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Field Metallography and Replication on Liquefied Natural Gas Assets: Non-Destructive Metallurgical Testing to complement other regular Non-Destructive Tests

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ABSTRACT

During plant shutdown maintenance, some components cannot be removed out of service immediately for metallurgical examination due to high cost involved and loss of production hours. The best alternative is to replicate the lab based metallography work under the field conditions. Field Metallography and Replication (FMR) also known as in-situ metallography is a powerful non-destructive test (NDT) tool used to examine the microstructure of the component when it is still in service. Moreover, FMR is also used to study the microstructural alterations for the fitness for service assessment.

This paper provides case studies of materials in Natural Gas Processing facility where FMR was used as an NDT tool without sectioning the component. This paper discusses the damage mechanisms such as sigma phase embrittlement, stress relaxation cracking and creep.

Keywords: Microstructure, Metallography, Replication, FMR, Sigma phase embrittlement, stress relaxation cracking, creep.

INTRODUCTION

Field metallography and replication (FMR) is a non-destructive technique (NDT) that can be used as a substitute for standard laboratory metallography. The test simulates the procedure of standard laboratory practice. The FMR technique involves making a replica of the polished and etched surface of a component with a cellulose acetate tape.

Grinding and Polishing

ASTM[1] E3[1] describes the surface preparation methods for laboratory based metallography. For field metallography and replication, the surface preparation method can be modified as appropriate to obtain polished areas free from scratches, micro pits and other artifacts that might yield false results.

The selected area has to be ground with grinding stone using potable grinder. Proper measures should be taken to avoid overheating of the ground area. Later, coarse grinding should be performed using Silicon Carbide 60 and then 100 grit emery papers.

Later, coarse to fine grinding should be performed using the emery papers in the order: 240→320→400→600→1200 Grits. It is very important to make sure that the polished area is free from scratches before proceeding to next grit. The best practice to avoid scratches is to ensure that the next grinding step is 90° to that of the previous step. After fine grinding, final polishing has to be performed with 6, 3 and 1 µm diamond paste until a mirror-like surface was obtained. Between each grinding and polishing step, the polished area should be cleaned with ethanol to remove debris and dirt. The polished surface should be examined using portable optical microscope to make sure that no scratches were present.

Etching

After ensuring that the polished area is free from scratches, etching with the suitable etchant has to be performed. However, selecting a proper etchant for replication is a real challenge. From our experience, the list of etchants for FMR of various materials are shown in the Table 1. The information on the list of etchants and the etching procedures can be obtained from ASTM E407.[2]

Table 1
List of Etchants

Serial No.	Material	Etchants for FMR
1	FERRITIC & MARTENSITIC STEELS	Nital: • 2 - 10 ml Nitric acid (HNO_3) • 98 - 90 ml Ethanol
2	AUSTENITIC STEELS	Diluted Aqua regia: • 40 ml hydrochloric acid (HCl) • 30 ml nitric acid • 30 ml water
3	SUPER AUSTENITIC STEELS	To reveal sigma phase: (Marbles reagent) • Electrolytic etch with 10 g $CuSO_4$, 50 mL HCl and 50 mL water. Adding few drops of H_2SO_4 before etching will help significantly. To reveal grain boundaries: • Swab with 40 ml HCl, 30 ml HNO_3 and 30 ml water

Replication with cellulose acetate replicas

ASTM E1351[3] describes preparation and evaluation of cellulose acetate replicas which have been obtained from polished and etched surfaces. Replica has to be produced as soon as possible after the etching. One side of the replica film should be wetted with acetone and the wetted side should be placed on the etched surface. The film should be pressed against the surface for at least 30 seconds. Allow the film to dry for 15 minutes. After the

(1) ASTM International, 100 Barr Harbor Dr., West Conshohocken, PA

replica film is dried, remove the film and the other side should be adhered to the glass slide for subsequent microscopy. Analysis and reporting of the extracted replicas are evaluated similarly as normal specimen under the microscope.

CASE HISTORY 1: FMR OF SUPER AUSTENITIC STAINLESS STEEL FLANGES

INTRODUCTION

Multiple cracks were noticed in a 14 inch (35.56 cm) diameter flange when it was welded to the pipe. The cracks were found on the bevel of the flange. The material of the flange is 254 SMO (6Mo steel) (UNS S31254) which is a very high end, molybdenum and nitrogen alloyed super austenitic stainless steel with low carbon content. The flange is solution annealed according to ASTM A182 F44 (UNS S31254); heated to 1150°C (2102°F) & held for 3 – 6 min/mm and then quenched in water. The flange is solution treated/annealed to ensure the solution of carbides that precipitate at lower temperatures.

Bearing in mind the limited quantity available, destructive test was not feasible. So, FMR was considered to check for the presence of microstructural defects at the cracks.

IN-SITU TESTING

Visual examination

Figure 1 shows the condition of the flange. Visual examination has revealed a crack emanating from the weld and propagating into the flange material. No evidence of cracks in the pipe material. Pipe was detached from the flange and the weld metal was gouged (Figure 2). Dye penetrant testing (DPT) was performed to verify the presence of undetected defects at the inner & outer surface of the flange (Figure 3). The test did not reveal any other significant defects on the surface except for the indication of the visible crack itself (Figure 4).

FMR

Before proceeding with FMR, positive material identification using X-ray fluorescence (XRF) gun was performed to identify the material grade and the results indicated that the material of the flange is 254 SMO (UNS S31254) super austenitic stainless steel.

FMR was conducted on the crack at the flange bevel. Area of around 1 sq. inch (6.45 sq. cm) at the crack was ground and polished to 1 µm surface finish and examined under portable field microscope to ensure that surface is free from scratches and replicas can be taken from the polished area. Then, the polished area is electrolytically etched with Marble's reagent to reveal sigma phase and then swabbed with aqua regia to reveal grain boundaries.

Figures 5 and 6 show that the microstructure around the cracks consist of sigma phase at austenite grain boundaries (GB). Sigma phase is the main particle precipitated at austenite grain boundaries in S31254 stainless steel during solution treatment from 950 - 1150 °C.[4] Chemical analysis test results showed that carbon content of the flange is <0.01 wt%, so possibility of sensitization is not possible.

Sigma (σ) phase is a hard and brittle intermetallic compound that usually forms from delta ferrite (δ) or directly from austenite (γ) in stainless steels in the temperature range 500 - 1000 °C. It develops rapidly near the temperature 900 °C - 1000 °C and will be sluggish at 500 °C. Presence of σ phase is undesirable as it imparts ambient-temperature brittleness, reduces mechanical properties, corrosion resistance and weldability of the material.

SMO flanges of other sizes were also checked for the presence of the sigma phase to determine the fitness for service capability of the flange material. Replica was taken at the inner surface (Figures 7 and 8) of the 24 inch (60.96 cm) diameter flange. Microstructure consists of equiaxed austenite grains with presence of sigma phase along grain boundaries and sensitized grains. Replica was also taken on the bevel (Figures 9 and 10) and the neck (Figures 11 and 12) of the 8 inch (20.32 cm) diameter flange. Observed microstructure is similar to the ones observed in 14 inch (35.56 cm) and 24 inch (60.96 cm) diameter flanges. Figures 13 and 14 show equiaxed austenitic structure that is free from sigma phase and sensitization; a typical microstructure.

LABORATORY INVESTIGATION

Before taking any decision on accepting or rejecting the material based on FMR results, laboratory investigation of 14 inch (35.56 cm) diameter flange was considered to check if the lab findings are in line with the results obtained from field testing.

