Advances in the Production and Use of STEEL with IMPROVED INTERNAL CLEANLINESS

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Foreword

This publication, *Advances in the Production and Use of Steel With Improved Internal Cleanliness*, contains papers presented at the symposium of the same name held in Atlanta, Georgia, on May 4, 1998. The Symposium was sponsored by ASTM Committee A-1 on Steel, Stainless Steel, and Related Alloys. The symposium chairman was John K. Mahaney, Jr., LTV Steel.

Contents

Overview

As ASTM Committee A-1 reached 100 years of service to industry, consumers, and the government, the Committee wanted to reflect on the status of a major source of problems in steel. From earliest times, the presence of nonmetallic inclusions has been a major source of problems and failures. Failures due to inclusions have been seen in major structures and boilers as well as the inability to successfully form material into usable shapes and parts. The Committee sought to determine the status of the state of the art of inclusion identification and prevention, as well as the relative status of different parts of the industry in the attempt to produce material with improved internal cleanliness.

The papers presented in this book cover areas from bearing steels to castings. The various authors clearly show that level of inclusion identification and control through processing improvements is greatly dependent upon the sector of the industry. The level of inclusions desired in bearings is several orders of magnitude from the majority of the casting industry. At the same time, manufacturing methods such as continuous casting and other tonnage industry methods are not available in the discrete steel and iron casting segments of the metal melting industry.

The papers in the Special Technical Publication show the state of the art in inclusion identification, prevention, and understanding of the deleterious effects of those inclusions. Products covered include bearing steels, high-strength plates, steel castings, stainless steel medical implants, and test methods to determine the presence and effect of nonmetallic inclusions in the steel products. The papers emphasize the effect on the products rather than manufacturing methods.

The authors of the papers in this publication include researchers and practitioners from the United States, Europe, and Asia. The companies and research institutions represented by those authors include The Timken Company, The University of Alabama at Birmingham, the Steel Founders Society of America, SKF Engineering & Research Center, Ovako Steel, Thermax Ltd., Synthes (USA), and Bethlehem-Lukens Plate Company.

Bearing Steels

The presence of even very small inclusions are clearly shown in the papers presented here to adversely affect performance in bearings. The problem is shortened life of the bearing. The related problems with bearing failure include major machine failures. One must be able to detect the presence of inclusions and then determine the source of such nonmetallics and develop methods to either prevent the formation of such materials, typically oxidation products, or if those deleterious materials are formed, proper treatment of the molten product to minimize or prevent the occurrence of inclusions in the product made from the molten metal, generally an ingot.

Within the bearing community, the emphasis on minimizing inclusions has been successful to the extent that the conventional methods of detection are no longer sufficient. The researchers recognize that one can not eliminate what one can neither detect nor identify. They further recognize the effects of extremely small inclusions on bearing life and thus the performance of the product.

However, one must first be able to determine the presence or absence of inclusions to understand the performance issue.

A method using ultrasonic methods to determine internal cleanliness of the material and then to relate such findings to bearing life is described in the paper by Eckel, Glaws, Wolfe, and Zorc of The Timken Company. Beswick, Gabelli, Ioamides, Tripp, and Voskamp presented information on bearing life models that take into account the hardness or strength of the bearing and the effects of the very fine inclusions ''micro-inclusions'' that are the result of today's production technology for bearing steels. Recognizing that production methods have to be understood and controlled in order to achieve the cleanliness levels necessary for the Beswick et al. model, the work by Lund and Ölund examine how the various steel making and processing operations can affect internal cleanliness and thus bearing life.

Stainless Steels

A major use of stainless steels has been for various implants in the human body to replace body parts damaged for some reason. The human body is not a particularly friendly place for a foreign object, such as an implant. Inclusions act as corrosion initiation sites and thus lead to rapid deterioration of the implant and thus the effectiveness of that medical device.

While oxides are the primary inclusion forms of concern in many sectors of the industry, the medical implant concerns also include manganese sulfides, carbides, delta ferrite, and other secondary phases as sources of corrosion cells and thus problems with implant life. Disegi and Zardiackas review developments in this area.

Steel Castings

While bearings and many other forms of steel can be handled in ways to avoid contact with air, continuous casting and bottom pouring for example, in the casting industry, numerous castings are by necessity poured in air from the ladle into the mold. This action, along with turbulence in the pouring stream and within the mold, as well as mold design, can lead to the trapping of large non-metallic inclusions at the surface of the casting. These inclusions then lead to machining failure during processing of the castings. Blair, Monroe, and Griffin review the numerous technique and processing modifications that have been studied in this segment of the industry to minimize what they term ''macroinclusions.'' The casting industry must adapt practices in use in other portions of the iron-and-steel-making business to their industry, which is very different in scale and manufacturing methods from the bearing, bar, and flat-rolled sectors of the industry. Developing an understanding of the causes of inclusion problems is fundamental to resolving the problems. Blair et al. present evidence of how the proper application of various techniques can significantly improve the product by preventing the very large inclusions.

