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INCLUSION RATING OF CLEAN STEELS

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Summary

This report covers a study concerning the methods that are available for determining the cleanness level for steels. No outstanding method is available for rating clean steels, when clean steel is defined as free of fatigue initiating inclusions. Fatigue strength is to a large extent dependant of the biggest inclusion size in the stressed volume. Examples of methods that can be used for finding and testing material for the presence of big inclusions is reported.

Contents

1. Background

Det Norske Veritas (DNV) Maritime, Section for Machinery, is responsible for approval of machinery in ships for DNV Class. Allowable fatigue stresses in forged steel is a key input parameter for such evaluations, and are regulated in DNV's Rules.
It is well known that the cleanness of the steel is important for its fatigue strength. To account for this, "clean steel" has been defined in DNV's Rules for forgings (Section A). For "clean steels", an extra 10% is allowed on the components fatigue strength, compared to ordinary forged steels. Clean steel is given a factor of 1,1.
Recently, one leading steel manufacturer has claimed that even higher benefits can be gained by using "superclean steels", and a factor 1,15 has been claimed. The improvement has been demonstrated by fatigue testing.
In order to approve such increased fatigue strength, DNV Maritime, Section for Machinery, needs to know more about the relations between very high cleanness steels and fatigue strength. From the limited investigations DNV has done, it appears that although the claimed 15% may be justified, there are limits to how much gain it is possible to obtain by increasing steel cleanness. Further, it is needed to find ways to demonstrate cleanness – linkable to increased fatigue strength – by chemical and/or metallographic means without performing extensive fatigue test series. Only with such criteria, it is possible to certify "super clean steel", and accept the use of its increased fatigue strength with reference to the material certificate.

2. Procedure

The main part of this work has been a literature survey, reviewing scientifical reports for information on how steel cleanness is evaluated today, and also how the steel cleanness is related to the fatigue performance of clean steels.

I have attended a workshop at Jernkontoret - the Swedish Steel Producers' Association, with the topic *detection of macro inclusions and pores* (with focus on ultrasonics). Six additional meetings with persons concerned with steel cleanness has also taken place. The topics inclusions and cleanness has been discussed. Relevant information from those events has been reported separately to DNV.

3. Introduction

The definitions of *clean steel* are various. Confusion about terms describing cleanness has been mentioned in the steel making and fatigue literature. In [4] and also this thesis report clean steel is a steel being free of big non-metallic inclusions that act as fatigue initiating faults for a defined loading condition. There are some problems with using superlative-like terms to differentiate clean steels from even cleaner steels. As long as steel manufacturing is developing there will always be some new grade that has improved cleanness (in the above sense) compared to former grades.

The non-metallic inclusions in steel is the root of many negative steel properties such as low fatigue strength, toughness and ductility. For good machinability, at least one inclusion type is often listed as beneficial, e.g. sulphur containing inclusions, for example manganese sulphide (MnS) [5].

This study has its origin in the request to DNV from a steel manufacturer. The request was higher rating of fatigue strength of steel from a certain steel manufacturing process. The cleanness level (fatigue properties) was proposed to be assessed by the following type of information:

- Inclusion ratings according to ISO 4967 Method A and DIN 50602 Method K. (low index)
- Oxygen and sulphur levels in the steel. (low contents)

The steel has been proved to have a higher fatigue limit by fatigue testing (performed by manufacturer). The steel grade is corresponding to a DIN 42CrMo4 composition (1% Cr).

4. Objectives

- To what extent is steel cleanness desirable/profitable and how much is the fatigue stress values improved in the *superclean* steels?
- Are there other factors that should be considered when evaluating clean steels?
- What kind of documentation should be requested for asserting that crankshafts made of highly clean steels have certain fatigue properties?
- What standards are applicable for proving that super clean steels hold certain fatigue properties?
- What other ways are there to demonstrate steel cleanness – linkable to increased fatigue strength without performing extensive fatigue test series?

Comments to the objectives are found in chapter 14.

5. Inclusions and fatigue

Fatigue stress is an alternating stress. The biggest occurring inclusion size is possibly the most important factor for determining fatigue strength in steels. So, to improve the fatigue strength for a certain steel a reduction of the maximum sizes is necessary [6]. According to [1] the inclusion size, inclusion distribution (occurrence levels) and the inclusion shape, make out the most important properties of the inclusions when looking at fatigue properties of steel. There are many factors to consider when fatigue strength needs to be improved, such as surface finish, residual stresses and corrosive environment. This study is not dealing with any of those.

