# Application of metallography in failure investigations – some case studies

# A. N. SINHA

National Metallurgical Laboratory, Jamshedpur - 831 007, India

#### ABSTRACT

Metallography is a tool to know the internal condition of the material and no investigation can be initiated without this. It can be both nondestructive i.e., in-situ metallography, as well as destructive i.e., cutting the relevant parts from the site to undertake investigation in the laboratory. In both the cases grinding, polishing and etching to reveal the microstructures remain the same. The only difference is the special tools and portable optical microscopes are used in the in-situ techniques to obtain replicas for laboratory use. Two types of etching are used to reveal macro and microstructures. The macro structures are viewed at 5 to 10 x, which reveal flow line, cold rolling fibres, welding zones, pores, dendrites, coarse grains etc. whereas microstructures reveal the micro constituents, pearlite, martensite, inclusions, precipitates, carbides, creep voids etc. at magnification of 50 to 1500 x. Metallography alone gives lot of information regarding the deviations of microstructures which are indication of the causes of failures. Some typical case studies are described in the present paper where metallography plays very important role, like : (i) Improper weld deposits to salvage the undersize rail axle, which led to premature failure, (ii) improper welding joints and lack of post weld heat treatment, which gave stress on the joint of cover dome of pressure vessel tank, which caused failure, (iii) revealing of tool marking and sulphide inclusions in the steel isolation valve of LPG storage tank, which led to premature failure, (iv) sensitization and stress corrosion cracking in S.S. cover dome of centrifuge used in the manufacture of NG explosive, which caused premature failure, (v) Improper heat treatment and forging were indicated by the microstructures which led to the premature failure of mining dragline sleeve. These are only a few investigations carried out in the laboratory in the recent past which justify that without metallography it is not possible to diagonise the causes of premature failures.

#### INTRODUCTION

Five prematurely failed samples from different areas like Railway, Petroleum Industry, Explosive Industry and Mining fields were received at NML for investigation. Normally the failed samples are visually examined and all the features that are worth recording are noted. The sites showing any abnormality are cut with hacksaw for metallographic study. The sizes are variable. If welding parts are involved, then the size could be large for example in the case of rail axle the sample was 17 cm dia axle plate. This was essential as the whole axle transverse section was helpful in knowing the depth of welding as found to be on etching. Similarly in the case of pressure vessel cover dom, it required bigger joint portion to reveal the weld defects.

However in the case of brittle failed portion of dragline sleeve and a portion of rail axle, the portions were seen in the SEM. By virtue of its depth of focus, the SEM has got unique advantage of over optical microscope to study the fractured surface in the as received condition.

The tool marking on the failed isolation value of the LPG storage tank was yet another example of utilisation of SEM for such investigation. Additionally, the application of EDAX in finding out the nature of inclusions (elemental analysis) gives a better understanding of the causes of failure.

SEM has enhanced the quality of microstructural studies so far done by optical microscopy. Sensitisation and grain boundary studies in S.S. centrifuge was possible only under SEM/EDAX. Chemical analysis of the samples are also carried out to verifywhether the materials are conforming to the specification.

Physical characterisations like hardness measurement, tensile test, impact test are also carried out to confirm the desired mechanical properties expected as per specification.

In some cases TEM investigations are also carried out to known the phases of the precipitates/carbides etc. so that a better meaningful reasoning can be arrived at in the end of various studies of the failed samples.

#### **CASE STUDIES**

## FAILURE OF A LOCOMOTIVE AXLE

The axle failed after having been in use for only three months  $^{[1,2,3]}$ . The axle failed near the journal by fracturing transversely. The surface that failed was close to one of the wheels about 45 to 55 mm from the inner wheel seat collar (Fig. 1). The axle had been proof machined and assembled in the carriage after it had been



Fig. 1 : Sketch showing location of fracture of the rail axle

ultrasonically tested. The chemical composition corresponded to the stipulated ASTM A 21-78 specification as given in Table - 1.

