MICROSTRUCTURAL STUDIES IN FAILED SAMPLES

P. Prasad & R. Singh
National Metallurgical Laboratory
Jamshedpur 831007

What is Microstructure ?

Metallic specimen consists of very fine constituents which cannot be seen through naked eyes. These constituents can be seen only at higher magnification say 25X and above. Macrostructures in contrasts, are those features which can be seen through naked eyes. In order to see microstructure, a metallic specimen undergoes a surface preparation consisting of polishing (mirror finish) followed by etching in suitable reagent .

The microstructure often deals with:

i) grain size and shape
ii) inclusion content
iii) Phases like ferrite or a mixture of two phases i.e. ferrite and cementite as in pearlite.
iv) distribution of precipitates like carbides and nitrides
v) dislocation structure

Some of the precipitates and dislocation structures require very high magnification and hence can be studied only by using transmission electron microscope (TEM). Scanning Electron Microscope (SEM) can also be used for studying some fine precipitates. In this lecture only microstructural features studied with optical microscope will be dealt in.

Microstructural Examination

Before we start microscopic examination of a sample, removal from an engineering component whether failed or unfailed, we require to know the following:

i) Material specifications including composition; some broad guideline are material delivery condition like heat treatment, hardness, tensile strength and other relevant properties.

ii) Service Conditions : This should be in terms of operating stress and temperature, environment (open air, gaseous etc.) in which it is working.

iii) Length of service rendered along with fluctuation in the service condition, e.g. occasional increase in temperature/ pressure.

These are illustrative and may not be exhaustive. The main purpose of these information is that one must know before hand expected starting structure and structure after failure.

Grain Size Structure

Metallic materials are invariably polycrystalline i.e. they consist of a large number of grains which are nearly equal in shape and sizes. The size and shape of the grains may vary depending on the prior history of the specimen, i.e. the treatment offered to the metal and alloy. Fig. 1 shows sketch of different ASTM grain sizes. In an engineering component, generally grain size number 5-6 is encountered. Grain size No 8 is considered to be very fine grain and grain size number 1 to 3 are coarse. Fig. 2 shows relationship between temperature and strength. The figure shows that the strength of grain boundaries decrease rather more sharply than that of the grain and there is a temperature called equiquasive temperature where both grain boundaries and grain have equal strength. The failure will be inter granular above equicohesive temperature where as the fracture is trans-granular below equicohesive temperature.

The grain size may become coarser if operating temperature is rather high. In case of steel grain size coarsening can also occur, if temperature goes to around 900°C.

Ductile and Brittle Failures

Two broad categories of failures are (i) ductile and (ii) brittle. Features associated with these two failures are illustrated in fig. 3. Fibrous zone of the ductile failure can be seen in the fig 3a where as fig 3b shows large cleavage facets of brittle failure. It may be seen that in ductile failure, a good amount of plastic deformation takes place where as in brittle failure, the deformation is not appreciable. When deformation takes place, grain changes from equiaxed to elongated ones. In annealed samples, the grains are generally equiaxed. However, if the sample is in the cold worked condition, grains may remain elongated even before failure. Another way to distinguish ductile and brittle fracture is to see whether the fracture is of cup & cone type and separation has occurred along a plain without any evidence of necking. or not ? In fig. 4 cup and cone type fracture shows initiation of crack at the center of in a ductile fracture. Brittle failure in metallic material is indeed a cause of concern. One of the example of brittle failure and associated features are dealt with in fig. 5.

Creep Failure

Creep is a slow plastic deformation of material at elevated temperatures under sustained stress. Creep failure is said to have occurred either when deformation exceeds 1-2% or when a component has broken into pieces. In case of steel, creep generally occur above 450°C and failure of high temperature components should occur after the design life which is about 10 years in case of thermal power plant equipment. To illustrate microstructural studies some examples of tube failures are discussed below:

Fig.6 shows a failed water wall tube due to overheating. For microstructural examination samples are taken from several locations e.g. from rupture lip, diametrically opposite to the rupture zone and far away from the rupture zone. Several samples must be studied because the microstructure may vary along the length of the tube. Under the figure microstructures are given with their descriptions. The elongated grains may be noted, which indicates that the failure is essentially of ductile type. This type of failure is also called fish-mouth opening or thin wall lip rupture.