Steel for Plates

Steel plates are a fundamental building block of American industry. The strength and load-bearing capabilities of structural steel plates are critical to the construction of everything from major office buildings to offshore oil platforms. Wilson describes how improved internal cleanliness has improved material toughness, increased ductility, and improved the fatigue life of structures. He also notes the interactions that can take place between and among production variables. Such interactions can affect the reliability of inclusion control during steelmaking and must be understood and controlled to prevent the development of less than desired properties in the finished product.

Steel plates are also used for pressure vessel applications, and many heads for such vessels are formed using spinning techniques in the heavier thicknesses. The development of lamellar separations at mid-thickness is not desirable. Dutta, Chandawale, and Vanchinath have developed a test method to determine the tendency of materials to crack internally during spin forming. They emphasize the importance of low levels of chemical segregation and lowering sulfur content as ways of improving the performance of materials when spun into tank heads.

Conclusions

Steel cleanliness means many things to many people. The levels of internal cleanliness or freedom from inclusions achieved by the bearing industry are clearly well beyond that associated with castings. On the other hand, the manufacturing methods available to the casting industry make the application of bearing-type manufacturing techniques very difficult, and other manners of inclusion prevention and control must be developed.

Improvements in performance of materials are the result of continuing efforts to understand why failures and problems occur. The bearing industry has greatly improved bearing life through the control of internal cleanliness and seeks continued life spans for the bearings. The casting industry is achieving reductions in material and time losses due to inclusion problems. The absence of such large inclusions also has to improve the life of those castings, especially in fatigue situations. A better understanding of inclusions has provided medical implants with improved corrosion resistance and thus better life. Buildings and pipelines with improved performance, and thus far fewer failures and other problems, have resulted from improved internal cleanliness in steel plates for structural and pressure vessel applications.

The need to continually improve the products used by all of us means that the drives to improve internal cleanliness are only beginning. The presence of any nonmetallic particle in the steel matrix can be the initiation site for failure. Our efforts to minimize such problems have resulted in great improvements in bearings, structural steels, and medical implants, to mention a few products. However, further improvements will come but only with a firm understanding of where we are today. The papers contained in this Special Technical Publication provide an excellent base for such understanding and for future improvements in the products we all use.

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Symposium Chairman and Editor

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CLEAN ENGINEERED STEELS - PROGRESS AT THE END OF THE TWENTIETH CENTURY

REFERENCE: Eckel, J. A., Glaws, P. C., Wolfe J. O., and Zorc, B. J., **"Clean Engineered Steels—Progress at the End of the Twentieth Century,"** *Advances in the Production and Use of Steel with Improved Internal Cleanliness, ASTM STP 1361*, J.K. Mahoney, Jr., Ed., American Society for Testing and Materials, West Conshohocken, PA, 1999.

ABSTRACT: The Timken Company, a manufacturer of alloy steel and bearings, has developed a 15 MHz ultrasonic inspection method that correlates steel cleanness to bearing fatigue performance. It is used to qualify worldwide bearing steel suppliers for cleanness requirements, to monitor their compliance and qualify process changes. This method has permitted the appropriate steel cleanness to be selected for bearing applications. Through Continuous Improvement (CI) methodology, steelmaking productivity advancements have occurred along with advancement in steel cleanness. These efforts have led to 4 orders of magnitude steel cleanness improvement, and nearly 20 times bearing performance improvement over the past 15 years.

KEYWORDS: ultrasonic testing, clean bearing steel, steelmaking, steel cleanness, fatigue life test

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Not only has the quality of steel improved dramatically over the past 100 years but the definition of steel quality has also improved. Chemistry content used to be the chief requirement, but now total quality can embrace chemistry, size tolerances, surface conditions, hardenability, delivery performance and steel cleanness as well as customer specifications. But a necessary component to any quality requirement is a reliable measurement. When it comes to steel cleanness, the steel industry has been frustrated for many years with the lack of a reliable cleanness measurement. Standard test methods,

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such as JK inclusion ratings and magnaflux testing, are inherently challenged by small sampling volumes, sampling location selections and insensitivity to very clean steel. Recent work concerning ultrasonic testing of material from over twenty steel companies throughout the world has shown considerable variation. These results show why the high resolution test is so valuable in clean steel evaluation.