Murakami makes far going conclusions of the similarities between cracks, pores, inclusions and other defects. The inclusion (defect) size is often defined as the area that the defect is showing when it is studied along the principal loading direction. The square root value of that area is used in many of the theories encountered in this study. The inclusion (or defect) size is the most important property of the inclusions, together with the hardness of the surrounding steel (matrix). Even if one crack has a three dimensional shape and the other has a planar two dimensional shape the (projected) area can be the same, and therefore both are expected to effect the fatigue limit to a comparable extent [1].

It is the biggest defect in the fatigue stressed volume that will initiate the crack that leads to failure, if all inclusions are subjected to the same level of stress [(p. 44, 54) 1].

As the biggest inclusions are rare it is sometimes more probable that a smaller inclusion starts the (primary) fatigue crack (assuming that the defect is big enough to initiate the crack at its local stress level). An example of a fatigue design model which incorporates this idea is described in [7].

Research is ongoing to show to what extent the chemical composition and mechanical properties of the inclusions themselves will influence fatigue (ability to initiate and also facilitate fatigue crack propagation). These matters are not included here.

Low fatigue limit values for high strength steels are caused by the presence of inclusions larger than a critical size, which is determined by the the steel hardness. According to Murakami it may be concluded that such high strength steel fatigue testing specimens all have individual fatigue limits which leads to a wide scatter of the fatigue strength data. The reason of the scatter is because of the defect size, as well as the location of the defect, will influence the fatigue limit. [(p. 94) 1]

If the sizes of the big inclusions are reduced one can expect a higher fatigue limit for the steel. For steels with low or medium carbon content the critical inclusion sizes for initiating fatigue cracks can be below the critical value. If that is the case the alternating stress does not cause any of the available inclusions to become the origin to the fatigue failure crack. These steels (more) usually initiate the failing crack by *slip band* cracks or *grain boundary* cracks (not inclusions). These initial cracks are initiated in the steel matrix. This special case leads to negligible scatter of experimental fatigue limit data. [(p. 95) 1].

In [1] a simple formula is discussed. It can be used for estimates of the fatigue limit in some special cases.

$$\text{Fatigue limit} = 1{,}6H_v.$$

H_v is the Vickers hardness of the steel matrix close to the inclusion. It's stated to be valid for low and medium strength steels with a Vickers hardness number below 400.

This formula is not usually valid for high strength steels ($H_v > 400$) because of the non-metallic inclusions present, but for very clean steels (without any critical sized defects) the formula is more accurate. The formula estimates the upper fatigue limit (intrinsic fatigue limit) for specimens that contain no defects. When these specimens fail, the fatigue cracks are initiated in the specimen surface by the relative movement of atomic planes (slip bands) as described above.
[(p. 78, 86, 94) 1].

For extremely clean steels (i.e. without any inclusions of relevant size) variations in the microstructure can become the origin of failure. For instance when a martensitic steel has patches of bainite inside [(p. 154) 1]. Inclusions are more dangerous at lower tempering temperature than at higher because the matrix then has higher strength [6] [(p. 42) 1]. Inclusions that are just below a surface (not visible) has the most critical location because the defect will interact with the surface, in addition to a high localized stress [(p. 90, 94) 1].

The fatigue crack that is formed in a component during cyclic loading will grow differently in the different stages of fatigue crack propagation (growth of crack) and the growth rate is known to depend on several microstructural factors. For failures that are caused by a *very high cycle fatigue* stress Kazymyrovych has showed that as much as 99 % of the component life can be spent in the first stage in which propagation is not something that occurs every cycle [8].

Small inclusions are unimportant for crack initiation but may contribute to fatigue crack propagation [6] [9]. Inclusion size and volume fraction (occurrence of inclusions) affects fatigue crack propagation properties of steel. Crack growth is enhanced by the presence of a big number of inclusions. The crack seeks ways to propagate from inclusion to inclusion, similar to a *connect the dots* game. This behaviour is enhanced for flattened MnS inclusions and gives rise to a fatigueanisotropic material [(p. 9) 9][(p. 137) 1].

Low sulphide levels in steel also counteract quench cracking during heat treatment [10]. Manganese sulphides (MnS) are sometimes regarded as not damaging to the fatigue limit but might still become fracture origins if the inclusions has been elongated in one direction [(p. 137) 1].

Some inclusions coalesce and form clusters (clouds). Such impurities are very negative for fatigue strength [11] [12].