Fig. 7 shows another creep failure of a boiler tube, associated microstructure is also shown. It may be noted here that the failure is of brittle type i.e. thick wall rupture. In the microstructure, one can see creep cavities at the grain boundaries which is the main cause of the brittle failure. The microstructure has also spheroidised due to long term exposure at elevated temperatures.

Hydrogen Damage

Fig 8 shows typical window type failure in a boiler tube. The usual features associated with such hydrogen damage are:

* decarburization
* intergranular cracks
* chlorine distribution at the interface.

Hydrogen is one of the normal corrosion product of the reaction in between iron and water

$$Fe + 2H^+ = Fe^{++} + 2H \quad \textit{(in acidic environment)}$$

This nascent or atomic hydrogen diffuses into the steel and reacts with iron carbide to form ferrite and methane.

$$4H + Fe_3C = 3Fe + CH_4$$

Methane is a large molecule and cannot diffuse out of the steel. It collects at ferrite grain boundaries and when the pressure is high enough, leaves cracks behind.

Intergranular cracks

Hydrogen damage manifests discontinuous intergranular cracking due to accumulation of hydrogen and CH_4 at the grain boundaries and subsequently leaving cracks. In fig 8b decarburisation and discontinuous intergranular cracking has been shown. The porous deposits retains the cleaning acids and may cause variations in chloride ion distribution at the interfaces. This may accelerate the anodic reactions at the specific sites.

Fatigue & Corrosion fatigue Failures

Fatigue failure results due to cyclic loading of the component much below its yield strength and appear brittle on macroscopic scale. Beach marks are observable through naked eye. Microscopic examination of failed sample at very high magnification reveals characteristic striations on the surface which is function of the stress experienced by the component.

In fig 9 schematic illustration of fatigue and corrosion fatigue failures are shown. An example of such failure is shown in fig 10 where beach marks of fatigue failure can be seen distinctly.

Failure due to Intergranular Corrosion

Grain boundaries are more reactive compared to matrix. So localised attack of corrosive environment takes place adjacent to grain boundaries resulting in falling out of grain or loss in the strength of the component. Austenitic stainless steel suffers failure due to this reason in the environment where it otherwise shows good corrosion resistance. When austenitic steel is heated approximately in between the temperature range 510°C to 800°C, it becomes "sensitized". In this temperature range chromium carbides are formed at the grain boundaries removing chromium atoms from the nearby regions of grain boundaries . So the corrosive environment attacks at those chromium depleted regions. Schematically the phenomena has been illustrated in fig 11 (a & b). The depletion of Cr and consequent formation of Chromium carbides at the grain boundaries as mentioned above is called sensitization.

Measures to avoid sensitization:

(1) Quenching or rapidly cooling from the solution temperature (i.e. from 1050°C) . If cooling is slow the entire structure will be sensitized and susceptible to intergranular corrosion.

(2) By rendering the amount of carbon very low (less than 0.03%) so that it is not available for the carbide formation at the grain boundaries. Such steels are called extra low carbon steel.

(3) To add strong carbide forming elements like Columbium, Tantalum or Titanium in traces amount which will form carbides in preference to iron and Chromium carbides at the grain boundaries.

Graphitization:

It is microstructural change that some times occurs in carbon or low-alloy steel subjected to moderate temperature in its use. It is due to decomposition of pearlite into ferrite and carbon i.e. graphite and can embrittle steel components, specially when the graphite particles form along a continuous zone.

$$Fe_3C \rightarrow 3Fe + C$$

Iron Carbide Iron Graphite

It is usually a slow process. This kind of failure is usually encountered in under ground pipelines etc. fig 12 represents graphitized microstructures in plain carbon steels.

Selected bibliography

1. Mechanical Metallurgy by G.Dieter
2. Corrosion Engineering by Mars G. Fontana
3. Interpretation of Metallographic Structures by William Rostoker, Academic Press, New York.
4. General Metallography by Ralph.L.Dowdell, H.S.Jerabek, A.C.Forsyth, C.H.Green, Chapman & Hall, Limited, London.
5. Metals Hand Book vol. 11,9th Eds, ASM
6. Metals Hand Book vol. 8, 8th Eds, ASM.