Since rolling element bearings are one of the most demanding mechanical devices that challenge steel cleanness, advance clean steel production has been a major focus at

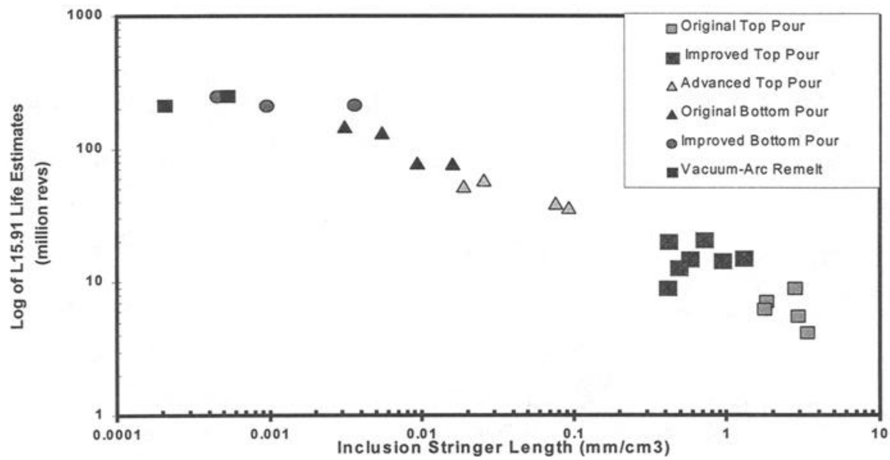

FIG. 1—*Bearing Fatigue life improves with reduced inclusion length.*

The Timken Company. During the past 15 years, dedication through investment and process improvements have shown that air melted steel cleanness can approach vacuum arc remelted steel. The key measurement tools used were bearing fatigue testing and an ultrasonic inspection that correlated with bearing performance. The correlation is shown in Figure 1 for 21 cm diameter bearing cone. All points on this graph were caused by inclusion mode of fatigue.

Measurement Methods

Many methods have been used over the years to measure the cleanness of steel. Early tests employed the visual examination for inclusions on a fractured surface. Later developments involved macro etching in acid to detect segregation or inclusions. Microscopic and chemical methods were developed to monitor the number and size of inclusion stringers. By knowing the chemistry and locations of inclusions, process changes could be implemented to prevent formation or preclude inclusions from being trapped during the solidification in the steel manufacturing process. Today even more sophisticated techniques utilizing Scanning Electron Microscopy (SEM), Energy

Dispersive Spectrometry (EDS), image analysis and ultrasonic testing method are assisting the manufacture of near zero levels of inclusion contents.

The primary objective at Timken Research has been to look at sufficient volumes of material to assure the elimination of harmful inclusions that are possible sites for fatigue failures[1]. The ultrasonic test has proven to be the biggest asset in evaluating steel cleanness and monitoring process improvements. Previous publications have documented the techniques and the correlation of ultrasonically detected inclusions to bearing fatigue life[2-5].

One critical factor in the evaluation process is the test specimen size compared to the original cross section, i.e., reduction ratio, because this affects how the inclusion stringers are lengthened during the hot reduction. It has been shown that extensive hot working can elongate or string out and then break up detrimental inclusion stringers[6]. The inclusions become individual particles and become less reflective ultrasonically. Therefore, the test specimen size and cleanness criteria must be considered when comparing material from different cast section sizes. The reduction ratio is also a significant factor to consider when using other techniques such as microscopic inclusion ratings.

When evaluating a steel process, it has proven beneficial to maintain the identity of sample location during the manufacturing process from ingot to billet to tube to ring specimen. An overview of the processing of ring specimens from an ingot is diagrammed in Figure 2. This specimen identity facilitates the characterization of size and spatial distribution of inclusions within an ingot or bloom.