Tuble 1	. Chemical composition	(111. 70)
С		0.52
Si	-	0.23
Mn	· .	0.63
S	-	0.014
Р		0.02

Table 1 : Chemical composition (Wt. %)

The failed ends of the axle were examined visually and the longer side wa found to be badly mutilated. The other end which was shorted, showed signs o fatigue failure (Fig. 2). The macro investigation revealed that weld metal was de posited all around the circumference of the axle. This was apparently done to builup an undersize area, which is not a recommended practise. Further investigatio



Fig. 2 : Macrograph view of failed axle showing signs of fatigue failure x 0.75

supported that contention and showed distinct wavy patterns of weld pool solidification, fatigue striations and hairline cracks around the cricumference of the axle. The depth of the weld was found to vary from 5 to 9 mm. It was concluded that the failure was due to fatigue and cracks were initiated by welding discontinuities.



Fig. 3 : Microstructure of base metal of the axle away from failed parts showing normalised ferrite pearlite structure x 400



Fig. 4 : Widmanstatten ferrite structure in the welded zone x400



Fig. 5 : Presence of acicular structure close to the surface and weld deposit x 400

Microstructural study of the base metal and weld zone revealed (Fig. 3, 4, 5) initiation and propagation of the fine cracks at the edge of the surface. The base metal showed ferrite pearlite structure, whereas weld samples showed paths of fatigue cracking, Widmanstatten ferrite and accicular structures. These are typical cast structures with coarseness of the prior austenite grains. These structures are harmful and can generate stress that could initiate and propagate fatigue cracks. This stress was confirmed by X-ray measurement. Hydrogen content and mechanical properties support the above conclusion, which were evaluated and are shown in Table - 2

	Table 2. Mechanical properties						
W.	UTS	EL %	RA%	Hardness	Hydro		

	UTS	EL %	RA%	Hardness	Hydrogen content
Base metal	54 kg/mm <sup>2</sup>	30	50	180	0.84–0.86 ppm
Weld	dolg it <mark>-</mark> aonaais	ng s <u>a</u> bos	róde <u>-</u> dqs	330	2.6-3.5
					the load to take the proves of a state of

This brief metallographic investigation shows that the premature failure of the locomotive axle occurred because of the use of welding as a means of salvaging an undersized axle.

# FAILURE OF PRESSURISED STORAGE TANK

Various storage tanks are used by petroleum industries for storing crude and its fractions for final products. They are of different sizes and have dome shaped fixed roofs. These roofs are welded in segments and strengthened to withstand higher than atmospheric pressures in many cases. In case if the welding is not properly done, some voids and fissures along with microstructural anisotropy can persist. Particularly damaging is hydrogen pickup during welding. Since these tanks go through fluctuating pressures during filling in and releasing of the stored product, chances of low cycle fatigue during service are probable. This can result in the coalescence and progress of the voids and fissures When criticality of crack size is reached an unstable and cataclysmic growth takes place and failures of the component takes place.

In the present case <sup>[4]</sup> a pressurised storage tank handling naptha fraction (60–90°C) as the feed for toluene and benzene separation unit failed. Extensive microstructural and mechanical properties indicated defects in welding, hydrogen pick up and also build up of cyclic stress. The failure had taken place all along the circumferential weld joints. Chemical composition of the material is given in Table 3.

Tank No.	С	<b>S</b> <sup>1</sup>	Р	Cu
Failed tank	0.18	0.025	0.03	0.0011
Another tank	0.23	0.023	0.02	0.0076

Table 3 : Chemical composition (Wt. %)

The composition roughly corresponds to BS-15 and also to ASTM 516.

#### **Microstrucal Study**

Macrostructural photographs showed brittle fracture (Fig. 6) which is possible in ductile material only when fluctuating pressure develops. Optical microstructures of the base material indicated ferrite pearlite and banded structure as is normally found in rolled products. However welded zone microstructures showed great variation and defects like fissures Widmanstatten.. structures and voids (Fing. 7). SEM study of weld pool and HAZ showed voids along the grain boundary and transgranular cracks. TEM photographs showed the presence of globular inclusions, which was not desirable.