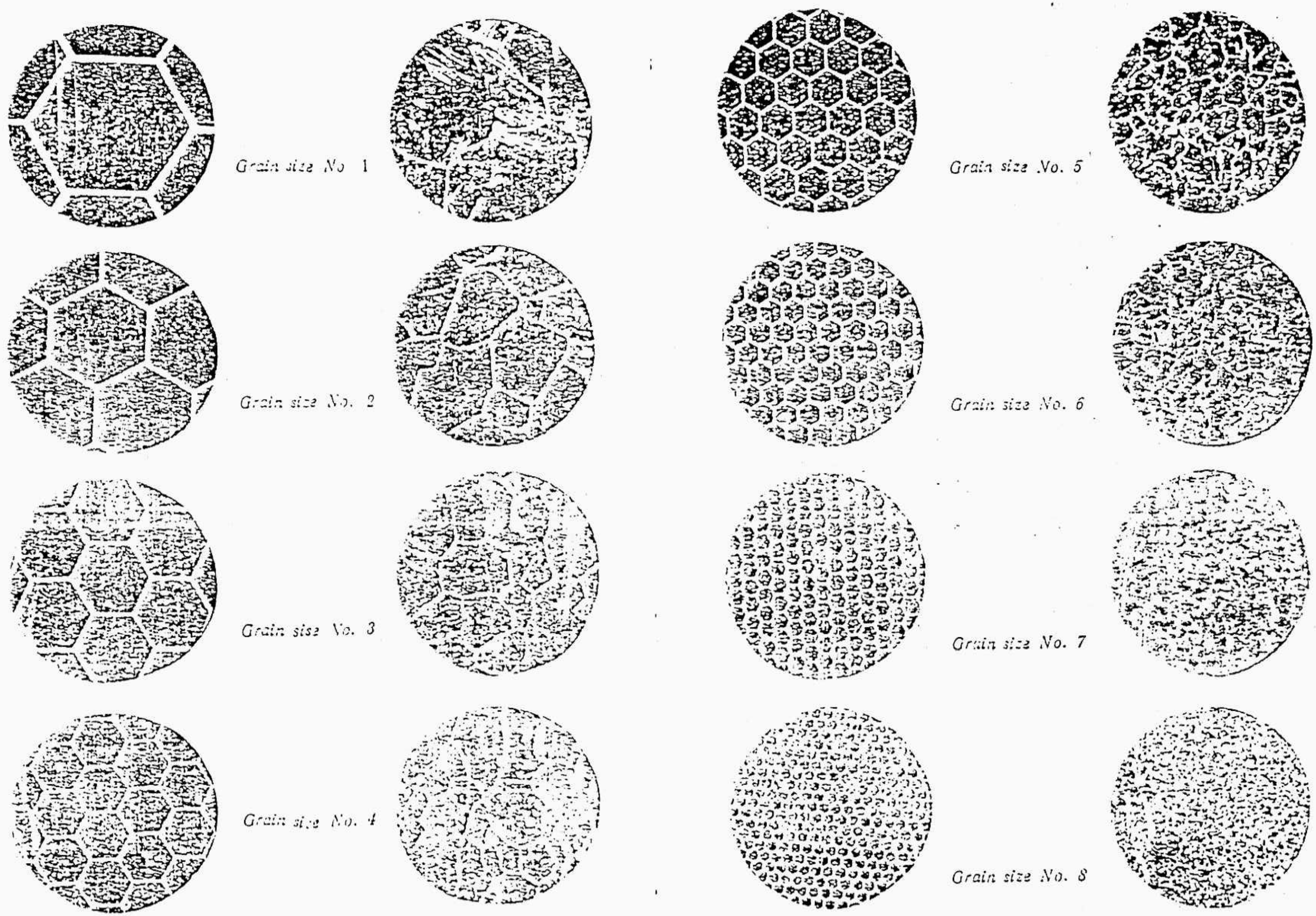

Fig 1 Sketch of different ASTM grain sizes.