Optical Microscopy

For metallography, a cross-section was taken from the bulk material away from the failed region (Figures 15 and 16). The sample was prepared in accordance with ASTM E3. Sample was electrolytic etched with Marble's reagent at 9.5V for 10 secs to reveal only sigma phase.

Microexamination of the two cross-sections revealed sigma phases which suggest that the material is not properly heat treated.

Microhardness

The microhardness was taken on the sigma phase. The microhardness test was done using the Vickers method in accordance with ASTM E384. The microhardness values of the sigma phase and bulk material are tabulated in Table 2.

Table 2

Microhardness values

	Hardness (HV)					
	Load (kgf)	**1**	**2**	**3**	**4**	**5**
Sigma Phase	0.200	310	407	324	480	341
Bulk Material	0.200	226	217	204	212	257

The hardness test results confirm that the sigma phase is a hard & brittle intermetallic compound which has higher hardness compared to the bulk material.

SUMMARY OF FINDINGS OF CASE STUDY 1

From the combined test results of in-situ testing and laboratory investigation, it is evident that sigma phase is present in the flange material. Inappropriate heat treatment is the reason for the quantity and type of deleterious phases existing in the flange material. Appropriate heat treatment and controlled chemical composition organize the phases in the material.

CASE HISTORY 2: FMR OF SUPERHEATER TUBES OF A BOILER

INTRODUCTION

Boiler tube failures are the most common cause of forced outages. So during a planned shutdown, ASME SA-213 Grade T11 (UNS K11597) super heater tube of a boiler was considered for in-situ metallography to determine if it had been exposed to temperatures higher than design operating temperatures.

FMR

Visual examination of the super heater tubes has not revealed any abnormalities such as cracking or bulging. Four locations were selected along the length of a tube for FMR. The tube dimensions are not reported. The selected location was ground and polished to 1 µm surface finish and examined under portable field microscope to ensure that surface is free from scratches and replicas can be taken from the polished area. Then, the polished area was etched with Nital for 30 seconds. The etched region was examined under portable field microscope to ensure that microstructure is revealed. If microstructure is not revealed repeat the etching step for another 30 seconds. One side of the replica film was wetted with acetone and the wetted side was placed on the etched surface. The film was pressed against the surface for 30 seconds. The film was allowed to dry for 15 minutes. After the replica film was dried, the film was removed, and the other side was adhered to the glass slide for subsequent microscopy.

In-situ metallography and replication revealed that the microstructure of the tube is not uniform (Figures 17 – 20). General microstructure of ASME SA-213 Grade T11 (UNS K11597) super heater tube consists of ferrite and pearlite. However, only ferrite phase was observed with some evidences of carbides at the grain boundaries. The microstructure along the tube OD was mostly decarburized and grain coarsening was also observed.

SUMMARY OF FINDINGS OF CASE STUDY 2

FMR findings indicate that the super heater tubes are overheated. As a proactive measure, the following steps as a part of condition assessment of the super heater tubes is recommended to be performed annually or semi-annually: 1) visual inspection with photo documentation, 2) oxide thickness measurements using ultrasonic or eddy current units, if possible 3) wall thickness measurements using ultrasonic thickness gage, 4) Dye penetrant testing, 5) hardness testing using potable hardness tester and 6) FMR at the same spots.

CASE HISTORY 3: FMR OF CRACKED IMPELLER

INTRODUCTION

During service, it was observed that impeller had cracked and resulted in forced outage. The material of the impeller is AISI 4340 steel (UNS G43400). The impeller dimensions are not reported. Destructive testing is not allowed as there were plans to repair and reuse the impeller to avoid further delays. In-situ metallography was considered to check the microstructure at the crack and to confirm that the material is uniform.

<u>FMR</u>

Visual examination of the impeller revealed that the crack was present at the hub (Figure 21). It was observed that the material was gouged at this location (as received condition) and the crack is restricted only to the gouged location. Four locations around the crack were considered for in-situ metallography. The selected location was ground and polished to 1 µm surface finish and examined under portable field microscope to ensure that surface is free from scratches and replicas can be taken from the polished area. Then, the polished area was etched with Nital for 30 seconds. The etched region was examined under portable field microscope to ensure that microstructure is revealed. If microstructure is not revealed repeat the etching step for another 30 seconds. One side of the replica film was wetted with acetone and the wetted side was placed on the etched surface. The film was pressed against the surface for 30 seconds. The film was allowed to dry for 15 minutes. After the replica film was dried, the film was removed, and the other side was adhered to the glass slide for subsequent microscopy.

Figurers 22 and 23 of the replicas 1 and 4 taken on the either side of the crack (at a distance of more than 100mm) has revealed that the base material has tempered martensitic structure typically seen in quenched and tempered carbon steels. Further examination also revealed retained austenite in the examined areas.

Replicas 2 and 3 were taken in the gouged location. Optical microscopy of replica 2 revealed recrystallized structure i.e., fine grains of ferrite and pearlite in some regions and the other regions had coarse ferrite grains and bainitic structure (Figure 24). Replica 3 is close to the weld. Optical microscopy of replica 3 revealed lower bainitic structure in heat affected zone close to the base metal (Figure 25). Microexamination points that the material is not uniform. The impeller material (quenched and tempered alloy steel) is repaired by the supplier and gouged location is clearly visible to the naked eye.

SUMMARY OF FINDINGS OF CASE STUDY 3

FMR findings indicate that the cracking is restricted only to the repaired location. The continuous material with uniform microstructure will have a better stability and integrity. The repair of the material will have adverse effect on the mechanical and corrosion properties due to variation in the microstructures.

CASE HISTORY 4: FMR OF FAILED SPOOL FROM A HYDROCARBON CRACKER UNIT

INTRODUCTION

In-situ metallographic replication and examination of a damaged spool from a cracking unit was performed to determine whether the spool steel had experienced any damage by exposure to high temperatures. The spool was manufactured from ASTM A106 Grade B (UNS K03006) carbon steel.

FMR

The selected location was ground and polished to 1 µm surface finish and examined under portable field microscope to ensure that surface is free from scratches and replicas can be taken from the polished area. Then, the polished area was etched with Nital for 30 seconds. The etched region was examined under portable field microscope to ensure that microstructure is revealed. If microstructure is not revealed repeat the etching step for another 30 seconds. One side of the replica film was wetted with acetone and the wetted side was placed on the etched surface. The film was pressed against the surface for 30 seconds. The film was allowed to dry for 15 minutes. After the replica film was dried, the film was removed, and the other side was adhered to the glass slide for subsequent microscopy. Optical microscopy of the replica gathered from visually unaffected area has revealed coalesced grains of coarse ferrite with some precipitated carbides at the grain boundaries (Figure 26). Typically ASTM A106 Gr. B (UNS K03006) material is delivered in the normalized condition e.g. the microstructure usually consists of fine equiaxed grains of ferrite and pearlite. Replica collected from the suspected location has exhibited heavy carbide precipitation and voids most likely caused by creep (Figure 27).

SUMMARY OF FINDINGS OF CASE STUDY 4

FMR findings revealed indications of creep damage which indicates possible prolonged exposure to temperatures near or exceeding the austenitising temperature (approx. 750 - 900ºC (1382 - 1652 ºF)).

CASE HISTORY 5: FMR OF REGENERATION HEATER COIL

INTRODUCTION

Multiple cracks were noticed in the pipe spool of a regeneration heater coil. The material of the pipe is ASTM A312 TP304H which is a heavily cold worked austenitic stainless steel.

