Development of Clean steels – Advantages in Ladle metallurgy and testing technology

Technical Report Archive

DEVELOPMENT OF CLEAN STEELS
– ADVANTAGES IN LADLE METALLURGY AND TESTING TECHNOLOGY

Ovako has an extensive R&D since many years, an area that now is in an even higher intensity. Some of the R&D work is published in our technical reports.

Due to that Ovako of today has had a number of different company names and used various trade marks we have until now chosen to not have these reports publicly available. However, many of these technical reports contain valid data about material and steel grades that we still promote, but with other names etc

The following Technical Report from 2000 is part of a series of technical reports we issued in the late 1990's describing various aspects on how to achieve the clean steel we produce. These also show the development from earlier technical reports. See Ovako Archive Technical report 8/1983 and 1/1985, as well as 1/1997.

Data and processes in this report represent state of art at time of publishing, and is to a large extent base for our current technology and capability.

In this Technical Report there is used the following Company names and trade marks that no longer is used by Ovako AB.

Ovako Steel; This company name is no longer used. The organization is now part of Ovako AB.

SKF; Is a separate company with no link to Ovako.

OVAKO STEEL

Technical Report 1/2000

Development of Clean Steels – Advantages in Ladle Metallurgy and Testing Technology

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OVAKO STEEL

Ovako Steel is the world's leading manufacturer of bearing steel and a major producer of other special engineering steels. We are a fully owned subsidiary in the SKF group.

Our main strengths is in the field of long special engineering steel products – seamless tube, bar, and surface removed wire. Rolled rings are also a specialty. A large part of the production is further processed by machining. This share is increasing and illustrates our intention to progress towards further processing and higher specialization.

The most important customer segments are the rolling bearing industry, the automotive industry together with their subcontractors as well as the rock drilling and general engineering industries.

We have manufacturing units in Sweden, France, the U.K and the United States. Steel production is confined to Hofors, in Sweden. The production capacity is approx. 500,000 tonnes of special engineering steel per year.

Research & Development

Our R&D mission is to pursue an efficient product and process development, adapted to existing and new technology, and within our product areas be recognized as the world leader in metallurgy, materials technology, machinability and metal cutting technology as well as heat treatment. The ultimate targets is to offer our customers the best total economy in their production.

Abstract

The advances of ladle metallurgy have led to very significant developments in the internal cleanliness of low alloy steel products. This development, which started with the design of the first functioning ladle furnaces with effective stirring facilities, has now reached a level where 'standard' produced steels outperform complex and costly remelting processes. The steel making advances necessitates corresponding developments in testing procedures, perhaps in particular as concerns the control of the non-metallic inclusion contents of steel. The developments in steel processing are reviewed and recent progress in the rating of non-metallic inclusions is detailed, in particular as regards test methods encompassing ultrasonic techniques. Further, recent advances in assessing fatigue initiation causes in bearing steels are discussed and related to contents and morphology of micro-inclusions.

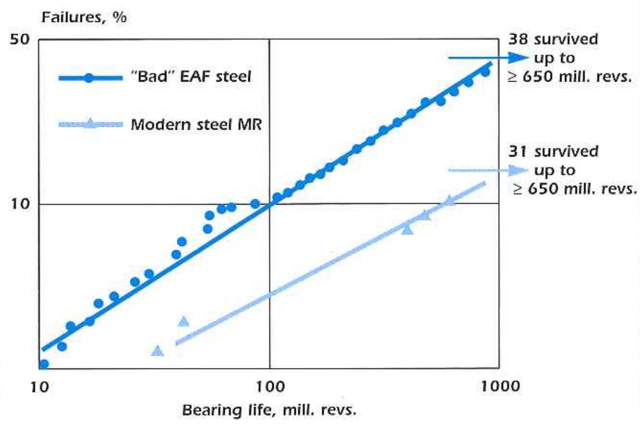
KEYWORDS: bearing steel, fatigue, non-metallic inclusions, ultrasonics.

Introduction

The strive to produce ever cleaner steel for demanding applications took a large step forward in the later part of the 1960's when the ladle furnace technology was developed and put into large scale use. The difference in rolling bearing life achieved using ladle furnace technology in lieu of the conventional two-slag practice in electric arc steel production is very significant. [1]

Since the 1960's continuous efforts to improve deoxidation practices have led to a gradual but significant improvement of the internal cleanliness. Part of the improvements achieved are not only due to the enhanced knowledge of deoxidation, stirring and vacuum treatment but also in better and more stringent teeming procedures. Introduction of up-hill teeming and effective teeming shrouds have vastly reduced reoxidation and thus significantly contributed to the reduction of the oxygen related inclusion contents.

The developments seen since the 1960's thus are partly due from successful technology changes but also to a large extent based on consistent and continuous developments in all the procedures and auxiliary materials associated with production of high-quality steels, from scrap selection to solidified product.



Heat	Oxygen ppm	Titanium ppm	Sulphur %
MR	11	11	0.013
"Bad" EAF	39	10	0.013

Fig 1. Fatigue life of ball bearings produced at the same time based on two-slag practice ('dirty') and ladle furnace produced ('clean') bearing steels.

Cleanliness development

Oxygen content

As the solubility of oxygen in solidified steel is very limited, the total oxygen content is an adequate measure of the total amount of oxide inclusions which is present in steel. It certainly does not tell the whole story, the morphology and the chemical composition of the oxide inclusions can vary widely depending on processing and deoxidation and this will strongly affect the properties of the finished product even if the total oxygen content remains the same. Similarly, the content of large inclusions caused by contamination from auxiliary materials (as teeming powder, sand etc.) is unrelated to the oxygen content, and thus a steel with very low total oxygen may still contain significant amounts of harmful inclusions.