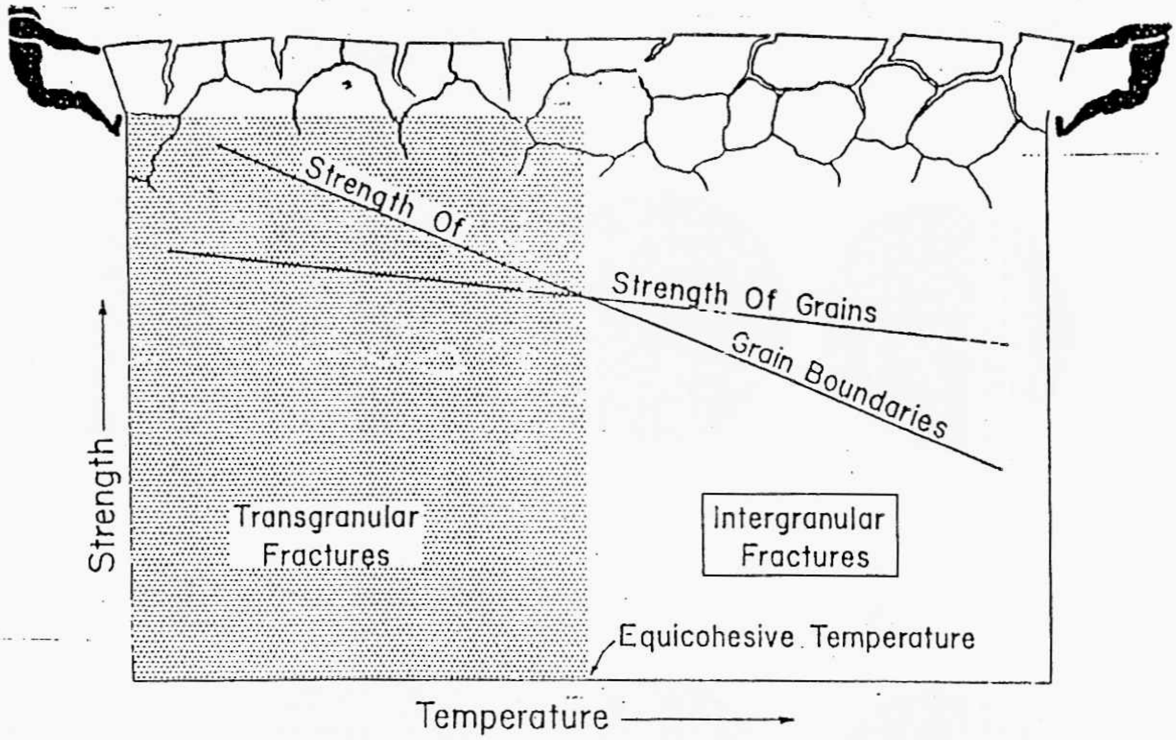

Fig 2 Nature of fracture is related to Equicohesive Temperature.