6. Formation, altering and behaviour of non-metallic inclusions

As said; The (projected) size of the initiating inclusion (or defect) is a main factor for the fatigue limit and performance of the steel. This chapter deals with some aspects of the formation of non-metallic inclusions and how inclusions can be altered during steelmaking (inclusion engineering). Focus lies here on aluminium deoxidised steel, although this process has not been confirmed from the steel manufacturers part. Also calcium treatment is a way of altering the inclusions in steel and can improve certain type of inclusions effect on fatigue. Two inclusion-types are pointed out by the steel manufacturer to be the dominant ones; MnS and Al_2O_3.

Effort is laid on describing and predicting how many inclusions are present in the different steels but one should remember that the big inclusions are not evenly distributed in ingots or continuously cast material. The streaks of segregation/macro inclusions in the cast material should be known and the risk volumes should preferably end up in a low stress part of the application.

Inclusions which are introduced by material that is outside the melt are called *exogenous*. For example teeming powder, reactions with refractory (brick) materials or mixing or pulling down slag. Exogenous inclusions are regarded as the most dangerous, but are also the least common inclusions. Big sizes are normal. They are unpredictable by their nature but have often known origins in the steel making processes. Even if the secondary steelmaking (purification and alloying stage) has produced a very clean steel, exogenous inclusions can appear before the process is ready.

Endogenous inclusions are more predictable and often effort is focused on predicting and monitoring these. In this text endogenous inclusions are discussed unless specified otherwise. When the steel is cast non-metallic inclusions are already present in the liquid steel and thereby differ from precipitates that are formed during solidification [(p. 1) 11].

Inclusions of different chemical composition behave differently when they undergo hot or cold working. If a big reduction is possible certain inclusions change shape and become less harmful (typical is MnS). Some inclusion types are elongated and some are crushed. There are also types that are more or less unaffected by hot working. Generally oxides are regarded as more harmful [1]. Non-metallic inclusions with low melting temperatures deform during rolling (hot working), for example manganese silicate and calcium silicate. An aluminium oxide (Al_2O_3) does not behave this way and it keeps its shape when the material is being wrought (deformed) [(p. 3) 11]. Compared to a soft inclusion, of the same original size, the hard will maintain the same influence on the fatigue strength of the material. For a material with softer inclusions the fatigue strength improves in the elongated direction but degrades in the transverse direction [9]. This also means that inclusion rating methods get different values depending on if the deformation has altered the inclusion sizes. Also void formation around hard inclusions can occur during working.

In aluminium deoxidised steel the oxide inclusions can contain around ten times more oxygen than the dissolved oxygen in the steel [11]. The most of the oxygen is therefore bound to the inclusions. The total oxide level is primarily lowered by the movement of oxygen-containing inclusions from the melt into the covering slag. This oxide inclusion removal is promoted by flotation and/or stirring the melt. The counteract to these purifying mechanisms is the *reoxidation* of the melt. Reoxidation happens when the melt comes in contact with air, slag or refractories. It is important to protect the melt from surrounding air at all stages of the steel making. The cleanliness depends on this [(p. 133, 137) 11].

As mentioned above the induction stirring and/or inert gas bubbling in the secondary steel making stage purifies the steel. A prolonged period of the purification stages does not necessarily make the steel cleaner [(p. 134) 11].

If calcium additions is performed the inclusion fatigue properties may be improved. This calcium treatment does not make the individual inclusions smaller and the inclusions in fact become more difficult to remove from the melt so it is important to have a low level of aluminium oxides when calcium is added. The benefit is that solid aluminium oxides will be transformed to liquid calcium aluminates that will not cause clogging and do not attach to each other causing aggregated inclusions (clusters). Another benefit of Ca-treatment is that it desulphurizes the steel and makes it more isotropic, by reducing the occurrence of manganese sulphide (MnS) [(p. 138-139) 11]. An increase of fatigue limits can also be expected (especially in the transverse direction specimens) [9]. Rare earth metals added to the melt are also used for this purpose.

Some steels are more likely to form inclusion clusters. If this clustering behaviour is decreased the steel might have the same maximum inclusion sizes (individually). When they are separate they are less likely to initiate cracks.

7. Conventional inclusion ratings of clean steels

The steel manufacturer that has originally initiated this study was unable to distinguish the new (superclean) steel from the clean steel when using ISO 4967 Method A. This standard is an inclusion rating method that uses microscope samples. The suggestion was then that the DIN standard 50602 method K3 should be used (a similar method). This DIN standard together with the sulphur and oxygen level of the steel was proposed to be used for asserting the new steel's cleanness. This part of the report deals with the (micro) inclusion rating standards. If the reader needs references to the standards mentioned here, please see the reference section.