At Ovako Steel, the oxygen content of bearing steel has been recorded since many years. The development in oxygen content, calculated as the average yearly oxygen for the about 200 000 tonnes produced per annum shows a distinct improvement also in the most recent ten years.

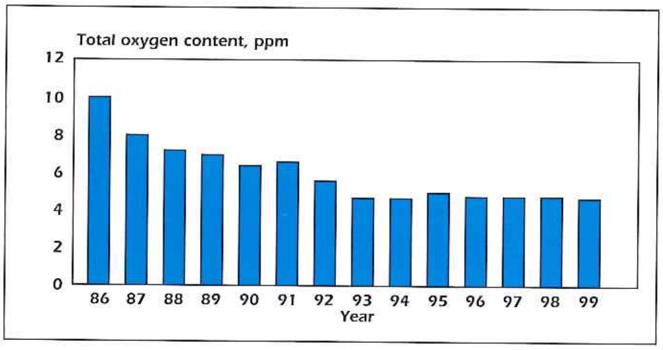


Fig 2. Oxygen content development for rolling bearing steel production.

Not only has the total oxygen content average been reduced, but the heat to heat variation has also been strongly reduced in the same time period.

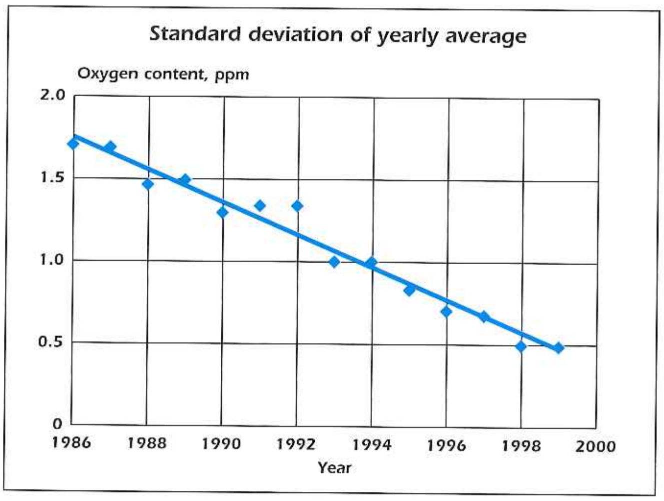


Fig 3. Oxygen content standard deviation for rolling bearing steel production.

Currently all serious bearing steel producers have access to fairly advanced ladle furnace steel processes, but in spite of this the variation in oxygen content is significant. The data shown in *Fig 4* have been produced by procuring bearing steel products from all major bearing steel producers in the world with a heat production date of 1995 or later. In most cases more than three heats have been tested and averaged for each producer. Evidently it takes time and effort to attain full grasp of the deoxidation process, and each producer needs to fine tune and develop the process steps involved in order to achieve truly low oxygen levels.

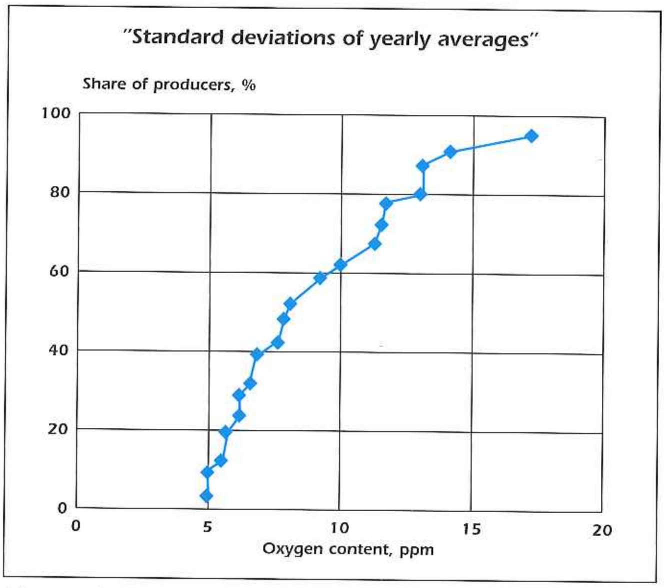


Fig 4. The 'world' bearing steel oxygen content distribution.

Differences in cleanliness between continuously cast and ingot cast bearing steel products are often discussed. Dividing the oxygen contents in the 'world' oxygen distribution into continuously and ingot cast products indicates that internal cleanliness largely is defined by the ladle furnace processing. The distributions for the differently cast products are very much the same as far as oxygen content is concerned.

The current situation as regards what can be achieved in oxygen content and variation is exemplified by a random selection of the bearing steel heats produced at Ovako Steel during a two week period in March 2000.

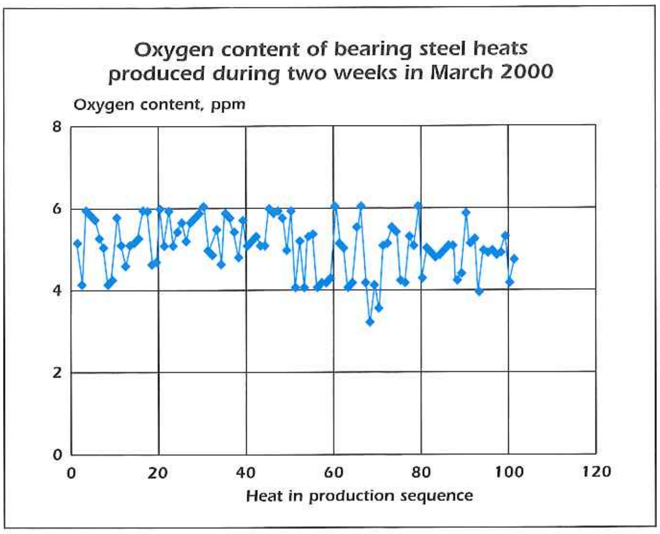


Fig 5. Oxygen content in Ovako Steel bearing steel produced during two weeks in March 2000.