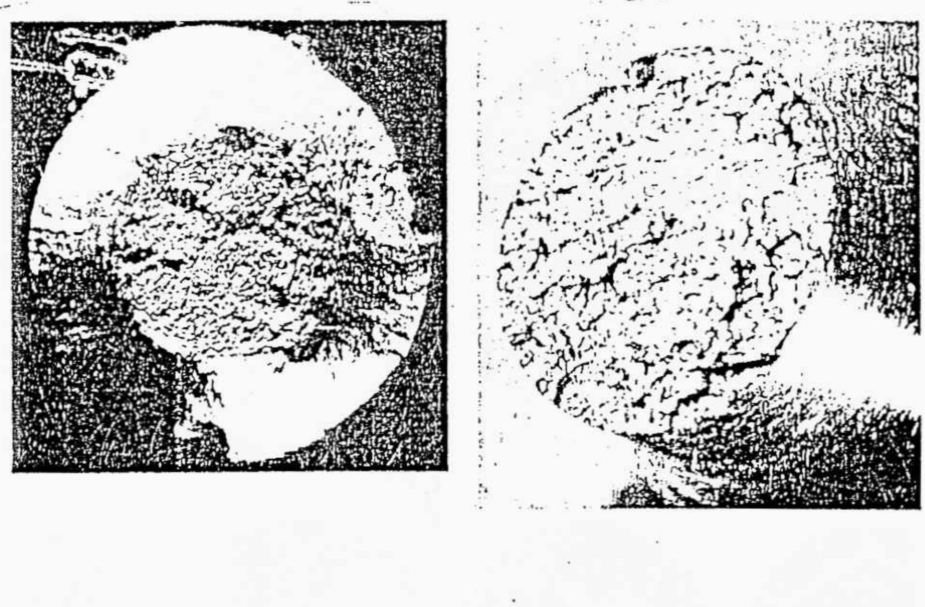

Fig 3: a) Ductile failure showing a fibrous zone.11X
b) Granular brittle failure having large cleavage facets. 2X

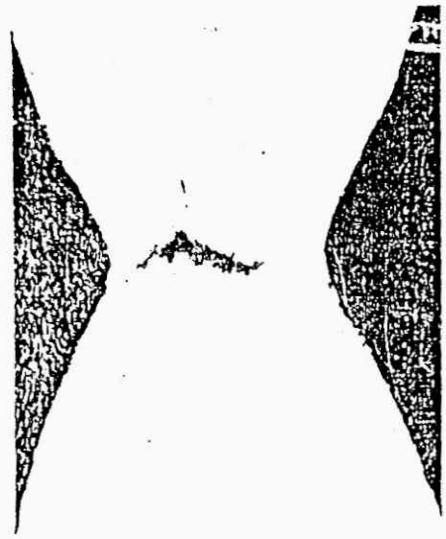

Fig 4 Initiation of crack at the centre and formation of cup and cone type of failure. 4.75X.

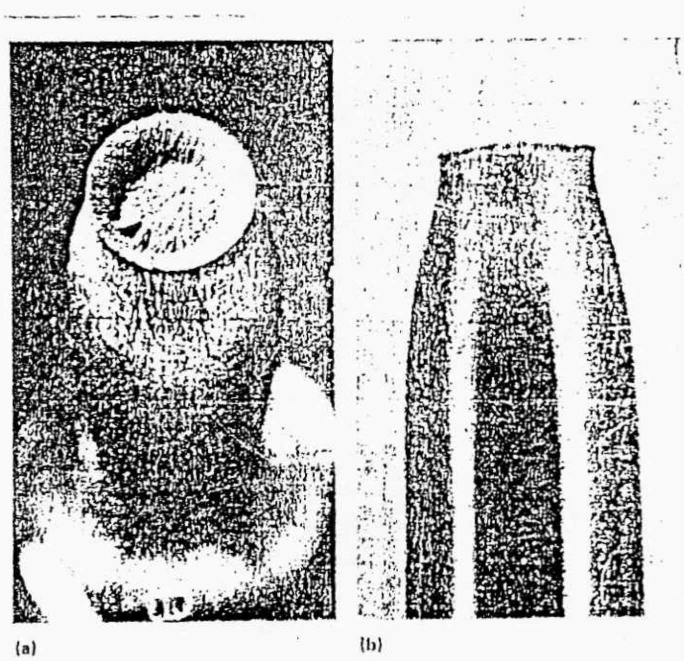

Fig 5 Macroscopic appearance of a) ductile & b) brittle fractures.