IN-SITU TESTING

FMR

FMR was conducted on the cracked pipe spool at both unaffected and cracked locations. Optical microscopy of replica collected from unaffected area has revealed equiaxed austenitic structure with evidences of annealing twins (Figure 28). No microstructural anomalies were observed in the examined area. Optical microscopy of replica collected from cracked area revealed cracks in the examined area (Figures 29 and 30). Interestingly, the cracks were confined only to the regions with prior cold worked grains. The cracks were similar to stress relaxation cracking.

LABORATORY INVESTIGATION

Visual examination of the as received failed regeneration heater coil segment has revealed circumferential crack on the outer surface (Figure 31). No evidence of loose corrosion products on the coil.

Optical Microscopy

Longitudinal cross sectional sample was extracted from the tip of the crack and prepared in accordance with ASTM E3. Electrolytic etching was performed in accordance with ASTM E407. The etching process was done at 2.5V using 10% Oxalic Acid. Optical microscope was used to study the microstructure.

General microstructure should contain equiaxed austenite grains with presence of annealing twins typical of solution annealed stainless steel as the material is supplied in annealed condition after heavy cold working. However, during optical microexamination, coarse grains were observed along the outer surface. Besides, austenite grains with strain lines were also observed along the outer surfaces (Figure 32). Intergranular crack was observed.

Interestingly, even the unaffected coil segment has microstructural evidences similar to affected coil segment i.e., coarse grains, sensitized grains, intergranular cracks (not visible to the naked eye) and oxide filled grain boundaries (Figure 33). Presence of sensitized grains through thickness, along circumference and in both affected and unaffected regions indicates that the coil was operating in a temperature range susceptible to sensitization 520° - 800°C (968 – 1472°F). As the service temperature is 533 - 577°C (991 - 1071°F), which falls within sensitization temperature window, observed sensitized grains is expected.

Microhardness

Transverse cross-section was extracted from the tip of the crack for microhardness test using Vickers method in accordance with ASTM E 384 (0.200 kgf load). The hardness test was performed at the outer surface and midwall thickness (Figure 34). Grains with strain lines have shown very high hardness values compared to grains free from strain lines. Microhardness test results indicate that deformation induced stresses (residual) that are induced during cold working process are still existent in some of the grains at the outer surface.

SUMMARY OF FINDINGS OF CASE STUDY 5

From the combined test results of in-situ testing & laboratory investigation, the following conclusion can be drawn:

- The operating temperature (533-577°C; 991 - 1071°F) is almost overlapping with the stress relaxation cracking (560 to 799°C; 1040 - 1470°F) temperature of austenitic steels.
- Strain lines within some austenite grains indicative of residual stress and hardness values beyond 200 $HV_{0.2}$ indicates that the material is susceptible to SRC in the normal operating conditions in service.
- High temperature austenitic stainless steel materials with hardness less than 200 HV and free from internal/residual stresses are suitable/fit in the operating tempering range 533-577°C (991 - 1071°F).

CONCLUSION

From the case studies, it is evident that the steel microstructures could be successfully replicated and the in-situ findings are similar to lab based findings. So, FMR is a very important NDT tool that can assist clients in decision making on whether to remove the components from service or not to confirm the presence of defects that were detected in the regular Non-destructive tests.

FMR could be used to check for the presence of sensitization, stress corrosion cracks in the stainless steels, signs of overheating, grain growth, general microstructure, degradation of microstructure and other abnormalities that are close to the external surface.

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3. ASTM E1351-01(2012) (latest revision), “Standard Practice for Production and Evaluation of Field Metallographic Replicas,” (West Conshohocken, PA: ASTM).
4. Jianchun Lia, Wei Liang, Min Wub, Shoulu Zhang, Wei Zhang, “Microstructure evolution in the segregation area of S31254 stainless steel plate”, Materials Today: Proceedings 2S (2015) S319 – S324.

Figure 1: Photograph showing the flange with a crack.

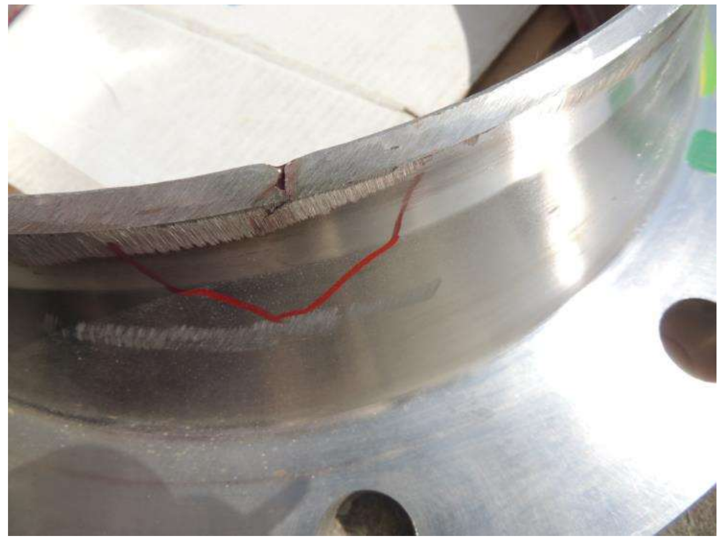


Figure 2: Photograph showing crack in the bevel of the flange.

Figure 3: DPT of the as received flange on the inner surface.

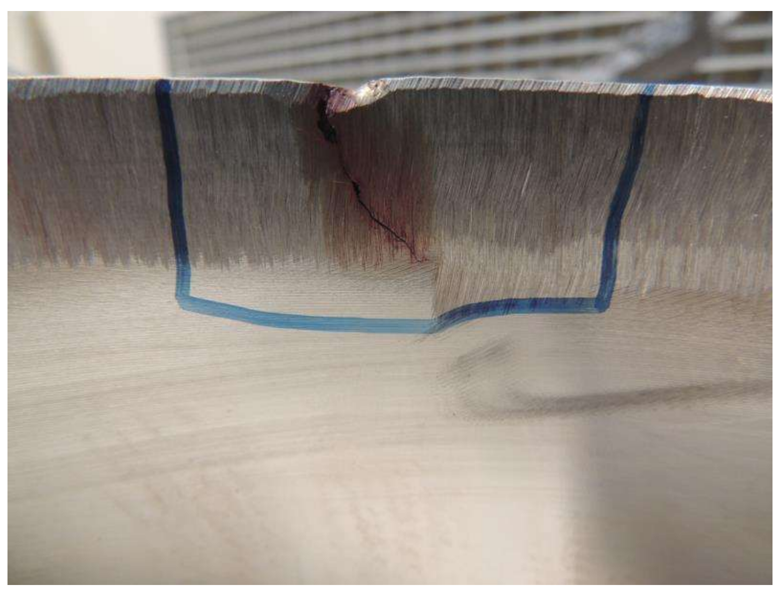


Figure 4: No other defects were observed other than the visible crack.