Fig. 6 : Fractured surface of the roof dome joint showing brittle fracture x 0.3

Fig. 7 : Microstructure of welded regions showing fissures x 160

## Hydrogen content

The base material showed the hydrogen content to be about 0.30 ppm, but near the weld joint it was greater then 2 ppm. This too was not desirable

## **Mechanical Properties**

The hardness was found to be 145 to 150 VPN at 30 kg load whereas it varied from 200 VPN to 220 VPN in welded zone. This naturally generated stress in the welded portion, which was not desirable. Grain size and mechanical properties are given in Tabel - 4

Tank No.	Grain size	Hardne	ss (VPN)	Tensile	RA%	EL%
	(ASTM)	Base	Weld	MPa		1
Failed tank	g adl 894003	150	220	490	59	27
Another tank	8	148	220	512	58	35

# Table 4 : Mechanical properties

## Remarks

The base material although appeared to be sound, while defects were introduced during welding, either because of shrinkage or because of higher hydrogen pickup <sup>[5,6,7]</sup>. The flux impurities, fissures, voids in HAZ and weld pool were identified by metallographic technique which justifies its importance in failure investigation

# FAILURE OF ISOLATION VALVE OF LPG STORAGE TANK

Another example of the utility of metallography is shown in the investigation of failure of an isolation valve fitted to a LPG storage tank. The analysis carried out included chemical composition, metallography, fractography and fracture toughness. This case study showed that the normal defects which would pass through

NDT examination, during quality control, could combine to form cracks leading t catastrophic failure, during service.

The chemical composition of the material were comparable to the specified star dards as shown in Table 5.

Element	Valve	ASTM-A216A(3)	Bolt	AISI4140(3a)
C	0.10	0.1-0.25	0.44	0.38-0.43
Si	0.37	0.6 max	0.19	0.15-0.30
Mn	0.77	0.7-1.2	0.82	0.75-1.10
Cr	0.21	0.5 max	0.92	0.8-1.1
Mo	0.13	0.2	0.18	0.15-0.25
S	0.02	0.045 max	0.012	0.040 max

Table 5 : Chemical composition (Wt. %)

Even though the mechanical properties like, hardness, impact, tensile etc. cor formed to the specifications, the material failed because of some other reasons.

The visual examination showed machining defects near the bottom neck of th valve (Fig. 8), where failure took place with brittle fracture. The brittle fracture i a ductile material as concluded from the mechanical properties and chemical analy sis indicated some fatigue failure. The ferrite pearlite structure of the valve materia and tempered martensite of bolt material were as expected. The  $D_3$  oxide type inclu sion was slightly on the higher side for the valve material.

The SEM study of the fractured surface (Fig. 9) showed the presence of high (0.11%) along with Ca, whereas the S content of the base material away from frac ture was 0.02% only. This high S in the fracture was responsible for the brittlenes



Fig. 8 : Upper portion of the valve showing machining defect x 0.65



Fig. 9 : SEM of valve material showing brittle fracture and presence of high 'S' & 'Ca'

of the material <sup>[9]</sup>. Sulfides are known to inhibit cathodic reaction and thus its presence can cause a build up of absorbed hydrogen in to the metal, which may increase the tendency to cracking. A defect, like the machined notch acts like a crevice, where the volatile S ingress into the material, enhances the local stress intensity and thus initiate cracking. A combination of material defect (inclusion), machining defect (tool mark) and operating environment (volatile sulphur) each of which individually cannot cause a failure of this nature, occurring around the same region has led to the catastrophic failure of the isolation valve <sup>[10,11]</sup>.

Reasons were confirmed by using extensively SEM/EDAX and optical metallography that the isolation valve did not fail due to material used, but because of other factors described above.

# FAILURE OF S.S. CENTRIFUGE

Austenitic stainless steels of AISI 300 series are having excellent corrosion resistance. It can handle concentrated oxidising acids without any adverse effect. However, while making containers or equipments cold drawn rolled products are needed. With higher deformation due to cold drawing, a part of the austenite is transformed to  $\alpha'$  martensite <sup>[12,13]</sup>. This may lead to stress corrosion cracking.