Titanium content

For the same material used to derive the 'world' oxygen content distribution, the titanium distribution has been assessed.

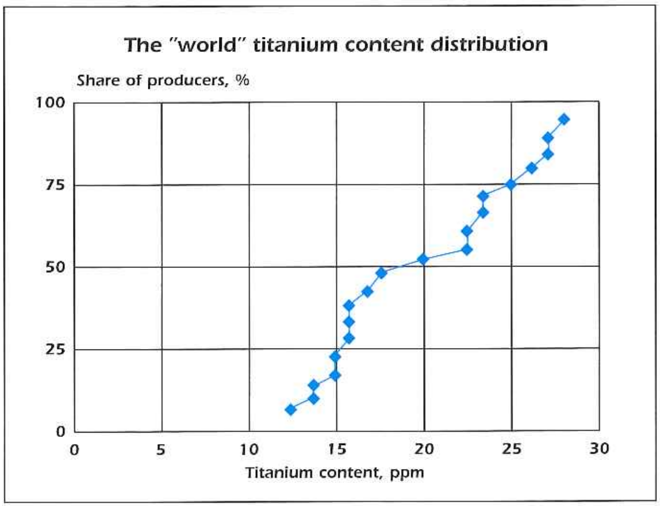


Fig 6. The 'world' titanium content distribution in bearing steels.

In this case the alloying material used has far larger influence on the final titanium content (and thus the amount of titaniumcarbonitrides formed) than the steel processing practise.

The titanium content is closely related to the titanium present as tramp element in the ferrochromium used, and thus the titanium content can be closely correlated to the chromium content. Controlling the titanium content in bearing steel thus is a matter of adequate alloying element selection and quality control enforcement.

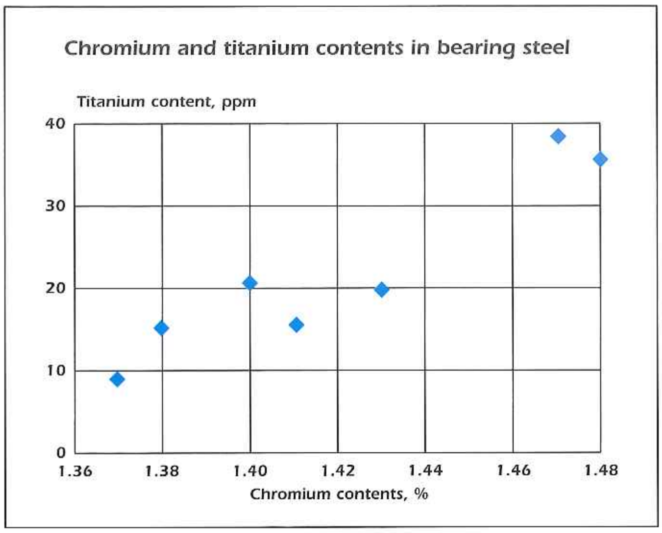


Fig 7. Relationship between titanium and chromium content for bearing steel produced using high purity ferrochromium.

As for oxygen, the current situation as regards what can be achieved in titanium content and variation in bearing steels can be visualised by displaying the results for a random selected production period

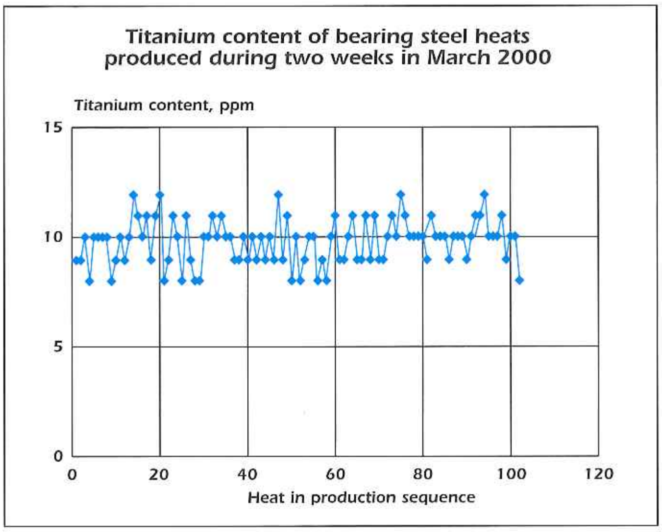


Fig 8. Titanium content in Ovako Steel bearing steel produced during two weeks in March 2000.

Sulphur

The sulphur content can be regulated to the desired level in the ladle furnace, so basically the sulphur level is determined by the customer specification. Today, quite low sulphur contents can be reached in the ladle furnace by combining argon bubbling under vacuum and inductive stirring. This means that bearing steel specifications which earlier only could be met by remelting operations, as for aircraft applications, today easily can be met with ladle furnace technology.

Macro inclusions

There are several standardised laboratory methods available for assessment of the contents of large inclusions. The two most common ones are 'step-down' and 'blue fracture' testing.

In step-down tests bars are fine turned in distinct steps and the defects larger than 0.5 mm are recorded. The results are normalised per unit area of tested surface.

The blue fracture test records defects larger than 0.5 mm on a bar cross section area which has been hardened, fractured and then tempered blue to increase the visibility of defects.