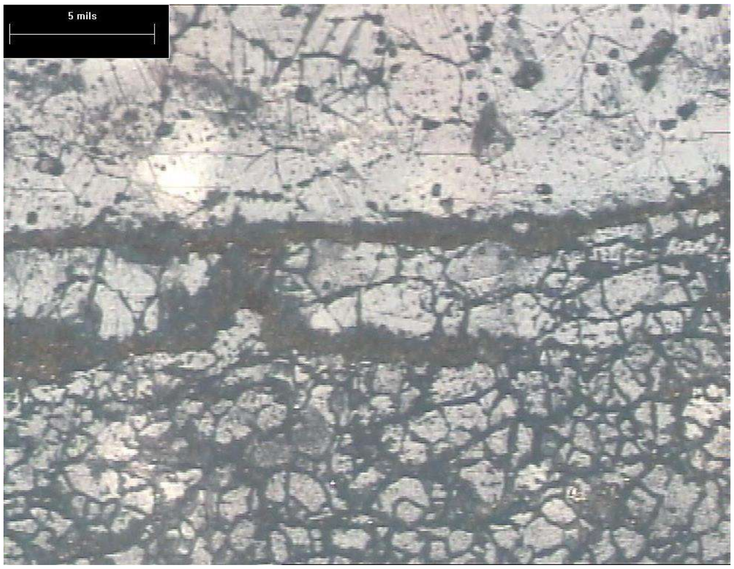


Figure 5: Photomicrograph showing cracks, distributed sigma islets and network of highly sensitized grains. Magnification: 100X

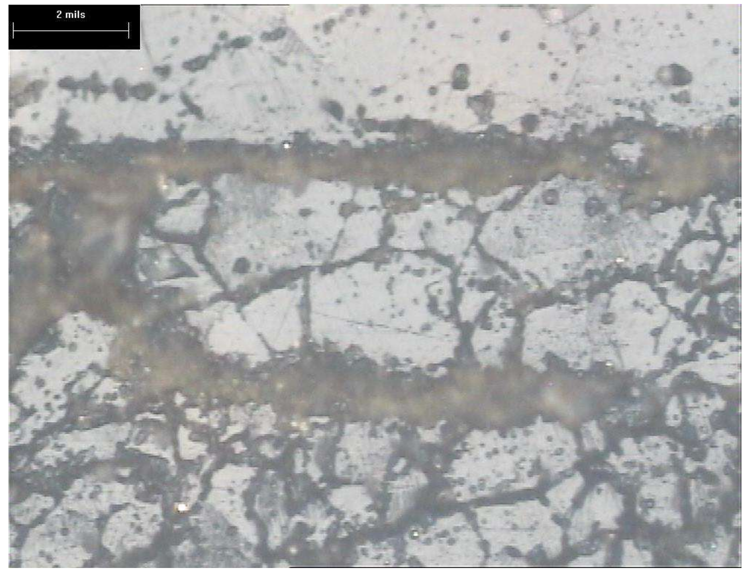


Figure 6: Closer view of Figure 5. Magnification: 200X

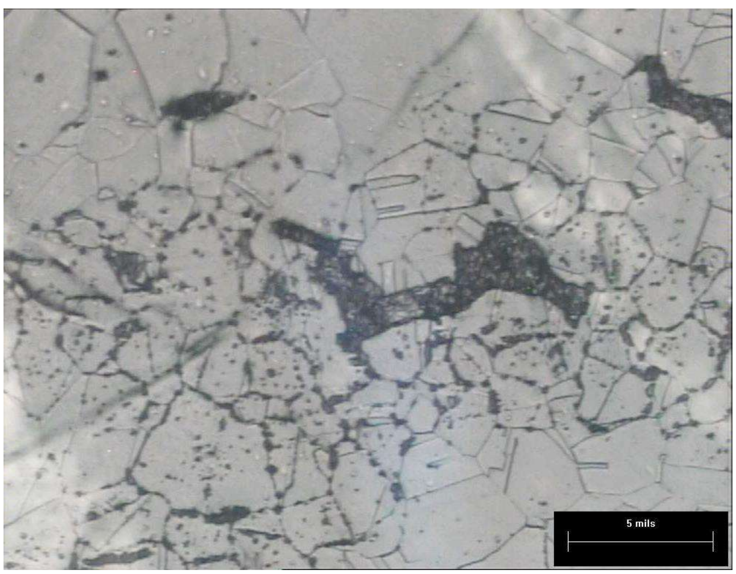


Figure 7: Photomicrograph showing equiaxed austenite grains with presence of sigma phase along grain boundaries. Magnification: 100X

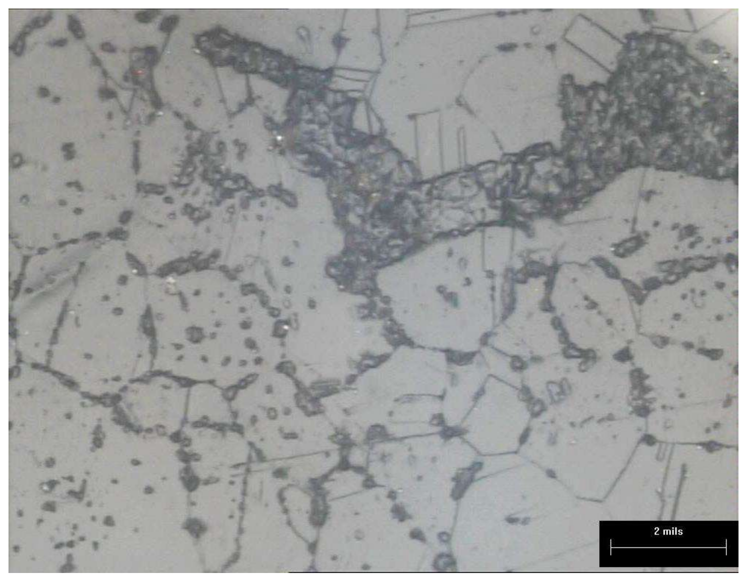


Figure 8: Closer view of Figure 7. Magnification: 200X

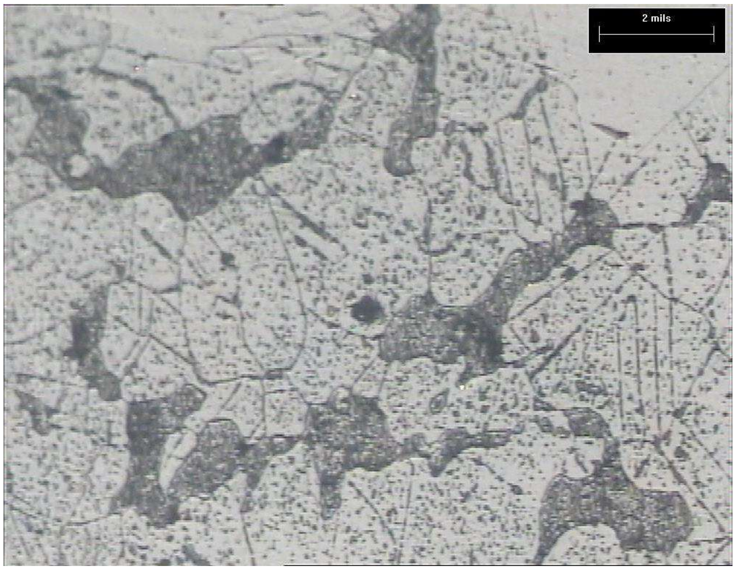


Figure 9: Photomicrograph showing equiaxed austenite grains with presence of sigma phase along grain boundaries. Magnification: 200X