Frequently used methods to evaluate clean steels are methods using microscopic evaluation of polished surfaces. These are for example AISI E45, ISO 4967, DIN 50602, SS 111116. Some of the methods uses comparative pictures showing inclusions, and some methods involves measuring of the inclusions. Also automatic image analysis software can be used and is standardised. The result of these tests are indexes (comparable values) that show to what degree inclusions are present in the sample. The inclusions observed have sizes of a micro scale and are not the inclusions that normally cause fatigue failure. Normal sample areas are 160 to 400 mm^2. These so called *conventional* inclusion rating standards are not a good way of rating highly clean steels (meaning free of critically big inclusions). The results has weak correlation to fatigue testing results [(p. 76) 1] [2] [3]. The inclusions that cause failures (in fatigue testing) are often reported as bigger than expected. In [6], the *comparative picture ratings* are said to give less quantitative information about the size and morphology of inclusions, than the ones using *image analysis* (an option to comparative charts). Many fields of view needs to be examined for assuring that the right index value is determined for clean steels.

As an example of when the rating is not applicable; In [2] a steel making process is described, that eliminates bigger inclusions and makes the steel more isotropic. In this type of steel the occurrence of the smallest inclusions (below 5 µm) are reported to increase at the same time as the fatigue properties are enhanced. This evidently cleaner steel might be rated as a dirtier steel if some conventional method is used (clean means free of harmful inclusions).

Also Juvonen [3] discusses the relevance of the conventional methods and the lack of correlation to fatigue properties (in calcium treated steels). He summarizes that chemical composition of the melt together with the DIN 50602 and EN 10247 evaluation only gives indications for the fatigue behaviour and not a complete picture.

There is a need for a method that finds big inclusions in clean steel. A dirty steel, measured by the traditional standards, can be more fatigue resistant than a cleaner steel [1].

8. Extreme value statistics methods

> *Prediction methods, shortly described here, can be a possible way around the fact that complete inspection of the material in a component is sometimes not possible or reasonable today. They may also be a way to predict the materials fatigue behaviour. Extreme value statistics methods can be a possible way to rate clean steels and make connections or even predictions on fatigue strength [1, 7 13-15] The polished areas that normally are observed (by microscope) are in the range of 160 to 400 mm^2 per sample. The use of small areas might be a practical way of assessing cleanness of a big volume of steel even though the inspected area (or thin volume) is so tiny in relation to the amount of steel in the component. The methods can be used to calculate the probability of finding an inclusion bigger than a critical size [6].*

It is by *statistics of extremes* applied on the smaller non-metallic inclusions found by microscope that information on the big end of the inclusion sizes can be derived. The goal of the extreme value statistics methods is to connect the small inclusion sizes, studied in a microscope, to rare bigger (endogenous) inclusions that does not normally show up in the microscope. The inclusions measured are unlikely to include any of the bigger inclusions that cause the failures. By predicting the size (and even occurrence) of these relatively big inclusions the fatigue behaviour can be calculated.

One statistical method is presented by Murakami in [1]. There evidence are presented for the correlation between actual fatigue testing and the inclusion size prediction. Additional developments of the same author to that theory/method have been published also. One is [13].

The statistical methods in *[1, 7 13-15]* are performed by measuring only the bigger (or biggest) inclusions that shows up in a microscope. The conventional rating methods differ in that procedure, because they normally consider all visible inclusions, even the smallest ones. For clarity, there are conventional methods that only measure the biggest inclusions in the sample but no extrapolation towards bigger (critical) inclusion sizes is done.

To make sure that the prediction is relevant the investigated area might sometimes need to be enlarged. Up to 8400 mm^2 according to [3]. The reason to this conclusion was that when using one of the statistics of extreme value methods, the predicted inclusion sizes was small in comparison to the ones found at the fatigue crack initiation sites (fatigue test specimens).

In [14] four different statistical methods are compared;
- Generalized Pareto distribution (GPD) and the Exponential distribution (EXPGPD). For these two methods inclusions above a threshold size is measured.

- Statistics of Extreme Values (SEV) method and the Generalized Extreme Values (GEV) method. Here only the biggest inclusions in every field of view is measured.

When using the statistical methods for predicting the biggest inclusions in steel there are some mathematical parameters that needs to be investigated. These parameters describe the shape of the inclusion size distribution function. This function models the occurrence of inclusions of different sizes. The parameters sometimes needs to be verified for different kinds of steel, and assumptions on the parameters might need evidence or data from other sources [14, 16].