Other methods, more seldom used are magnetic particle testing and ultrasonic tests, as Detection of Large Inclusions in Bearing Quality Steel by the Ultrasonic Method (ASTM E588).

The improvement in internal cleanliness as regards large inclusions as measured by these methods was remarkable already early in the introduction of ladle furnace technology.

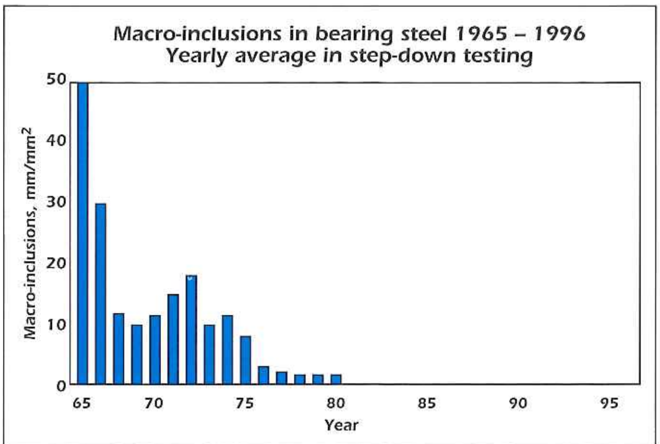


Fig 9. Yearly step-down testing averages for bearing steels produced at Ovako Steel.

Very evidently, better methods were needed to find and quantify the amount of large inclusions in modern, ladle furnace produced products.

Equipment to do this became available in the later half of the 1990's, and large efforts have been spent to develop standardised procedures for the assessment and quantification of large inclusions in bearing steel products.

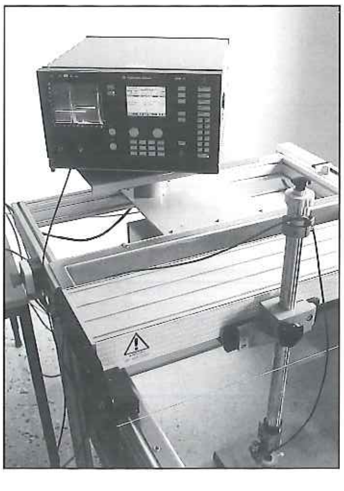


Fig 10. Ultrasonic scanning equipment.

At Ovako Steel today, billet samples are milled plane parallel and scanned with a 10 Mhz probe which enables resolution of about 25 mm steel and by scanning the billet slice a volume corresponding to several hundred microscope samples can be examined in minutes [2].

By careful evaluation it is quite feasible to distinguish and quantify porosity's, segregation's and oxide inclusions, and to count and size classify the inclusions.

To get a comparative number, inclusions are classified into three size groups, weight factors are applied to each group, and the result is normalised to 10 kg's of tested steel basically in accordance with the ASTM E588 procedure.

It early became evident that the degree of metalworking reduction very significantly affects the results obtained. This fact must be considered if specifications setting tolerance limits are to be made, and it must also be realised that above a certain degree of reduction (for a given testing frequency) zero results will always result, just as for the testing methods mentioned above.

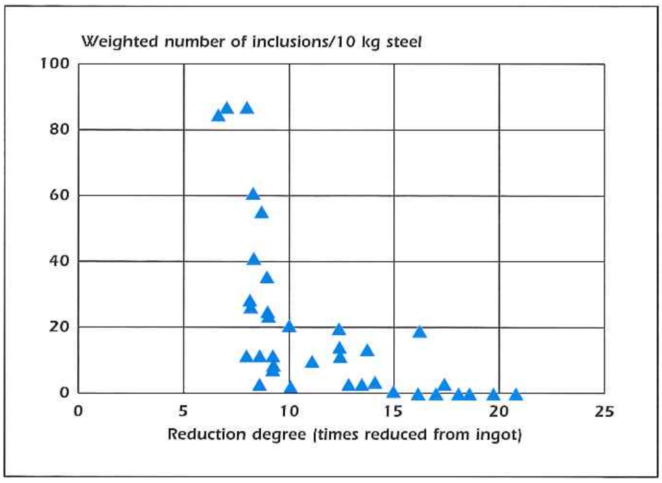


Fig 11. Reduction degree and ultrasonic scanning result on bearing steel billets.

The ultrasonic scanning technique ensures that the content of large inclusions can be monitored and fed back to development activities in the steel making plant. However, evaluation of the size and composition of a large number of inclusions detected in the ultrasonic scanning reveals that the majority of these inclusions are not the ones normally found in the step-down or blue fracture tests. Typically, the inclusions found in such tests are exogenous inclusions as teeming powder or sand entrapped in the steel during teeming. The ultrasonic scanning technique captures inclusions which are in the upper size range of the deoxidation or re-oxidation inclusion population. This has been proven by analysing the composition of a large number of oxide inclusions detected, pin-pointed and scanning electron microscope analysed.

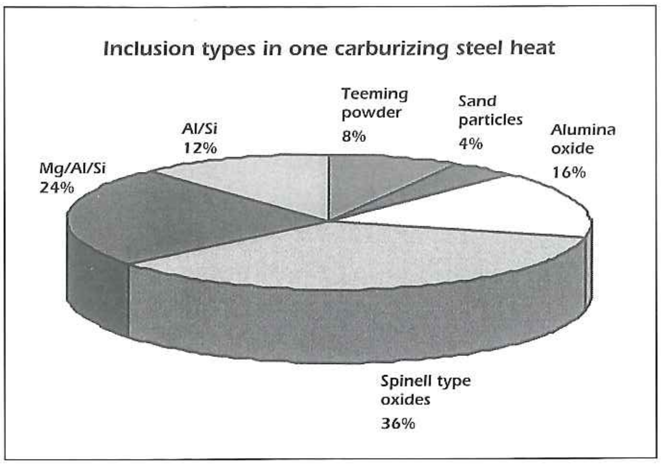


Fig 12. Chemical composition of inclusions found by UL-scanning in one single heat.