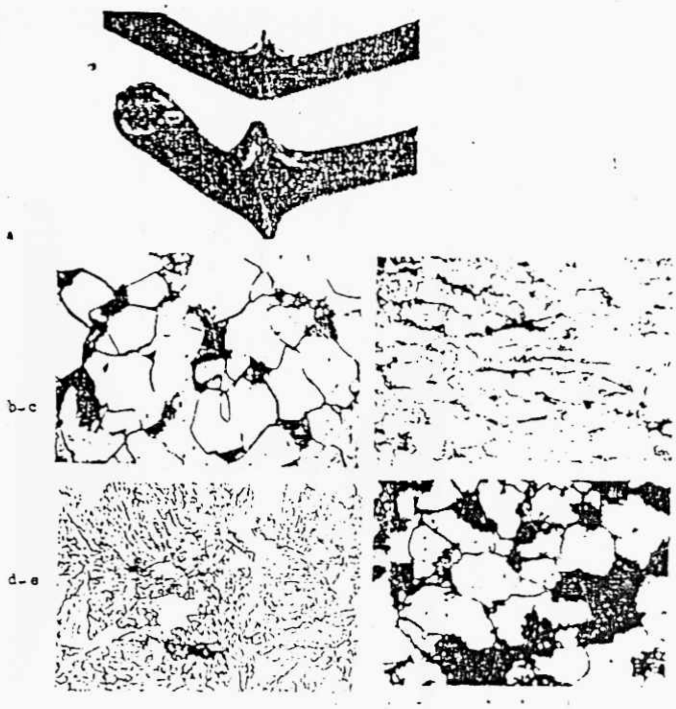

Fig 6: a) Thin lip rupture in low alloy steel superheater.

b) Normal ferrite (white) and pearlite/bainite (black) structure in a alloy steel tubing material.

c) Specimen exhibits elongated grain near rupture resulting from fast over heating below the recrystallization temperature.

d) Specimen displays mixed structure near rupture as a result of overheating.

e) Specimen displays completely transformed structure as a result of overheating.

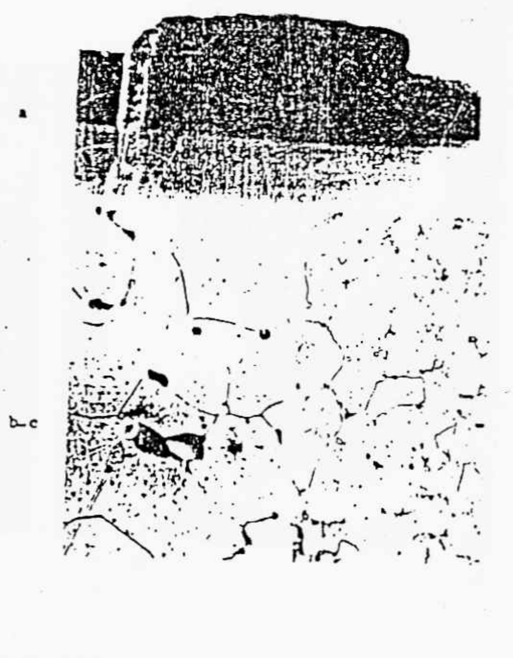

Fig 7: a) Thick irregular lip rupture of a boiler tube, after a service life of about 9 years.

b) Specimen displays creep cavities (black) at creep grain boundaries resulting from long-term creep deformation.

c) Specimen shows spheroidization (globular).

a

b

c

Fig 8: a) Brittle failure of the tube caused by hydrogen damage on inner surface.
b) Decarborized metal containing intergranular cracks beneath a corrosion pit.
c) Chloride distribution at the oxide/metal interface at the base of corrosion pit.

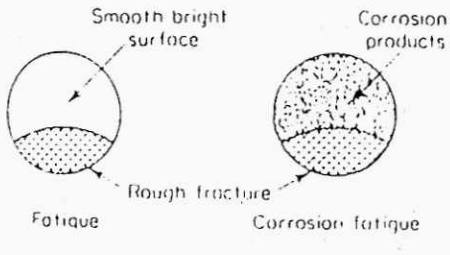

Fig 9 Schematic illustration of fatigue & corrosion - fatigue failures.