One benefit of the GPD approach as compared to SEV (if a certain parameter can be determined to have a negative value) is that the maximum inclusion size will have an upper limit whereas for SEV the maximum size might be unreasonably large [15]. It is reasonable to think that the endogenous inclusions do not have the ability or time to grow to ridiculously big sizes. Another key parameters of the GPD method is that not only the size of the biggest inclusion can be evaluated, but also the presence of other slightly smaller inclusions [7]. It is possible that the most stressed volume does not contain the largest inclusion. The probability might be higher, that the fatigue failure originates from an inclusion that is more common but smaller. From a design point of view this is beneficial. One drawback for GPD would be that more inclusions needs to be measured. Another paper for the SEV method [13], that has a related topic discusses how to do the sampling if several inclusion types are present in the steel at the same time. In that case the sampling is proposed to be modified so that the prediction will be on the biggest (less frequent) inclusion type.

In [12] clustering of inclusions made the procedure of estimating maximum inclusion more difficult. The extreme value model used did not predict the right size.

The fact that the conventional inclusion ratings sometimes do not correlate to fatigue strength is also mentioned in the description for the ASTM standard E 2283 [23]. This standard is for the use of extreme value statistics. I have not been able to study the standard itself but in [17] a successful round robin test has been performed with 17 different laboratories. The steel used there contained 0.001% sulphur, 0.031% aluminium, 16 ppm calcium and 5 ppm oxygen by weight; a steel with mainly oxides. See chapter 13.1 for more information on E 2283.

To conclude this chapter it can be restated that <u>if</u> these methods can be applied they give information of the cleanness of highly clean steels (and in the best case also fatigue characteristics). The steel inclusion characteristics and process route are deciding factors. Steels with the same chemical composition does not necessarily have the same distribution function of the inclusion sizes.

9. Very high cycle fatigue testing (tension-compression)

Rotating bending fatigue testing is perhaps the most used and useful way of describing material fatigue properties (inclusion and matrix behaviour) [(p. 129) 1]. These techniques are part of DNV material testing rules of forged crankshafts and are not discussed further here. Other types of fatigue testing exist. Also small sized crank throws (part of shaft) are tested (in full size) with high frequencies. Normally when high frequency methods are used, the part that is tested decides the frequency (oscillation). The relatively high frequencies enables to test more cycles than 10^7 that normally has been the case (and formerly almost the definition of "eternal" fatigue life). For marine crankshafts normally a higher fatigue life is used.

In Ekegrens research [16] the fatigue testing is done by tension-compression testing with a frequency of 20 kHz and 10^9 cycles. A high number of cycles at a relatively low stress level gives the big inclusions chance to be the crack initiators. The volume tested to bring out the largest inclusion is about 300mm^3. One of Ekegrens conclusions is that the used fatigue testing method is a good way for finding large inclusions in highly clean (tool) steels. The very high cycle fatigue testing (VHCF) is also used in [8]. The term *gigacycle fatigue* is also used to describe the testing. Tests are carried out at low stresses and will therefore enable the largest defects within tested material volume to initiate and cause (primary) cracks and cause fatigue failure. The inclusions found in fracture surfaces can be used for predicting bigger inclusions in the material. In [16] statistics of extreme values (SEV) is used to predict big inclusions in tool steel. See *chapter 8*. Predictions are made both from studying polished surfaces and also by investigating the fractured surfaces of a fatigue test specimens. Interesting remarks in [16] are; Other defects than inclusions might be found in fracture surfaces, which are not found in microscope or electron microscope. Ultrasound (and other non destructive methods) for finding defects may have problems with correctly indicating the defect size for the studied steel. The area scanning methods (measurement by microscope on polished surfaces and from that predicting big inclusions) can be useful, but it is important to assess big surface areas. Before one can predict the largest inclusion size in a certain steel by SEV, the distribution function (parameters related to the mathematical prediction) must be tested for the specific steel and the steels process route.

VHCF is a method that is testing material at the real number of cycles that crankshafts must withstand. The approach used in [8] and [16] is therefore interesting. The method is quick considering the high number of cycles.

One should remember that even if fatigue testing is performed there is no guarantee that the biggest inclusion (caused by the steel making process) is present in any of the fatigue test specimens. Big inclusions exist in components, and are unnoticed if they do not lead to failure, and possibly not even then.