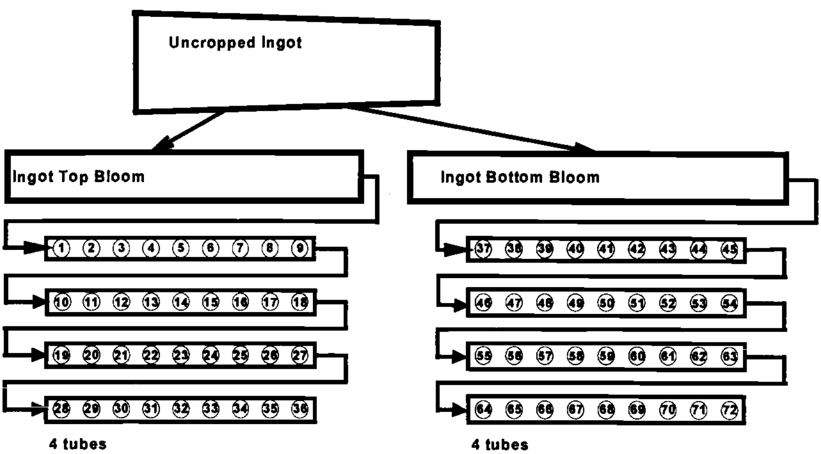

FIG. 2--*Manufacturing process for ingot evaluation ring specimens.*

The Timken ultrasonic unit used today is shown in Figure 3. It was purchased in 1997 to our specification and is the fourth generation of high resolution ultrasonic test systems. The original Timken design was developed in the early 1970's and the scanning units have been replaced about every seven years to employ the latest electronic and data collection advancements.

Our standard ultrasonic test employs a programmable coordinate measurement system to index a 15 MHz transducer along the test specimen surface as the specimen is rotated on a turntable. The transducer is focused from 2.3 to 4.6 mm under the surface. Figure 4 shows the volume inspected in our standard test specimen. The part geometry has given excellent surface to center volume inspection. The inspected surface must have a finish of 30 micro-inches or less. The standard ring specimen evaluation examines about 69,000 mm3 of material in about 4 minutes.

A typical specimen scan generates a 0.75 megabyte file. An analysis program calculates the statistics of number and length of detected indications per part from the raw scan data. This part data is transferred to a data base that permits cataloguing of statistics. Over 1500 ultrasonic steel cleanness studies have been performed from over 25 steelmaking plants.

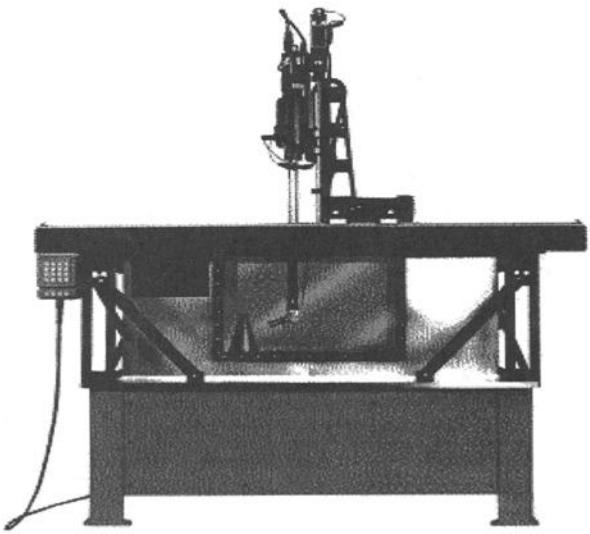

FIG. 3--*Ultrasonic inspection system immersion tank.*

Indication length and number of indications are counted within the gated volume for each test specimen. Indications on consecutive scan lines that exceed the set threshold are linked together to form a contiguous indication. The number of contiguous indications and their length are totaled. Generally, steel cleanness results are reported by total indicated length divided by inspection volume. Some supplier's cleanness improvements have nearly eliminated the large indications and only occasional single length indications are found. The search for the next ultrasonic tool enhancement to differentiate very clean steel is underway.

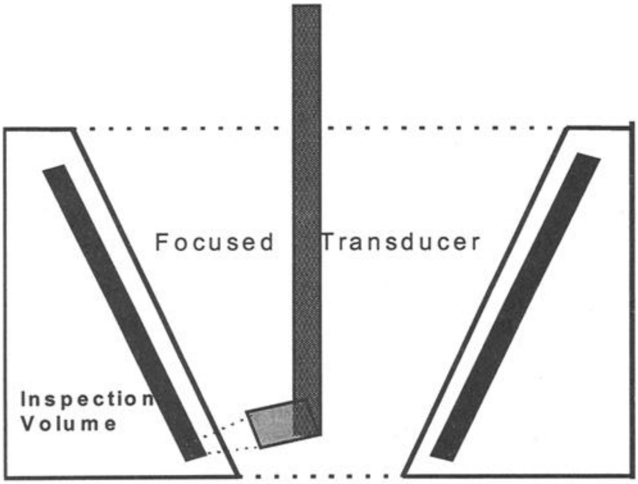

FIG. 4--*Ultrasonic test volume.*

Advantage of the Ultrasonic Test System

The primary benefit gained with the use of the ultrasonic technique is the significant sample volume inspected. To put this into perspective, the inspected volume of a single ring specimen is approximately 70,000 mm3. As shown in Figure 5, this is more than 50 times greater than the volume of one step down magnetic particle sample, more than 250 times greater than a one gram sample used for oxygen analysis and more than 11,000 times greater than a typical JK micro specimen. This increased volume provides far greater certainty in statistical comparison of steel cleanliness data as well as correlation with fatigue (Fig. 1).