The ultrasonic scanning technique not only has the capability to detect and quantify the content of non-metallic inclusions, it also can detect and quantify porosity's and can give a very accurate description of the segregation level in any given sample.

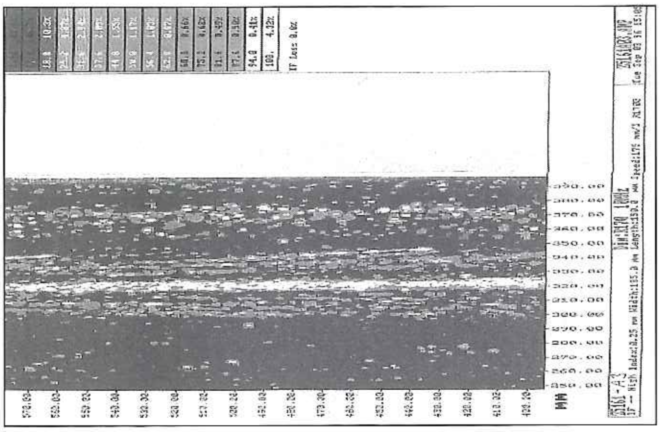


Fig 13. Steel sample with central porosity, segregation and large inclusions.

Micro inclusions

The world-wide standard for micro-inclusion rating today is the ASTM Standard Test Method for Determining the Inclusion content of Steel (E45) with the sampling and evaluation rules of ASTM Standard Specification for High-Carbon Anti-Friction Bearing Steel (A295) specification which fundamentally is based on the Jernkontoret inclusion rating scale developed at Ovako Steel in the 1940's. Even with the recent upgrades of the E45 the rating results on today's very clean steels are becoming meaningless. For oxide inclusions the vast majority of the bearing steel heats produced at Ovako Steel today rate zero and thus provide no meaningful development information.

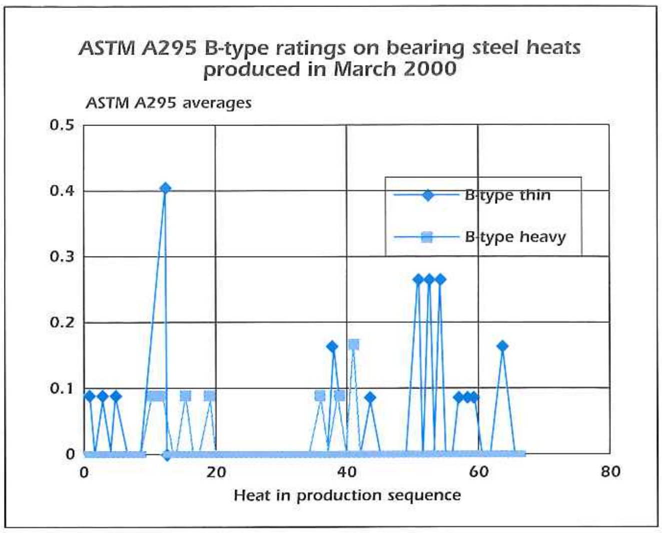


Fig 14. B-type oxide ratings on bearings steel heats produced in March 2000.

Attempts have been made at improving the precision of microscopic inclusion ratings, and one significant contribution is the expansion of the old JK scale Steel – Evaluation of non-metallic inclusions – microscopic methods – Jernkontorets plate II for quantitative assessment (SS 111116) which has been standardised in Sweden. This method does provide meaningful correlation to chemical composition results.

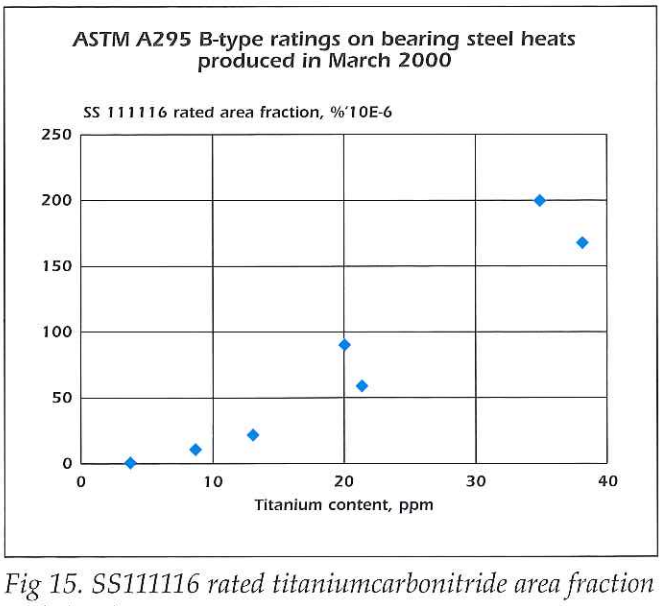


Fig 15. SS111116 rated titaniumcarbonitride area fraction and titanium content.

Even if the correlation is very strong it becomes evident that the rating values are approaching zero for the cleanliness levels typical of today's ladle treated steels for producers with a high quality profile.

This means that even semi-quantitative microscopic rating methods do not provide the required resolution to help in improving clean steel quality further.