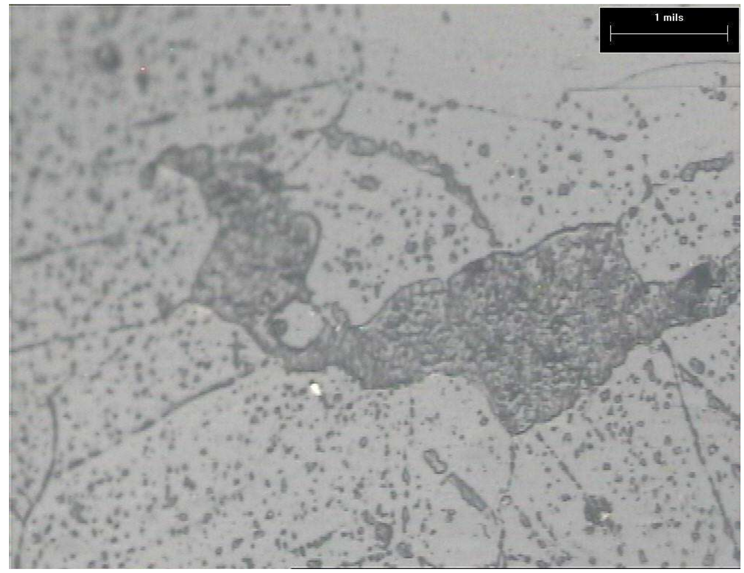


Figure 10: Closer view of Figure 9. Magnification: 500X

Figure 11: Photomicrograph showing equiaxed austenite grains with presence of sigma phase along grain boundaries. Magnification: 100X

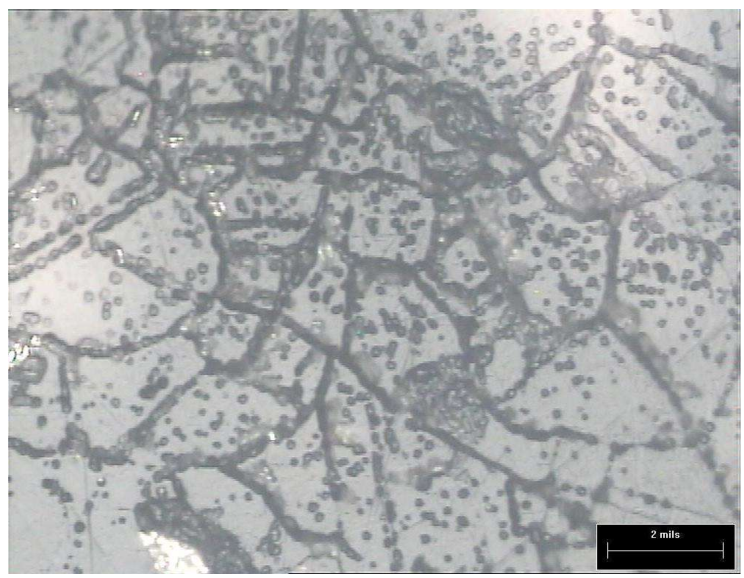


Figure 12: Closer view of Figure 11. Magnification: 200X

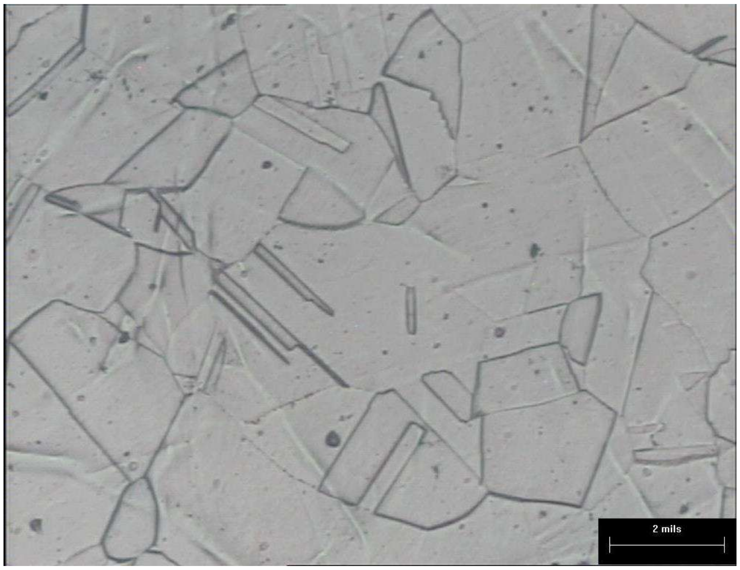


Figure 13: Photomicrograph showing equiaxed austenite grains. Magnification: 200X

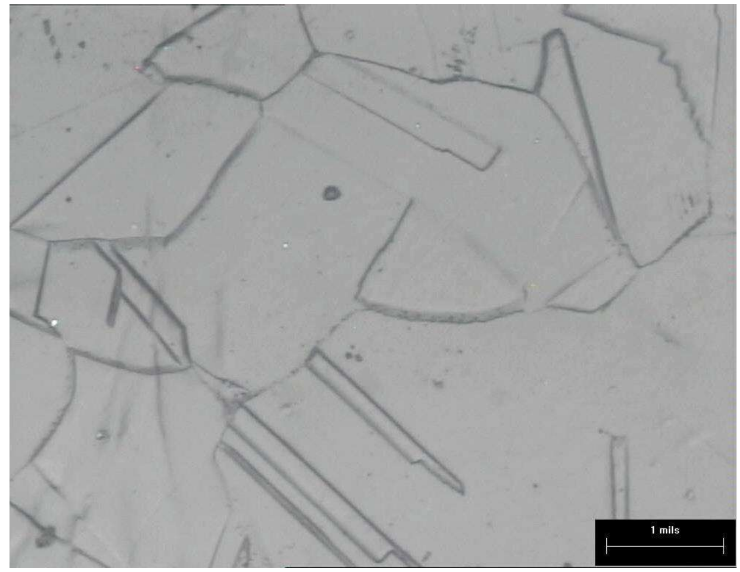


Figure 14: Closer view of Figure 13. Magnification: 500X

Figure 15: Optical micrograph shows presence of sigma phases indicated by arrows. Magnification: 50X. Electrolytic Etched with Marble's reagent for 10 secs.

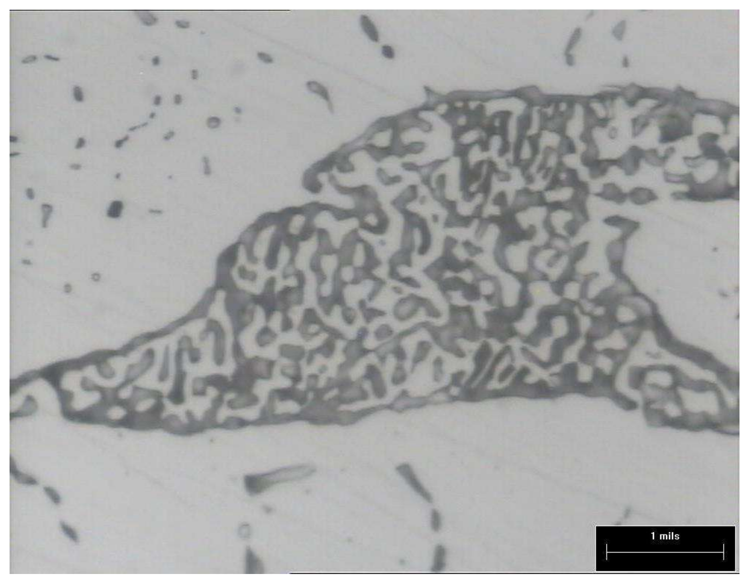


Figure 16: Closer view of the sigma phase shown in Figure 15. Magnification: 500X. Electrolytic Etched with Marble's reagent for 10 secs.