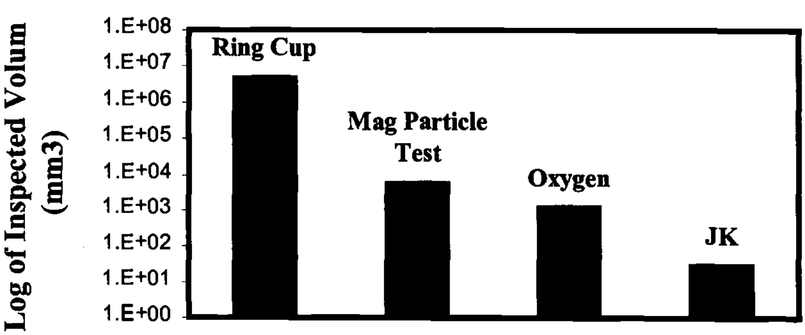

FIG. 5--*Volume of steel inspected per cleanness testing method.*

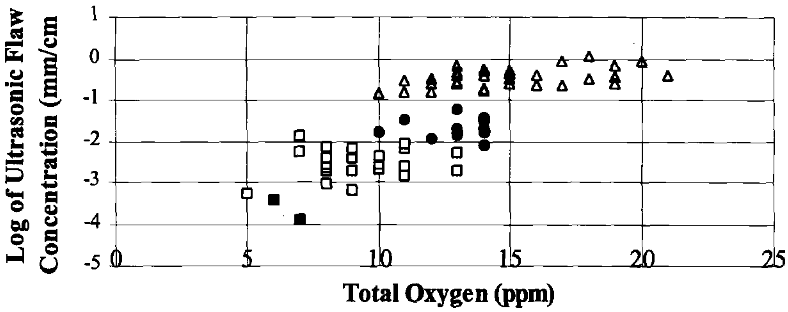

FIG. 6--*Inclusion length versus oxygen content.*

The volume advantage is clearly illustrated for the case of total oxygen measurements, perhaps the most common employed and often cited means of steel cleanness evaluation. Previous studies have shown that while total oxygen measurements roughly correlate to ultrasonically measured inclusion stringer length, the variance of the correlation is significant [2]. As shown in Figure 6, the range in total stringer length associated with a given total oxygen value exceeds two orders of magnitude. More importantly, comparisons of Figures 1 and 7 show that bearing fatigue performance correlates significantly better with ultrasonic stringer length than with total oxygen content, especially in low oxygen containing steels. Accordingly, total oxygen contents should not be used for accurate prediction of either inclusion stringer length or the corresponding fatigue life.

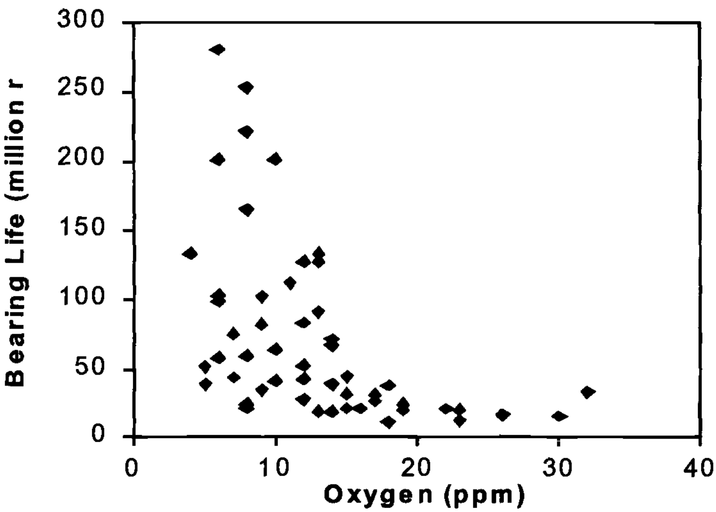

FIG. 7--*Oxygen content versus bearing life.*

Another important advantage of the ultrasonic technique is the ease and accuracy with which the detectable inclusion distribution within a given sample may be quantitatively determined. This is possible through the use of sophisticated analytical and windowing capabilities of the inspection system and the near-surface to near-center continuum inherent in the design of the specimen geometries employed. For example, this equipment has been used to quantify the inclusion concentration differences within the inner radius region of a continuously cast bloom (curved machine) with the balance of the bloom cross section under a variety of casting conditions[7].