As earlier indicated the resolution power of the ultrasonic scanning technique can be significantly enhanced by increasing the testing frequency. By increasing the testing frequency to 50 Mhz the resolution is very significantly increased, and inclusions bordering on the size range normally detected in conventional E45 ratings can be detected. However, the examined volume becomes much smaller which means that larger inclusions go undetected.

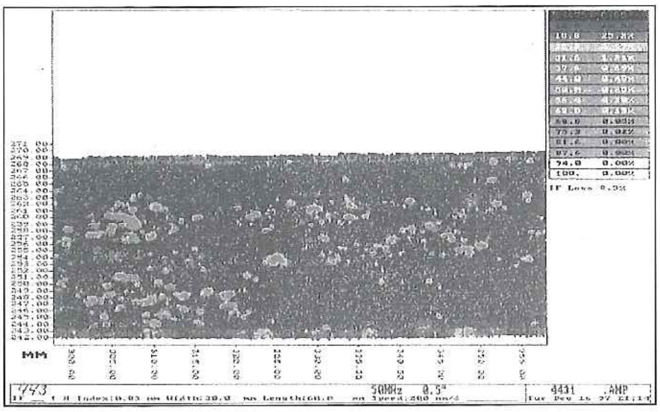


Fig 16. Example of scan at 50 Mhz on low carbon, medium oxygen content steel.

By combining the high resolution technology in ultrasonic testing with advanced sampling procedures new routes can be opened. Tests have been made with samples taken during different stages of the steel making process, and by giving the samples a predetermined and constant degree of reduction, the changes in inclusion size and total content during different stages of the steel making process can be established.

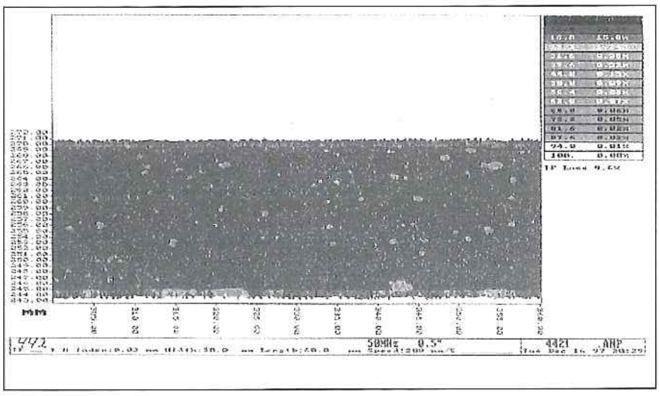


Fig 17. 50 MHz frequency scan of a sample taken just prior to teeming (rolled, reduced 4x).

In recent times a further exciting step has been taking in the attempts of trying to bring inclusion engineering into the steel making process [3]. Developments in the Optical Emission Spectrometry signal processing of the conventional chemical composition determination can be used to effectively analyse the content, size distribution and composition of non-metallic inclusions.

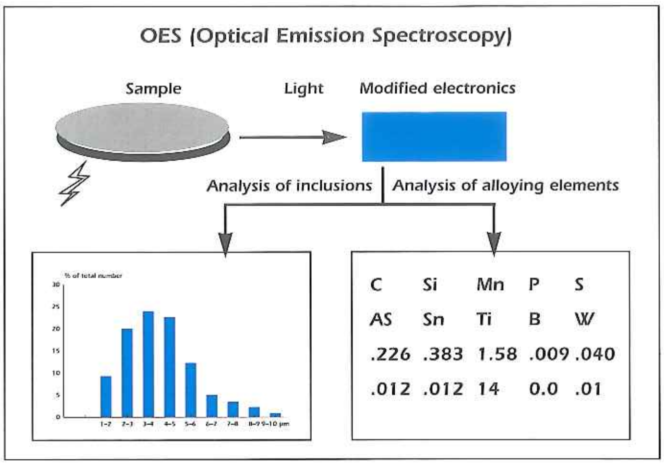


Fig 18. Signal capturing procedure for OES inclusion definition.

By adopting special signal interpreting routines, the size distribution of different oxide inclusion composition can be derived and this can be done separately for different inclusion composition groups.

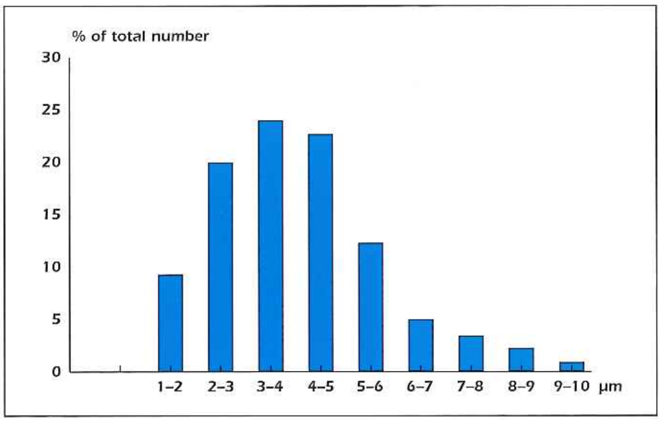


Fig 19. Size distribution of OES detected alumina oxide inclusions.

Internal cleanliness and properties

All particles present in steel products are potential failure nuclei in any component exposed to stresses. In particular alternating stresses generate a situation where fatigue failures initiate from defects present in the stressed zones.

A number of factors affect the fatigue initiating propensity of defects in steel. It is evident that large defects, as the ones detected in ultrasonic scanning in the 10 Mhz range are of a magnitude where immediate initiation occurs. The inclusion sizes detected by the high frequency ultrasonic scanning technique is close to the size range where practical initiation occurs, while the OES technology attacks the size range of inclusions determinant for the success of the deoxidation process.

