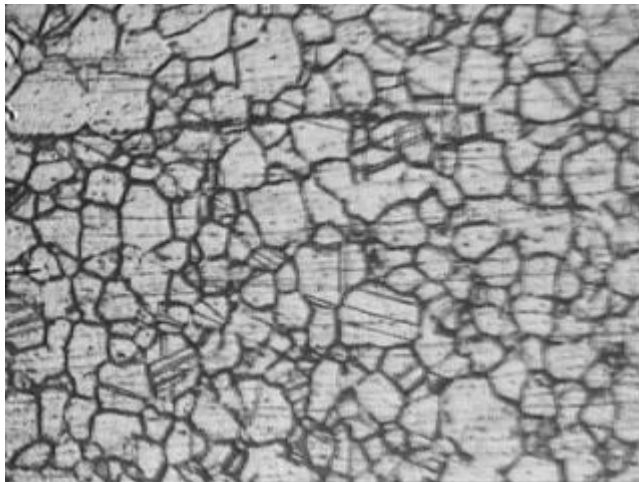


Figure – 12 PM (100X)

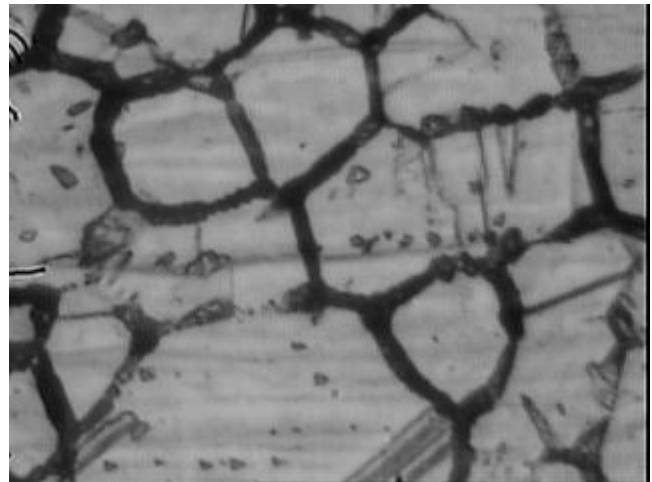


Figure – 13 PM (500X)

Figures 12 and 13 shows sensitized microstructure of Type 316 stainless steel indicating heavy carbide precipitation at the austenite grain boundaries.