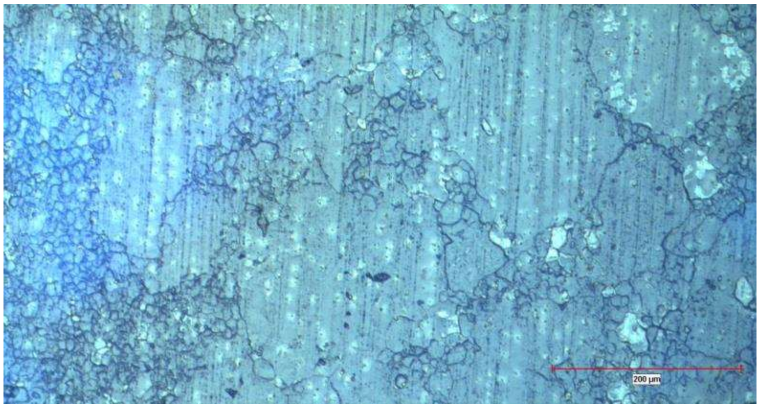


Figure 17: The microstructure is mostly decarburized. Grain coarsening can be clearly seen. Magnification: 200X

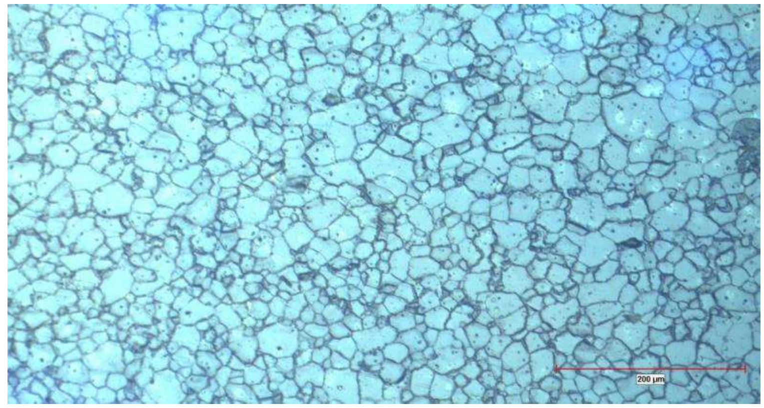


Figure 18: The microstructure is mostly decarburized. No evidence of pearlite in the examined area. Magnification: 200X

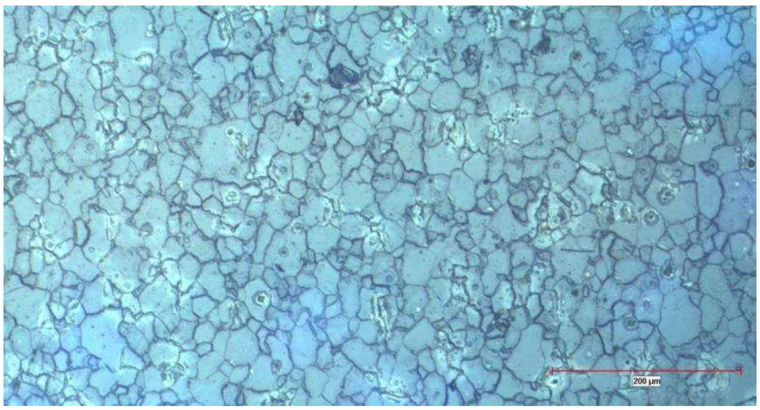


Figure 19: The microstructure is mostly decarburized. No evidence of pearlite in the examined area. Magnification: 200X

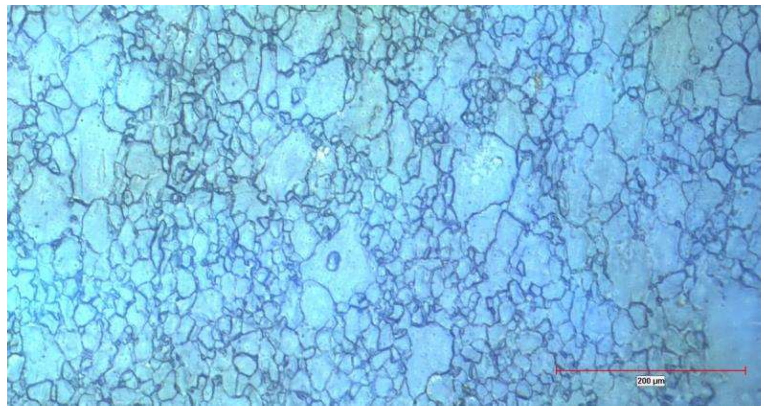


Figure 20: The microstructure is mostly decarburized. Grain coarsening can be clearly seen. Magnification: 200X

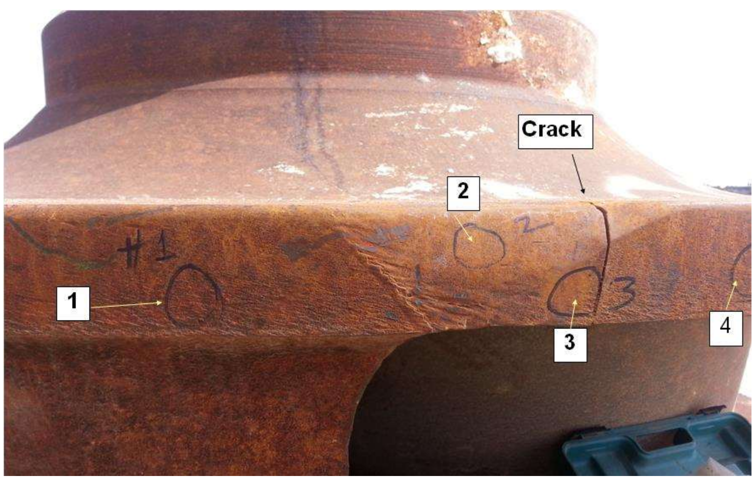


Figure 21: Photograph of the impeller.

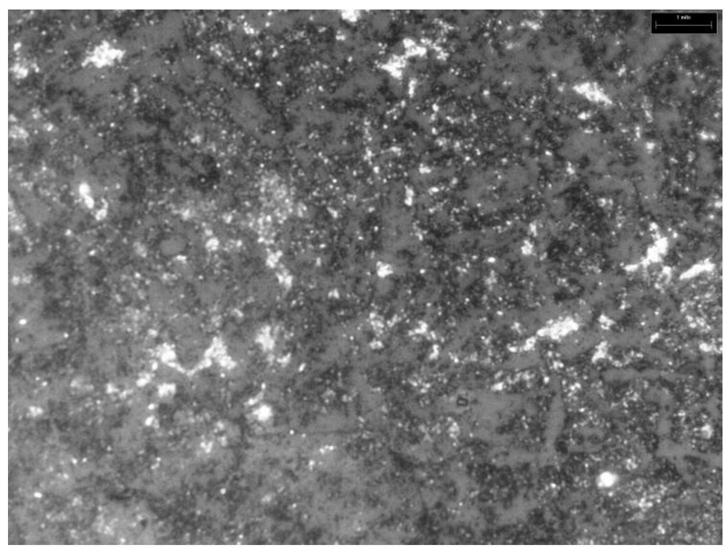


Figure 22: Replica taken at location 1 shows tempered martensite (dark phase) and retained austenite (bright spots). Magnification: 500X