10. Oxygen and sulphur levels

In the past the oxygen level has been a good way to describe the cleanness of steel. Today that is not the case. Only oxygen and sulphur content is not enough information to give the whole picture of the steel cleanness. This was already stated stated in 1981 by Kiessling [4]and is also stated today by Juvonen in [3].

The volume fraction of inclusions depends directly on the oxygen and sulphur content [6] but measuring the total oxygen level does not give any information of the characteristics of the inclusions. If a steel sample contains big inclusion clusters this will be indicated as a very high O content [(p. 18, 35) 11]. Such events are random and the sample is re-analysed. The oxygen (O) level of the steel is a good indication of the cleanliness. But even with relatively high oxygen content (and big inclusions) a rolling or forging operation can reduce the negative effects of those inclusions in the material. Murakami [1] suggests that the quality of the steel making process should be measured by fatigue testing of the steel and also by good process control with minimal variations.

A decrease of the oxygen content that does not result in a size reduction of the biggest oxide inclusions will not improve the fatigue limit [(p. 136) 1].

During the oxygen sampling (liquid sample) inclusions caused by auxiliary material is not present. The teeming powder and teeming refractories are part of later stages. Two steels with the same oxygen content can differ in fatigue strength because of the variations of the inclusions chemical composition [2].

Oxygen and sulphur measurements are methods to control the steel making processes which in their turn control the big inclusions, so information of the O and S levels are useful.

11. Ultrasonic immersion tank testing

To be more sure of exactly what defects the steel contains it would be of high interest to examine a large volume of steel. The UIT (Ultrasonic Immersion Tank) method is used by scanning a several kilogram piece of steel. The surface is milled and the test is performed with the specimen in water.
Most of the ultrasonic methods in the industry are developed to fit a certain production stage, steel-type or product. There are some standards related to the subject.

All of the methods in earlier chapters will find inclusions in small areas or volumes, when compared to ultrasonic volume scanning. The possibilities of using ultrasonic scanning is interesting I think despite the fact that few successful research results can be found in the references to this study.

Because of the difficulty to find big (infrequent) inclusions by conventional rating methods (microscope) the Ovako Hofors steelworks has implemented the use of ultrasound [18]. Ultrasonic immersion tank testing – UIT is used. The bigger end of the inclusion size distribution can thereby be monitored for quality and process control. Bigger inclusions are detected and the the scanned area (volume) is a lot bigger (kilograms instead of fractions of grams). 10 MHz is the normal testing frequency for bigger inclusions (100µm and above). Compared to ASTM E588 and SEP1927 the author of [18] claims that Ovako has developed a more sensitive ultrasonic procedure allowing better cleanness assessment. The inclusions smaller than 25 µm are assessed by optical and/or electron microscope [18].

A conclusion about UIT for calcium treated steels in [3] is: “UIT provide more relevant information about the fatigue properties and machinability of these steels than the conventional inclusion rating methods do, but its resolution capability still needs improvement”. The author could not correlate the UIT results to the inclusions that were in fact responsible for failure in fatigue testing.

The study in [19] has not been successful with detecting inclusions of the type MnS in 50CrMo4 steel. No defects could be detected although the inclusions were there. In that study the steel seems to be too “tight” and MnS inclusions are known to be hard to detect by ultrasound. No voids were present at the inclusion-matrix boundary. Voids could be beneficial for detection, but not for fatigue strength. The tested samples in [19] contains MnS clusters that limit the fatigue strength.

12. Other methods for finding big inclusions

In short, some additional ways of finding inclusions are described in this chapter. Magnetism and x-ray transmission methods are not included here. Some information of those can be found in [6].

There is a method for detection and assessment for inclusions called Laser Ablation-ICP Mass Spectrometry (LA-ICP-MS). Pulsating laser is used to sample the chemical composition of the surface. By mass spectrometry an inclusion map is created, of the measured surface (with information of the inclusions chemical composition). The area scanned by this method does not need to be polished (mirror finish) which is an advantage [20].

In [21] laser ablation is also used. There the maps are called LIPS maps. There a resolution of 4,8 µm along and 50µm between the *laser crater paths* is specified. A area of 6 mm^2 is covered in less than a minute.