Another factor is that if the equipment is heated to ~ 650°C as heat treatment or some other needs like decontamination, then sensitisation takes place and chromium carbide migrate to grain boundaries. This makes the matrix less corrosion resistance and also the chromium carbide dissolves in acids and grains can disintegrate and S.S. sheet can crumble to pieces. This could happen near the weld zone also. The explosive companies use this material S.S. Centrifuge for the separation of liquid nitroglycerine (NG) from nitrating acid, where  $H_2SO_4$  is a catalyst. Because of some contamination due to HCl acid or chloride ions, the corrosion starts.



Fig. 10: (a) Corrosion/erosion of cover dome of the centrifuge, (b) corrosion/erosion of top frame x0.50

In the present study <sup>[14]</sup> one such failure took place, where optical metallography and SEM/EDAX were found to be useful in the conclusion of the above two factors.



Fig. 11 : Optical metallographs showing (a) thick grain boundary x80 (b) twinning x160 and (c) delta ferrite x160

- i) Macro photographs are showing the badly corroded surface of the centrifuge (Fig. 10a,b), which indicated the intensity of corrosion.
- ii) Chemical analysis confirmed that the material was 304 AISI Stainless steel
- iii) Optical metallography : Optical microscopy showed thick grain boundaries and twinning indicating the austenitic structure with sensitisation. Some parts showed delta ferrite islands which can not exist in 304 S.S. (Fig. 11a,b,c).
- iv) SEM Study:- Cover dome of the centrifuge indicated intergranular corrosion and porosity in grains. White particles are dye penetrant (Fig. 12 a,b).



Fig. 12 : SEM photographs of cover dome showing (a) intergrannular corrosion and (b) porosity in grains



Fig. 13 : Welded part showing (a) pores x 40 and (b) ferritic structures x 160

v) Metallography of Welded part : Weld zone parts showed pores and ferritic structure. This also was not desirable (Fig. 13 a,b).

To conclude

- i) improper material was chosen,
- ii) welding was not done with care.

To avoid sensitization and  $\alpha'$  martensite formation 316L S.S. has been recommended. Also welding has to be done with proper welding rod and post weld treatment was recommended.

In this investigation also, metallography played important role in arriving at meaningful conclusion.

## FAILURE OF WALK SHAFT SLEEVE OF DRAGLINE

In mining to remove over burden, a dragline of different bucket capacity (25 cubic meter) are being used. Walk shaft sleeve is a static component made of cast forged steel and is a part of the structural assembly. The sleeve is a heavy steel casting of about seven tons. Proper casting, heat treatment and press forging are



Fig. 14 : Macro photo of failed part indicating brittle fracture x 1.1

needed to obtain suitable microstructures/grains to provide a fracture resistant material.

The present investigation is a case history of prematurely failed sleeve <sup>[15]</sup>. The chemical composition and mechanical properties were found to be as per specification. The chemical composition is shown in Table 6.

Element		Wt.%
C .		0.20
Mn		1.80
Si	-	0.32
Ni	-	0.32
Cr		0.35
Р	_	0.006
S	_	0.03

Table 6 : Chemical compostion

This composition is similar to ASTM A487 class 2 steel which is recommended for excavating equipment <sup>[16,17]</sup>.

*Mechanical Properties*: They were measured in Instron Universal testing machine and Vicker's Hardness measuring machine, and the results are given in Tabel 7.

Hardness		200 VPN at 30 kg load
UTS	-	57 kg/mm <sup>2</sup>
EL%	-	46%
02% PS	-	45 kg/mm <sup>2</sup>

Table 7 : Mechanical properties

The macro photo showed typical brittle fracture, which was not expected from such ductile material (Fig. 14).

The microstructure of the failed sample, polished and nital etched shows tempered fine bainitic cast structures indicating that cast structure has not been fully removed and proper heat treatment was still needed to remove the cast structure (Fig. 15a,b).