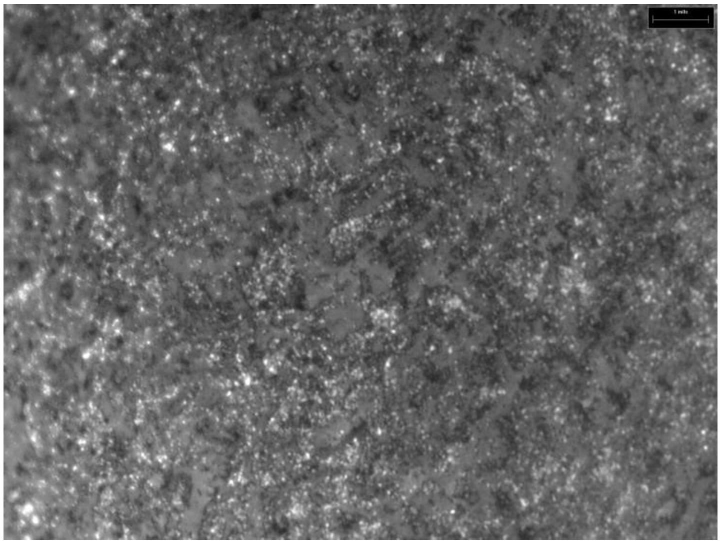


Figure 23: Replica taken at location 4 shows tempered martensite (dark phase) and retained austenite (bright spots). Magnification: 500X

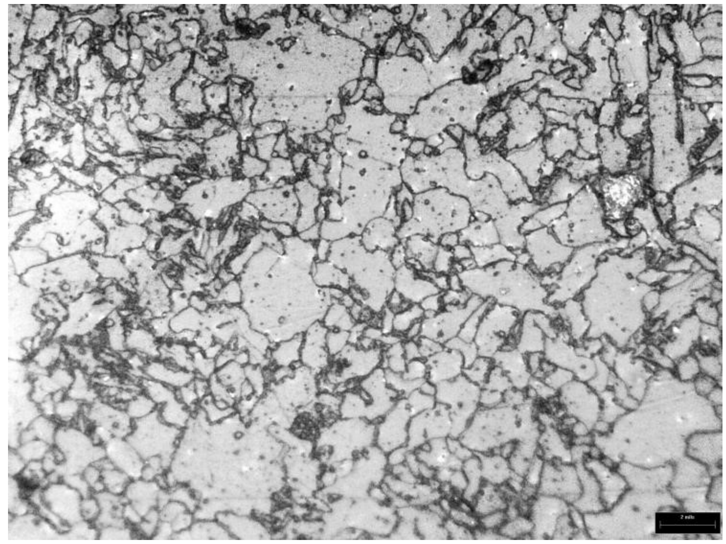


Figure 24: Replica taken at location 2 shows fine grains of ferrite and pearlite. Magnification: 200X

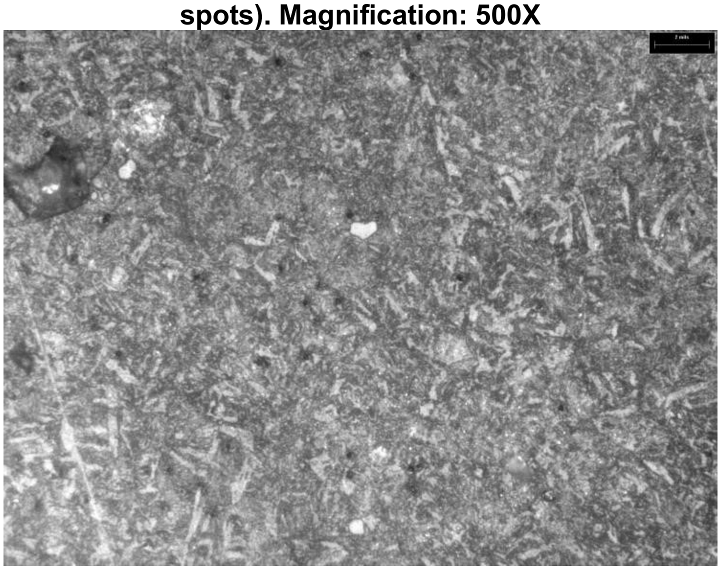


Figure 25: Replica taken at location 3 shows lower bainitic structure. Magnification: 200X

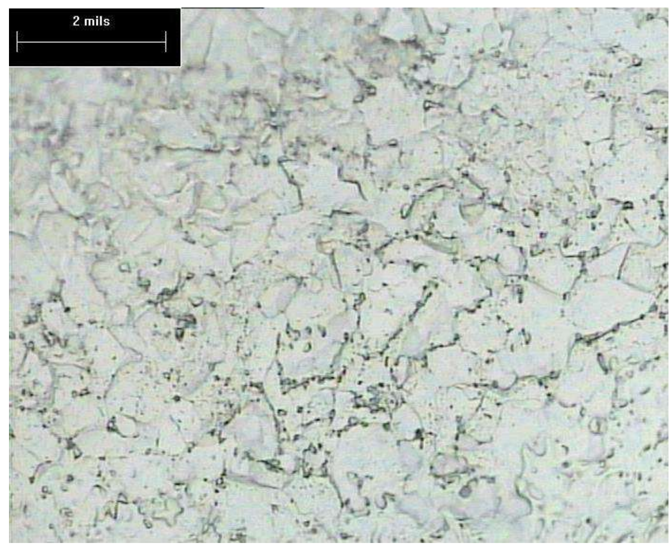
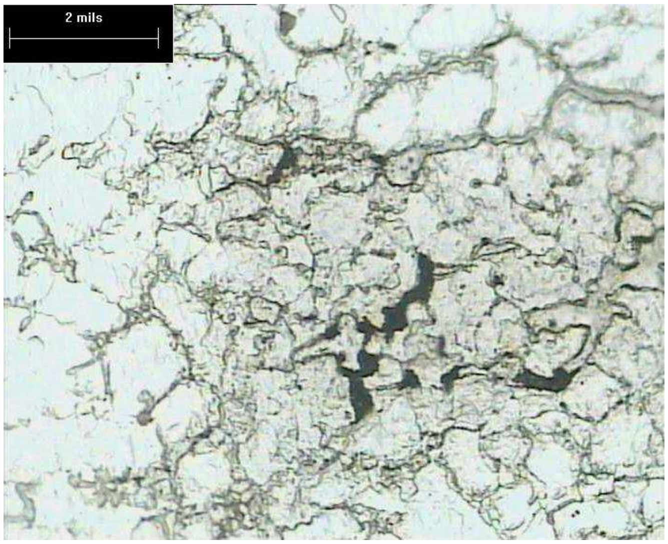


Figure 26: Photomicrograph showing coalesced grains of coarse ferrite with fine dispersed carbides. Magnification: 200X