However, here things become complicated as several factors seem to influence the fatigue initiating power of different inclusion types. Attempts have been made at understanding the relative importance of different inclusion parameters on fatigue initiation.

Fatigue testing

In the efforts to improve fatigue properties the main target has been to reduce the oxygen content and consequently the presence of oxide inclusions which could initiate a fatigue crack. However, not only the amount of oxides but their size distribution and chemical composition influences the fatigue properties. It is well known that smaller inclusions are beneficial for the fatigue properties [4] but investigations also show that different types of oxide inclusions may affect the fatigue initiation mechanisms [5]. Moreover, when the amount of oxide inclusions is decreased, other defects in the structure may act as fatigue initiation sites. When the oxygen content is reduced to below 6 PPM titanium carbonitrides starts to cause fatigue failures [6]. Even if the titanium content is as low as 10 PPM.

Due to their morphology the titanium carbonitrides are more hazardous than the oxides and a titanium carbonitride could therefore be much smaller than an oxide in size to cause a fatigue failure at a certain stress level. Finally, one fatigue initiation source may be carbide clusters. This has been found in heavily segregated bearing material leading to very poor fatigue properties.

Fatigue test procedure

Rotating beam fatigue specimens were machined from the half radius position in soft annealed SAE 52100 bars ranging from 80-100 mm. The heat treatment and surface preparation involved austenitization at 860°C for 20 min, oil quench, tempering at 160°C for 1h, grinding and shot peening. The heat treatment produced a martensitic structure with a hardness of 62-63 HRC. The shot peening was made in order to suppress surface initiation due to surface flaws.

Rotating beam fatigue testing was carried out at ambient temperature in laboratory air in an AMSLER UBM 200 machine. A single stress level, 950 MPa, was used and the lives were assumed to conform to the two-parameter Weibull distribution. Specimen surviving more than 10^7 cycles were suspended. All fatigue fractures were studied in a scanning electron microscope and the initiation sites were identified, measured and analysed.

Different oxide types

Two heats produced by the electric arc furnace route were fatigue tested at a single stress level of 950 MPa. Aluminium deoxidation, alloying and degassing were carried out in an ASEA-SKF ladle furnace. The steel was uphill teemed to ingots. The compositions of the heats are shown in *Table 1.*

Heat		Heat **D 7788**	Heat **G 1578**
C	weight%	0.99	1.04
Si		0.30	0.22
Mn		0.37	0.32
P		0.013	0.012
S		0.003	0.006
Cr		1.39	1.43
Ni		0.11	0.14
Mo		0.03	0.05
Cu		0.14	0.17
Al		0.028	0.028
Ti	ppm	10	12
O		3.5	6.0
N		58	100

Table 1. Chemical composition.

The majority of the fatigue failures were caused by oxide inclusions, approximately 85% in both cases and the reminder was from titanium carbonitrides. Surprisingly, the heat with the lower oxygen content had the lower fatigue life. This is shown in *Figure 20* with the L50 values which is the number of cycles which is probable to give 50% fatigue failures for a certain stress level. The initiating oxides found at the initiation sites were examined. It was concluded that the size distributions and consequently average oxide sizes, shown in *Figure 21,* were similar.

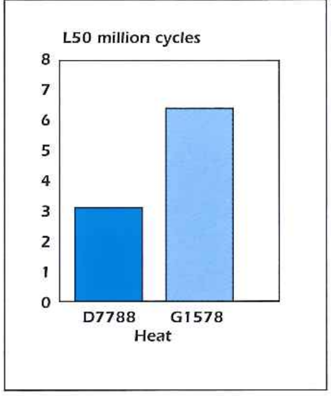


Fig 20. L50 life in 10^6 cycles for heat D7788 and G1578 tested at a stress level of 950 MPa.

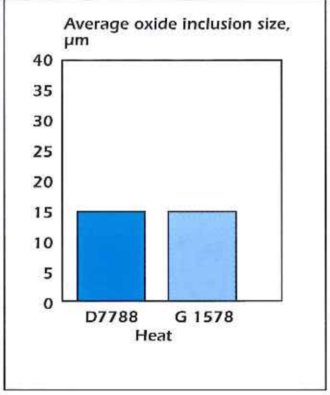


Fig 21. Average size of oxide inclusion initiating fatigue.

There must be another reason for the difference in fatigue life than the amount and sizes of oxides. The fracture surfaces also showed different oxide configurations. The oxide inclusions in both heats consisted predominantly of *Al*, *Ca* and *Mg*. In most of the cases also *Mn* and *S* were present. Unfortunately it was not possible to quantify the chemical composition of the oxides in detail.

However, during the examination it was noticed that some of the oxide inclusions were cracked leaving half of the inclusion on each fracture surface whereas others were solid and the inclusion remained on one of the fracture surfaces, see *Figures 22 and 23*. It is interesting to note that the majority, 67%, of the inclusions found on the fracture surfaces in the heat with lower fatigue life, D7788, were of the cracked type. The share of cracked oxides in heat G1578 was 25%.

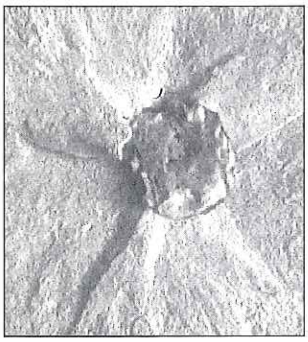


Fig 22.
Cracked oxide inclusion.