Under standard ultrasonic test conditions, the vast majority of indications have been shown to be oxide type inclusions[3,7]. In addition, EDS may be used in conjunction with ultrasonic inspection to determine the composition of large inclusions. This methodology, including the use of tracer materials introduced to the steelmaking processes, has provided an effective means of identifying the primary source of inclusions in many cases. Once the inclusion source has been identified, steel cleanness improvement efforts can focus on eliminating or minimizing the source. We refer to this as "Forensic Metallurgy," since it requires detective work to solve the mystery of what caused the inclusion.

High Carbon Steels Versus Low Carbon Steels

The bearing industry has debated the value of case carburized steels by citing the theory that high carbon steels are cleaner based on the thermodynamically lower oxygen potential in undeoxidized liquid steels of higher carbon grades. While that argument is theoretically valid, practical experience has shown otherwise[5]. With modern steelmaking practices there is no difference between low and high carbon steel cleanness. Figure 8 compares ultrasonic cleanness of high and low carbon steel grades from three steel plants. All three plants used ladle refined and bottom poured ingot practices. Data from Plant A and Plant B demonstrate that low carbon steel can be equal to or slightly cleaner than high carbon steel.

Additional requirements for clean steel include manufacturing with detailed metallurgical knowledge of process parameter interactions and establishing procedures for process stability. The ultrasonic test results along with the detailed metallurgical analysis create a continuous improvement feedback path.

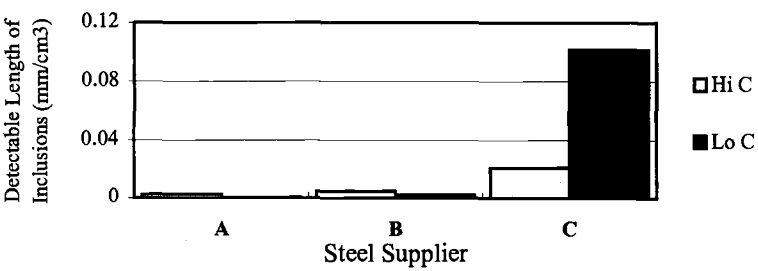

FIG. 8--*High carbon versus low carbon steel cleanness.*

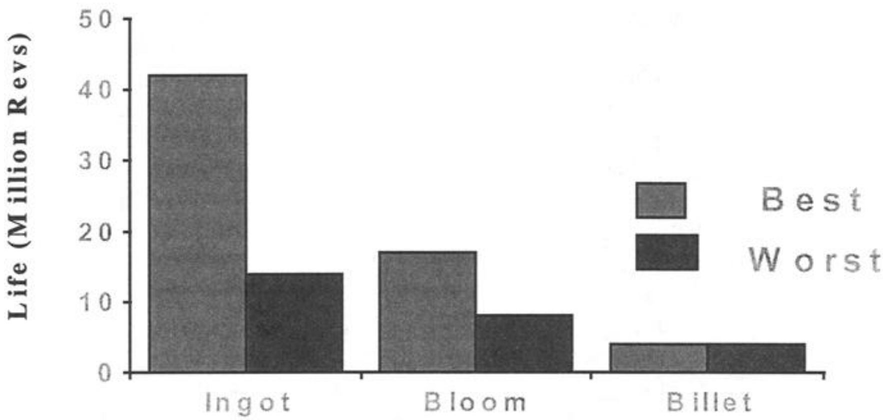

FIG. 9--*Casting method versus bearing life.*

Ingot Cast versus Continuous Cast

Another illustration of the range of bearing steel quality is shown in Figure 9, which graphs the best and worst fatigue life tests of ingot, bloom and billet casting methods which are classified by mold cross section[8]. The differences in original cast cross sectional area would contribute to but do not explain the range of fatigue values that were found, especially when looking at the ranges of life of one cast method. These differences are the results of the wide differences in process control and knowledge among the world wide bearing steelmakers. All samples in this plot met the requirements of ASTM Standard Specification for High-Carbon Anti-Friction Bearing Steel (A295) or ASTM Standard Specification for Carburizing Steels for Anti-Friction Bearings (A534). It is clear that differences in original cross sectional area can have a significant effect on steel cleanness and bearing life performance. This also supports the benefits of ingot casting processing versus the continuous casting route for demanding applications. The use of ultrasonic inspection has allowed the appropriate quality to be applied to the bearing application.

Continuous Improvement

In the early 1980's the Faircrest Steel Plant was conceived with two goals: to be world leading in steel production efficiency and quality. Initial studies of world steelmakers looked at equipment that would meet both goals. The ultrasonic test method was used to benchmark steel cleanness and compare equipment and processes and ultimately to guide the final equipment selection and start up processes. Surprisingly, the two goals for the plant have proven to be compatible, as seen in Figure 10. The plant was design to produce 500,000 tons per year, but finished 1997 at 870,000 tons. In addition to increasing output each year, many other operating efficiencies have been realized. The metallurgical measures of quality, like chemistry control and steel cleanness standards have not been compromised and in many cases have steadily improved. The ultrasonic tool has been an essential part of the continuous improvement methodology at the plant from the beginning. Over 30 process improvements have been verified with ultrasonic testing. Over 100 evaluations have been conducted to assure process stability.