Electron beam button melting (EBBM) is a method where the steel sample (up to 1kg) is melted by an electron beam in vacuum. Drops of the liquid metal are collected in a water cooled copper mould (testing metal is still liquid). The inclusion particles concentrate on the surface of the molten *button*. The inclusions can then be inspected. Microscope and/or SEM can be used to classify the inclusions. About 70% of the oxygen content (in inclusion form) will float. The method has been used for comparing clean steel and according to [6] the large sample ensures that statistically significant values can be obtained for inclusion concentration and size in super-clean materials [6]. *The Cold Crucible Remelting method* described in [6], also separates the inclusions from a molten sample (about 100 g). It is said to have potential for rating highly clean steels. The sample is not in contact with the mould because electromagnetic forces act and float the melt.

A time consuming but also relevant method is *chemical dissolution*. An acid dissolves the matrix and leaves the inclusion types that do not react with the acid. Some inclusions can therefore not be studied. It gives quantitative information but is time consuming [6]. The method is sometimes referred to as the *Slime* method.

Liquid Sampling and Hot Rolling Method (LSHR). This method is developed to detect macro inclusions in the steel making stage. A liquid steel sample (0,4 kg) is collected and solidifies to a rod and then rolled. The thin strip of rolled steel is scanned in a UIT tank. Inclusions above 25µm are recorded by this method. In [22] a description of this method is available.

13. Applicable standards for finding and rating inclusions

If the reader seeks references to the standards below he/she should look up the standard name (or issuer) in the second part of the References chapter. A WWW search will quickly lead to the source.
In this chapter I give a short description of the standards that I have found that is related to material testing for non-metallic inclusions. Some of them are are in use at DNV. Note that the DIN 50602 standard is proposed by the manufacturer to be used for highly clean steels but this specific standard will be replaced by EN 10247.

ASTM E45-05: The microscopic inclusion rating is performed by the use of comparative pictures (reference charts) or by automatic measurement. A variety of rating methods in E45 are described and different comparative values can be derived. Method D is described as a suitable rating method for low inclusion content steels. The E45 standard is not applicable for cast or lightly worked material. In the standard text it is stated that clean steels may be more accurately rated if automatic image analysis is chosen. E45 uses two different comparison charts. The JK-chart (see SS 111116) and the SAE chart (SAE standard J422). There are references in the E45 standard to standards that assess big inclusions (and segregations) as *macro-etch* tests (ASTM E381), (blue) *fracture test* (ISO 3763), *step down* (turning) and *magnetic particle inspections* (Aerospace Material Specifications, AMS 2300 to 2304).

If more information about the chemical composition of the inclusions is wanted there is a related ASTM standard, E-2142 -08, that incorporates the use of scanning electron microscope (SEM).

If automatic imaging is used the procedure is found in ASTM E1245 - 03(2008) *Standard Practice for Determining the Inclusion or Second-Phase Constituent Content of Metals by Automatic Image Analysis*.

ISO 3763:1976 alternatively SS 111110: *Fracture test and step down testing*. The SS 111110 and ISO 3763 standard describes how turned and milled bars can be inspected for big inclusions. Big inclusions will scratch the surface and leave marks. The applicability of these methods on modern steels can be strongly questioned and a more detailed description is therefore left out. The standards are despite this in use. The fracture test is not good for highly clean steels [6] These macroscopic methods deal with non-metallic inclusions visible to the naked eye or with the aid of a magnifying glass with a magnification of not more than ten times. Only inclusions equal to or greater than 1 mm long are taken into consideration.

SS111116 can be said to be the Swedish equivalence to ASTM E45 and EN 10247. There are differences but also similarities between them all.

ISO 4967 *Determination of content of non-metallic inclusions -- Micrographic method using standard diagrams*. This is in use by DNV and has been mentioned in earlier chapters.

EN 10247 (replaces DIN 50602) In this standard the recommended minimum reduction is five because of possible porosity.

The Japanese JIS lattice point standard for inclusion rating is reported in [(p.141) 1] to have weak ability to differentiate clean steels from each other.

13.1 Extreme value statistics standard

ASTM E2283 - 08 Standard Practice for Extreme Value Analysis of Nonmetallic Inclusions in Steel and Other Microstructural Features

From the E2283 abstract from [23]: “This practice describes a methodology to statistically characterize the distribution of the largest indigenous (endogenous) non-metallic inclusions in steel specimens based upon quantitative metallographic measurements. This practice enables the experimenter to estimate the extreme value distribution of inclusions in steels. The procedures in determining non-metallic inclusions in steel are presented and discussed in details.”