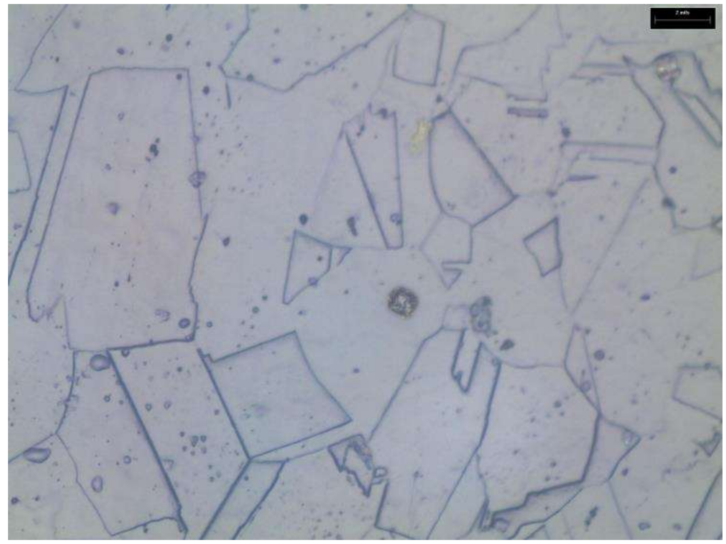
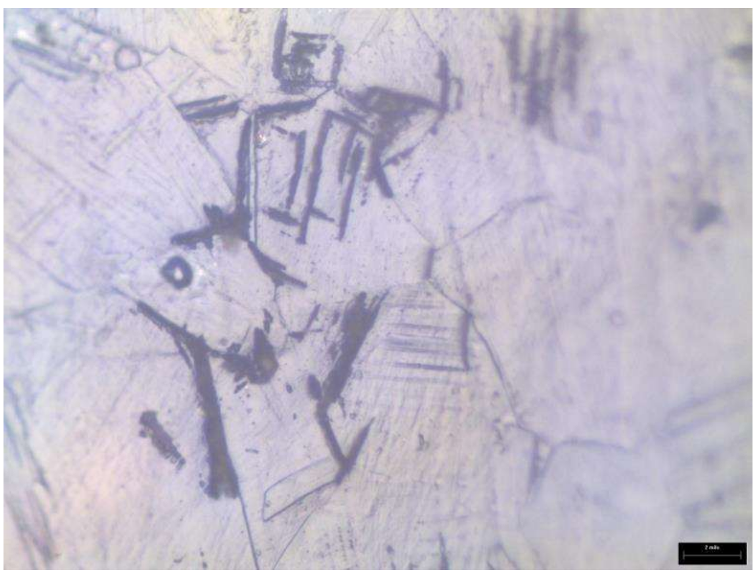


Figure 27: Photomicrograph showing coarse ferrite with network of heavy grain boundary carbide precipitation. Voids (dark spots) are visible at some areas. Magnification: 200X

Figure 28: Photomicrograph showing equiaxed austenitic structure with evidences of annealing twins. No microstructural anomalies were observed in the examined area. Magnification: 200X



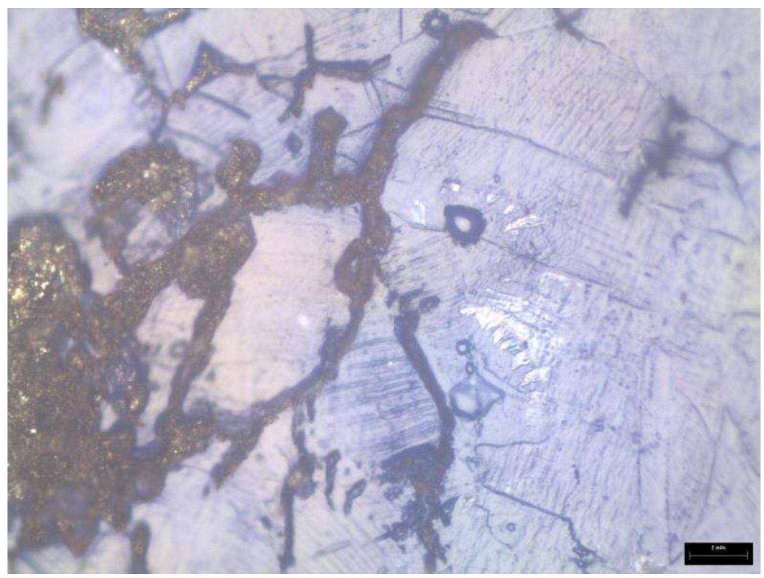


Figure 29: Photomicrograph showing cracks in the examined area. Interestingly, the cracks were confined only to the regions with prior cold worked grains. The cracks were similar to stress relaxation cracking. Magnification: 200X

Figure 30: Photomicrograph showing cracks in the examined area. Interestingly, the cracks were confined only to the regions with prior cold worked grains. The cracks were similar to stress relaxation cracking. Magnification: 200X

Figure 31: Photograph of the as received failed regeneration heater coil or affected sample which had a crack.

Figure 32: Photomicrograph showing intergranular crack and variation in the grain sizes is also clearly evident. Isolated voids (yellow arrows) were also seen. Austenite grains with strain lines were also seen at the outer surface. Magnification: 50X

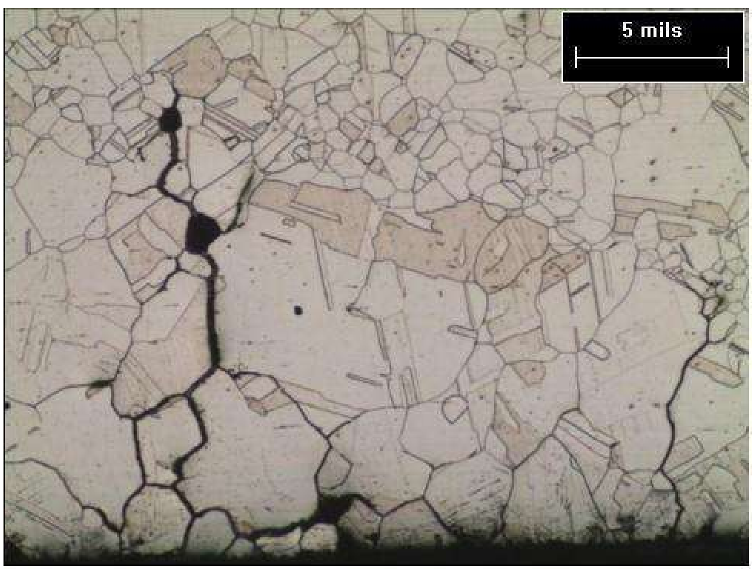


Figure 33: Photomicrograph showing intergranular cracks and coarse grains at the outer surface in another location. Magnification: 50X

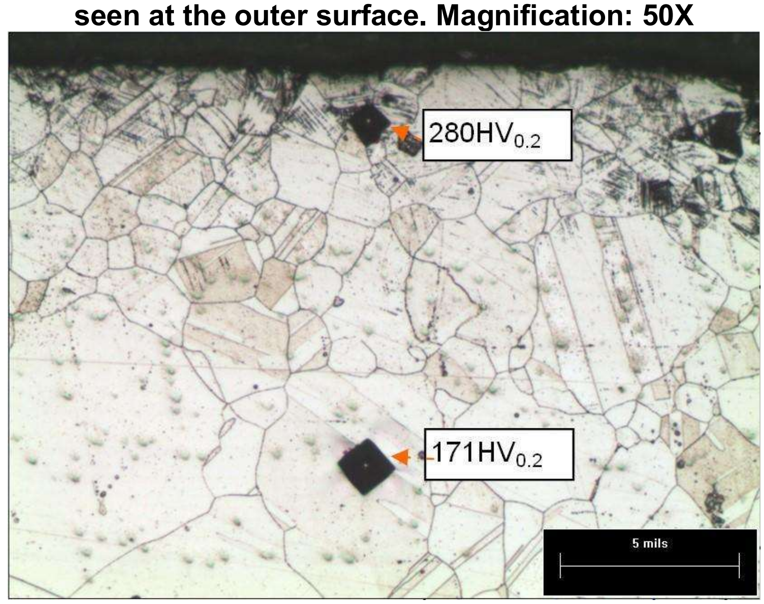


Figure 34: Photomicrograph taken at the OD in the vicinity of the crack showing hardness value above 200 $HV_{0.2}$. Magnification: 100X