The ASTM grain size was found to be no 3 indicating rather coarse grain, not desirable for such equipment.



Fig. 15 : Microstructure of failed sample showing tempered fine bainite and cast structures at (a) x200 and (b) x500

SEM study of fractograph of the sleeve showed that the material was not killed properly (Fig. 16). It shows the presence of coagulated iron oxide in the globular form indicating insufficient deoxidation which may also be responsible for poor resistance to fracture. EDAX was used to confirm that it was only iron oxide and no other inclusion existed.



Fig. 16 : SEM photo showing presence of coagulated iron oxide in the golubular form

## CONCLUSION

In the present paper, five case studies have been presented. In all the cases we observe that optical microscopy is essential for any failure investigation. The inclusion rating, grain size, cast structure, rolled structure, carburisation and its depth welding quality and so many other things can be studied and reasons concluded towards the desirability of the material for various components. The additional use of SEM/EDAX can further add to the authenticity of the conclusions. Particularly fractured surface can be studied by SEM/EDAX alone. Chemical and mechanical properties evaluation are to be done to support the investigation.

#### ACKNOWLEDGEMENT

The author wishes to thanks Prof. P. Ramachandra Rao, Director, NML for per mission to publish this paper.

#### REFERENCES

- A. N. Sinha, B. N. Halder and O. N. Mohanty, "Failure of a locomotive axle A metallo graphic investigation", Welding Journal, Vol. 70, No. 4, 1991, pp. 59-61.
- [2] G. F. Vander Voort, "Failure analysis and prevention", ASM Metals handbook, Vol. 11 1986, pp. 715-727.
- [3] Jai-Yong Lee and Jong Lam Lee, "Philosophical Magazine A", 56(3), 1987, pp. 292 309.
- [4] A. N. Sinha, N. Parida, S. Das, and O. N. Mohanty, "Low cycle fatigue failure of pressurised storage tank", NML Technical Journal, April-June, Vol. 36, No. 2, 1994 pp. 65-81.
- [5] R. E. Dolby, "Advances in welding metallurgy of steels", Metals Technology, Vol. 10 September, 1993, pp. 349.
- [6] R. G. Baker, et. al. "Weldability of structural and pressures vessel steels", The Weldin Institute, Cambridge, 1970.
- [7] "Low cycle fatigue and life prediction", ASTM-STP 770, Philadelphia, ed. Amzallag et. al. 1982.
- [8] A. N. Sinha, V. R. Ranganath, N. K. Das and O. N. Mohanty, "Failure analysis of a isolation valve of a LPG storage tank", NML Technical Journal, July-Sept. Vol. 36, No. 3, 1994, pp. 83-94.
- [9] J. G. Parker, "The effect of sulphide inclusions upon the stress corrosion behaviour of steels", ASM proceeding Intl. Symposium, 'Sulphide Inclusions in Steels', 1974, pp 141.
- [10] J. M. Barson and S.T. Rolfe, "Fatigue and fracture prevention and control in struc tures", Prentice-Hall, U.S.A., 1987.
- [11] J. F. Knott, "Fundamentals of fracture mechanics", Butterworts, London, 1973.
- [12] D. S. Sharma, "Physical metallurgy of stainless steel", Tool s Alloy Steel, Decembe 1985, pp. 405.
- [13] C. J. Novak, "Handbook of stainless steels", Eds. D. Peokner and I.M. Bernstein, McGrav Hill, 1979, pp. 4.
- [14] A. N. SINHA and I. Singh, "Failure of S.S. centrifuge due to deformation induce martensite and sensitization", Metals Materials and Processes, Vol. 7, No. 3, 1995, pp 175-184.
- [15] A. N. Sinha and R. N. Ghosh, "Failure investigation of walk shaft sleeve of dragline Presented at Seminar on Cost Effectiveness in Foundry, 22-23, Nov. 1996, I.I.J Jamshedpur Chapter.
- [16] "Low alloy steel casting", Metals handbook, Vol. 9, pp. 231.
- [17] "Properties and selection of iron and steel" Metal Handbook, Vol. 1, pp. 386.