For example, numerous bottom pour shrouding designs have been evaluated. Figure 11 shows the results of one such study. Attempts at monitoring the shroud oxygen content and correlating it with product oxygen were frustrated since the full range of total oxygen values was limited and significant overlap of data sets exited. There was a large uncertainty associated with the use of total oxygen measurements alone. Employing the statistically more significant ultrasonic method provided an improved level of confidence in the conclusions and a stable argon shrouding practice has been established.

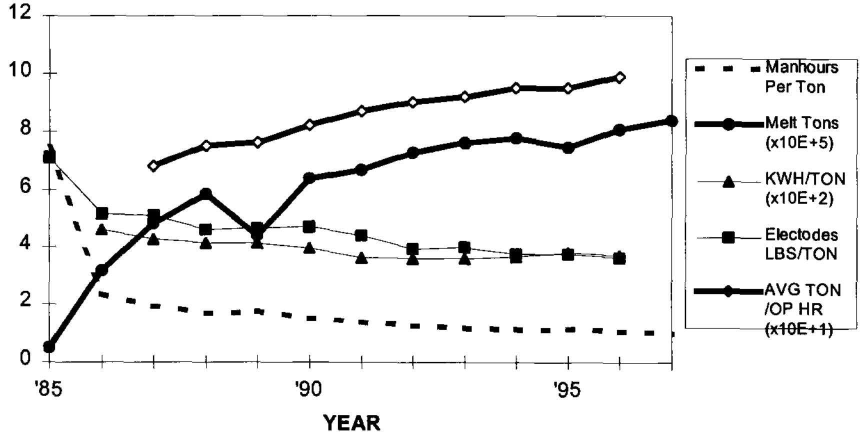

FIG. 10--*Faircrest steel plant productivity.*

The ultrasonic technique has been accepted by the operations with a high degree of confidence. It takes the uncertainty out of process changes. Cost saving or operational efficiency proposals that can affect steel cleanness are evaluated before implementation. Examples of these investigations include:

- ladle stirring methods
- bottom pour mold flux chemistry
- mold flux quantity
- mold flux supplier qualifications
- hot top system evaluations
- refractory suppliers and composition
- residual chemistry elements

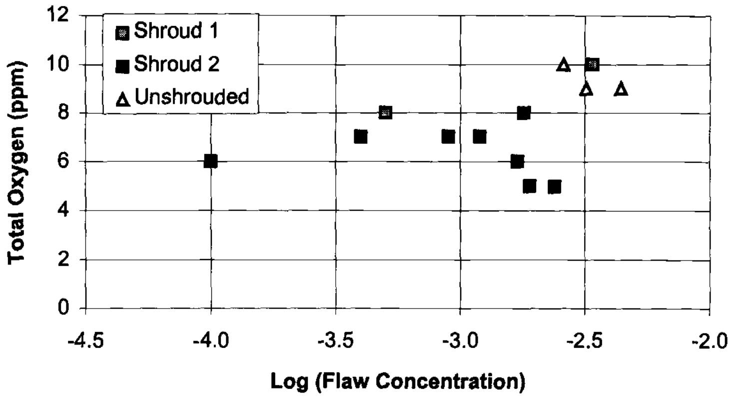

FIG. 11--*Flaw concentration versus oxygen content.*

The ultrasonic technique helped quantify and expand the metallurgical understanding of the steelmaking processes. The following issues have been evaluated:

- optimum teeming temperatures
- final aluminum levels
- mold wear effects
- nozzle bore fill
- finish pour off butt size
- oxygen assisted nozzle opening
- shrouding

Conclusions

In the continuous pursuit of superior quality, a high resolution ultrasonic test has been developed as a means to monitor steel cleanness improvements for over 20 years. The data supports the strong correlation between ultrasonically measured inclusion length and fatigue performance. The methodology has been used as an evaluation tool for both steel audits and continuous improvement projects. This tool has been used to match the steel quality to the fatigue requirements of the application. Through this work, many significant and sometimes unexpected findings have been realized:

- High carbon bearing steels are not inherently cleaner than carburizing grades.
- Similar steelmaking equipment does not guarantee equivalent steel cleanness.
- Equipment and process knowledge and control determine steel cleanness.