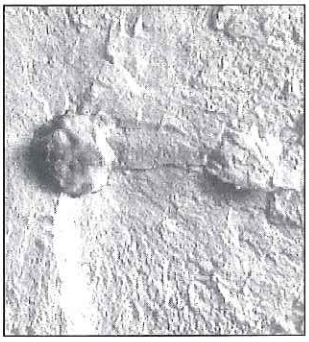


Fig 23.
Solid oxide inclusion.

Theoretical calculations on the two oxide configurations was made using a finite element routine [7]. This confirmed the observed result, i.e. that a cracked oxide is more hazardous than a solid oxide.

Oxides versus titanium carbonitrides

Two heats produced the same day by the standard route were fatigue tested at a single stress level of 950 MPa. Aluminium deoxidation, alloying and degassing were carried out in an ASEA-SKF ladle furnace. The heats were uphill teemed to ingots. The compositions of the heats are shown in *Table 2*.

The majority of the fatigue failures in heat G1578 were caused by oxide inclusions, approximately 85% compared to only 10% in heat G1581. The remainder was in both cases titanium carbonitrides. The fatigue life expressed as L50 values were 6.4 and 4.0 for heat G1578 and G1581 respectively. Fromthe composition one would not expect a big difference between the two heats.

		Heat G 1578	Heat G 1581
C	weight%	1.04	1.01
Si		0.22	0.24
Mn		0.32	0.28
P		0.012	0.012
S		0.006	0.007
Cr		1.43	1.39
Ni		0.14	0.14
Mo		0.06	0.05
Cu		0.17	0.21
Al		0.028	0.030
Ti	ppm	12	11
O		6.0	5.0
N		100	118

Table 2. Chemical composition.

However, due to different steel processing parameters, an increased amount of titanium carbonitride stringers in heat G1581 decreased the fatigue life somewhat and switched the main initiation cause from oxides into carbonitrides, see example in *Figure 24*. The size of the titanium carbonitrides is much smaller compared to oxides. *Figure 24* shows the sizes of inclusions found in a large number of fatigue failures. This means that for the same size titanium carbonitrides are more hazardous compared to oxides.

Fig 24.
Titanium carbonitride causing a fatigue failure in heat G1581.

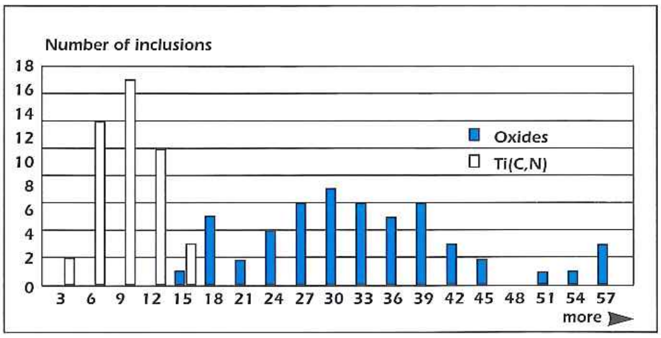


Fig 25. Size distribution of oxides and titanium carbonitrides initiating fatigue.

A theoretical calculation was made on these inclusion configurations [7]. Again the observed result was confirmed. It was shown that the reason why titanium carbonitrides are more hazardous was mainly due to the morphology of the inclusions. Effects of thermal shrinkage and Youngs modulus differences were minor.

Carbide clusters

One Vacuum Arc re-melted material was fatigue tested at a single stress level of 950 MPa. The composition of the steel is shown in *Table 3*.

		Heat RM
C	weight %	1.06
Si		0.20
Mn		0.31
P		0.006
S		0.0002
Cr		1.35
Ni		0.08
Mo		0.06
Cu		0.10
Al		0.030
Ti	ppm	25
O		4.3
N		34

Table 3. Chemical composition.

The majority of the fatigue failures were caused by carbides, approximately 90%. The remainder was titanium carbonitrides. A fracture surface is shown in *Figure* 26. An EDS analysis showed mainly a *Cr* peek with only traces of *Fe*. An analysis over a lager area showed the opposite. A closer examination of the fracture surfaces revealed a number of particle like areas at the initiation site.

The conclusions were therefore that the carbides or perhaps the carbide clusters in the structure had initiated the fatigue failures. The microstructure also showed heavily segregated areas with carbide clusters with an individual size larger than 20 µm, see *Figure* 27. The fatigue life expressed as a L50 value was only 0.02 million cycles.

Fig 26. Titanium carbonitride causing a fatigue failure in heat RM.

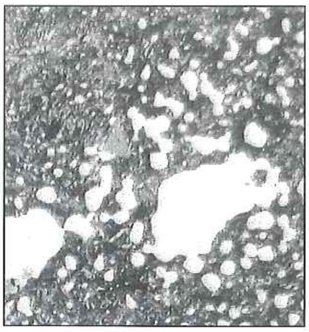


Fig 27. Carbide cluster (with a titanium carbonitride) in hardened sample of heat RM.

Conclusions

As steel making quality improves ever better methods to measure the content of internal defects must be developed.

Today's ladle furnace technology has reached a development level which meets or surpasses re-melting technology as regards internal cleanliness. New methods to assess the non-metallic inclusion content have been developed, and by combining a direct inclusion analysis based on OES technology with high frequency, high precision ultrasonic techniques the inclusion size distribution and inclusion composition can be actively steered on-line.

As regards fatigue initiation in hardened SAE 52100 ball bearing steel, it is evident that initiation can be caused by oxides, titanium carbonitrides or carbide clusters. Different oxide types may be more or less hazardous for fatigue loading, and for the same size titanium carbonitrides are more hazardous than oxides mainly due to morphological effects.

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