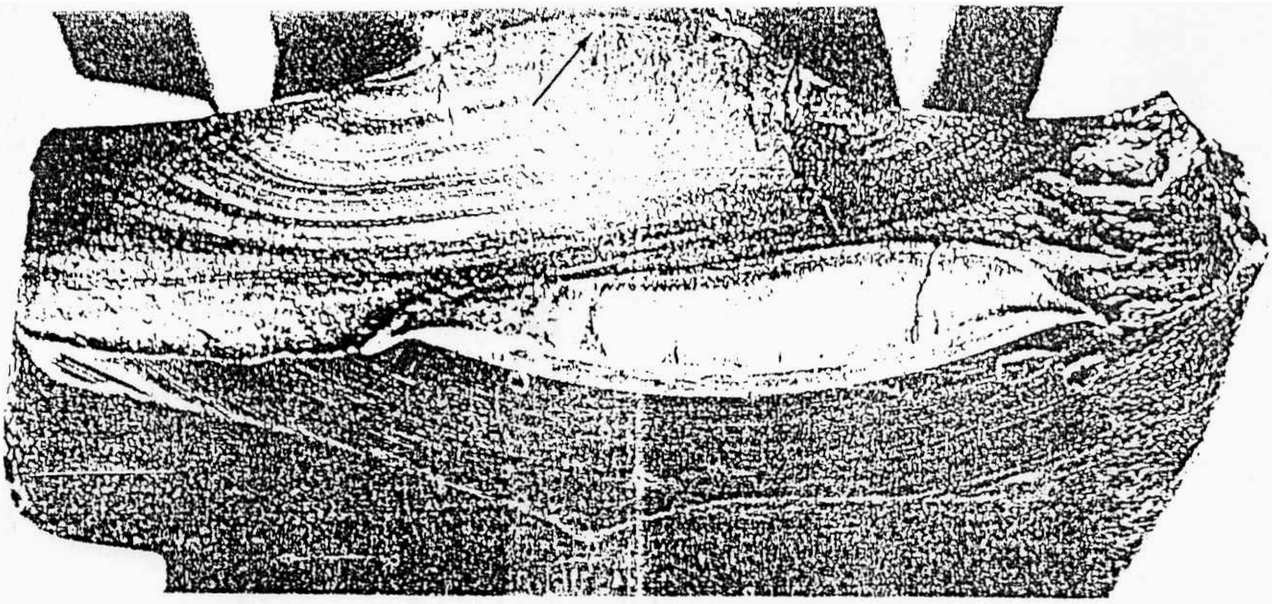

Fig 10 Surface of a fatigue fracture of a craneshaft. Beach marks in upper portion of the fracture surface is to be noted.0.75X