13.2 Ultrasonic methods standards

ASTM E588 - 03(2009) Standard Practice for Detection of Large Inclusions in Bearing Quality Steel by the Ultrasonic Method. This is a interesting standard if the steel can be inspected by the method. Inclusion chemistry, size, shape, location, and distribution may limit the ability of the method to provide indications distinct from those generated by the surrounding metallurgical structure [24]. Some related ASTM standards are *E214 Practice for Immersed Ultrasonic Examination by the Reflection Method Using Pulsed Longitudinal Waves* and *E428 Practice for Fabrication and Control of Steel Reference Blocks Used in Ultrasonic Examination.*

Other ultrasonic standards are listed below but have not been investigated or referenced.

MIDAS (Mannesmann Inclusion Detection by Analysing Surfboards) Industrial standard. Testing for macroscopic cleanness of continuously cast steel. Developed by Mannesmann. Inclusion detection by analysing rolled *surfboards*. (Comparable to LSHR but bigger samples)

RVI (Renault method)
SEP 1921 1984-12
SEP 1927 (Adapted for bar shapes (inclusions above 0,3mm))
DIN EN 10228-3 (Ferritic and martensitic steels)
DIN EN 10228-4 (For stainless grades)
ÖNORM M 3002 (Ultrasonic testing of forged and rolled products made of steel and other metallic alloys with higher requirements. Austrian standard.)

14. Conclusions

- To what extent is steel cleanness desirable/profitable and how much is the fatigue stress values improved in the *superclean* steels?

  Comment: The question should be put in relation to the component at hand. If the crankshaft has a highly stressed surface where the roughness is causing cracks then cleanness is not of importance. Wrong design or unsuccessful surface treatments also make cleanness non contributing to the fatigue strength. No clean steel can stop cracks that have already originated. In other words: Cleanness is not profitable if the fatigue does not start at the inclusions. If we look only at the material itself and its ability to withstand cyclic loading the cleanness is vital. References in this study support that smaller inclusions are necessary to get better fatigue testing results. When cleanness is no longer the issue other microstructural inhomogeneities is then initiating cracks (atomic slip bands that are rippling the surface, bainite spots in martensite, segregations). If the anisotropy level is lowered (by inclusion engineering) the fatigue performance for multi-axial stresses could benefit, although it might not be as evident if fatigue testing of longitudinal fatigue test specimens is performed. The improvement would show mostly in the transverse direction.

  The given fatigue data from the steel manufacturer shows that a more than 5 % fatigue strength increase has been achieved when comparing to the former generation of clean steels. I can not argue the results. A deep understanding of the actual steel making process (size distribution of inclusions) is needed to answer the second part of the question. A full understanding on how the inclusion size distribution has changed would explain the manufacturers fatigue results.

- Are there other factors that should be considered when evaluating clean steels?

  Comment: Yes. I think focusing the material testing towards bigger inclusions is important. Clean steels are often defined by the absence of harmful inclusions. The levels of small non harmful defects seems to be of less importance as long as they do not for clusters.

  Deformation of the material will improve fatigue strength if the inclusions are crushed and mixed into the matrix. Some inclusions do not deform and their fatigue properties are therefore maintained. The positive aspect is that they do not cause anisotropy.

- What kind of documentation should be requested for asserting that crankshafts made of highly clean steels have certain fatigue properties?

  Comment: This also needs more investigation. One possibility could be statistical evaluation by ASTM E2283 – 08 but this is perhaps preferably done on the final material. If this method is applied the sampling and preparation of stays the same as for conventional rating methods but the data is differently evaluated.

  Also ultrasonic evaluation is interesting, because measuring a big volume of steel gets rid of the enormous extrapolation and prediction that is necessary when small area samples are studied.

A discussion with steel manufacturer is beneficial and a query should be made on what macro inclusion evaluation methods they use today. Information on the metallurgical steps that the steel undergoes gives insight on what inclusions to expect (sizes, chemical composition and so on). Depending on how the manufacturer is keeping track of the macro inclusion levels the most suitable documentation may vary.

- What standards are applicable for proving that super clean steels hold certain fatigue properties?

  Comment: Fatigue testing standards. The ASTM E2283 – 08 is a possibility.

- What other ways are there to demonstrate steel cleanness – linkable to increased fatigue strength without performing extensive fatigue test series.

  Comment: None all-round outstanding method. All methods that assess a big volume of steel is safer if one wants to find big inclusions. Ultrasonics detects pores which also can be included in the term cleanness. UHCF/gigacycle fatigue testing can be a possibility to speed up and rationalize fatigue testing.

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For ISO, ASTM, SAE standards; search these sites to find the information:
www.astm.org
www.iso.org/iso/searchstandards.htm
www.sae.org