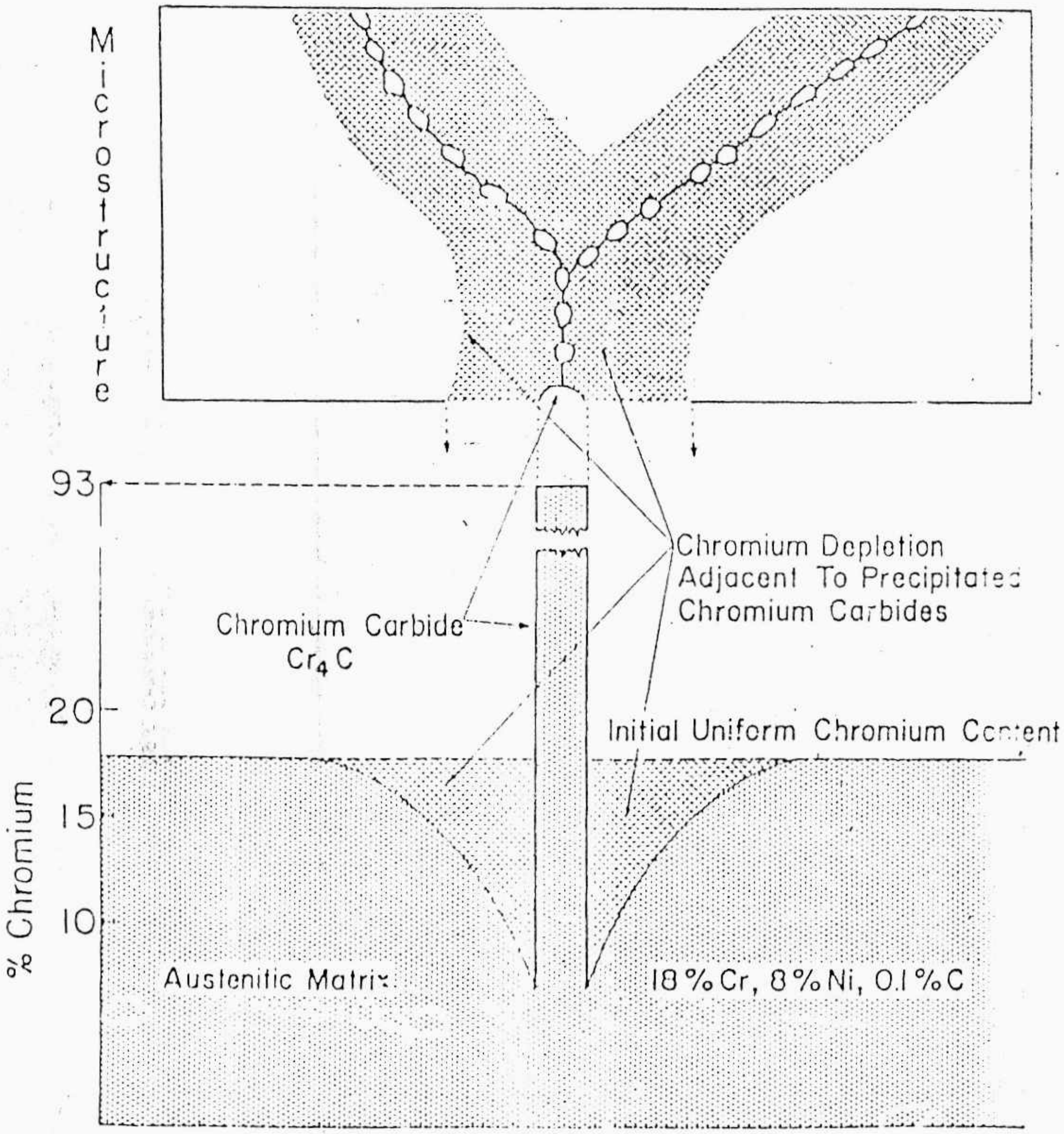

Fig 11 Schematic diagram showing grain boundary precipitation and chromium depletion in the adjacent areas .

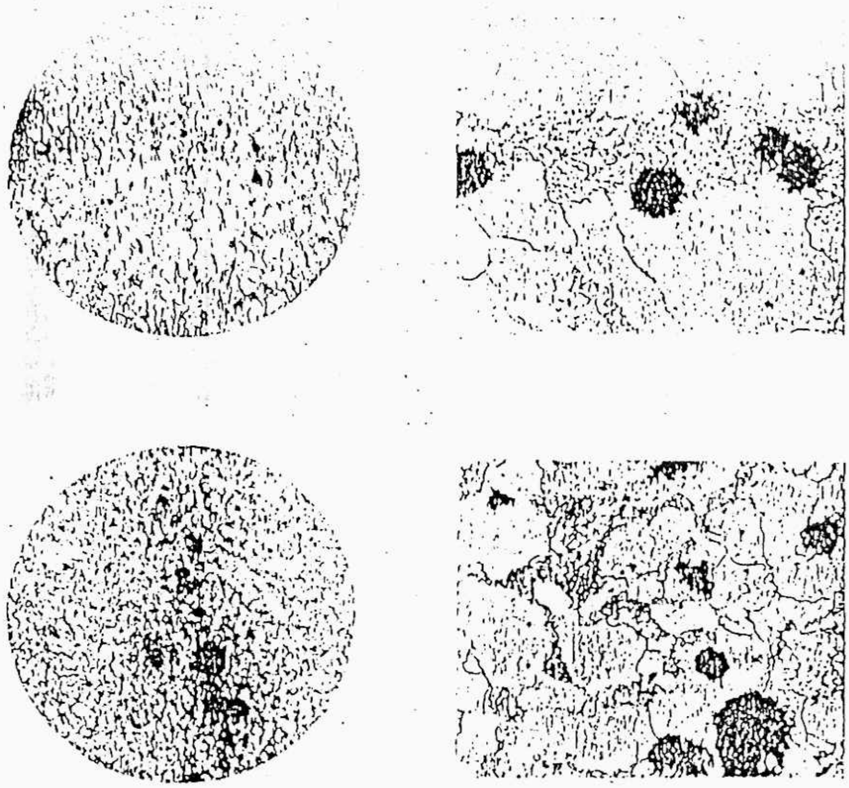

Fig 12 Graphitization of 0.5 Mo Steel
Upper : Isolated nolular formation
lower : Chain like nodule formation
left 40X right 400X