As we glimpse into the 21st century, we can see competition from other materials, but no substitute for clean steel to give excellent strength and fatigue resistance for highly stressed parts. Bearings, crankshafts, gears, suspension parts and transmissions have shown improved performance by using clean steel.

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John Beswick[1], *Antonio Gabelli*[2], *Stathis Ioannides*[3], *John H. Tripp*[4] *and Aat P. Voskamp*[2]

ROLLING BEARING LIFE MODELS AND STEEL INTERNAL CLEANLINESS

REFERENCE: Beswick, J., Gabelli, A., Ioannides, E., Tripp, J. H., Voskamp, A.P., **"Rolling Bearing Life Models and Steel Internal Cleanliness,"** *Advances in the Production and Use of Steel with Improved Internal Cleanliness, ASTM STP 1361*, J.K. Mahaney, Jr. Ed., American Society for Testing and Materials, West Conshohocken, PA 1999.

ABSTRACT: The most widely used steel grade for rolling bearings is based on a steel composition first used almost a hundred years ago, the so-called 1C-1.5Cr steel. This steel is used either in a selective surface induction hardened condition or in a through hardened heat treated condition, both yielding exceptional structural and contact fatigue properties. The Lundberg and Palmgren rolling bearing life prediction model, published in 1947, was the first analytical approach to bearing performance prediction, subsequently becoming a widely accepted basis for rolling bearing life calculations. At that time the fatigue life of rolling bearings was dominated by the classical sub-surface initiated failure mode. This mode results from the accumulation of micro-plastic strain at the depth of maximum Hertzian stress and is accelerated by the stress concentrations occurring at the micro internal defects. In common with all fatigue processes, rolling bearing failure is a statistical process: the failures of bearings with high inclusion content tested at high stress levels belong to the well-known family of "Weibull" distributions. Steady improvements in bearing steel cleanliness due, amongst other things, to the introduction of secondary metallurgy steel making techniques, have resulted in a significantly increased rolling bearing life and load carrying capacity. In recognition of this, in 1985 Ioannides and Harris introduced a new fatigue life model for rolling bearings, comprising a more widely applicable approach to the modelling of bearing life based on the relevant failure mode. Subsequently this has been extended to include effects of hardness and of micro-inclusion distributions in state-of-the-art clean bearing steel.

KEYWORDS: 1C-1.5Cr, 52100 bearing steel, progress in steel making, structural fatigue, Dang-Van fatigue criteria, internal cleanliness, micro inclusions, Lundberg and Palmgren, Ioannides-Harris, SKF Life Theory

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[1]Programme Manager Steels Technology, [2]Senior Engineer, [3]Director of Product Research, [4]Consulting Engineer, SKF Engineering & Research Centre, Postbus 2350, Nieuwegein, The Netherlands.

Introduction

The majority of through hardened bearings are produced from steels with compositions of nominally 1% carbon and 1.5% chromium, the so-called "1C-1.5Cr bearing steels". The origin of this bearing steel composition goes back almost a century and literature references of this composition from the early 1900's are available [1, 2], see Table 1. The 1C-1.5Cr steel is commonly known as ASTM A295 52100, 100Cr6, SUJ2, EN31 and SKF 3 steel but world-wide is also known under various other designations, see Table 2; many of these have minor composition variations as compared with ASTM A295 52100.

Table 1: Examples of early through hardening rolling bearing steels

	Prof. Stribeck 1903 to 1908, Berlin, German [1]	1905 Fichel & Sachs, Schweinfurt	Hess-Bright, Germany [2]	Societe Francaise des Roulement, France [2]
C	0.80 to 1.08	1.08	0.94 - 0.89	0.80 - 0.89
Si	0.20 to 0.37	0.28	0.2	0.19 - 0.20
Mn	0.30 to 0.37	0.37	0.15 - 0.43	0.19 - 0.20
Cr	1.00 to 1.10	1.58	1.21 - 1.24	1.17 - 1.60
Ni	0.04	-	none	none
Cu	0.02 to 0.04	0.03	-	-
P	0.016 to 0.018	0.02	0.015 - 0.022	0.019 - 0.022
S	0.008 to 0.014	0.02	0.034 - 0.039	0.03

Table 2: Various designations for 1C-1.5Cr rolling bearing steels

Country	Designation
US	ASTM A295 52100
UK	BS 970 543 A99, EN31
Germany	100Cr6 (DIN 17230)
France & Italy	100C6
Sweden	SIS 142258
Japan	SUJ2
Russia	SHKh15
Czech & Slovakia	14100
Rumania	RUL 1
Hungary	GO 2
Former Yugoslavia	C 4146
Bulgaria	7838 Ch
Brazil	VC-52
China	GCr15
Korea	SU2
ISO	683-XVII

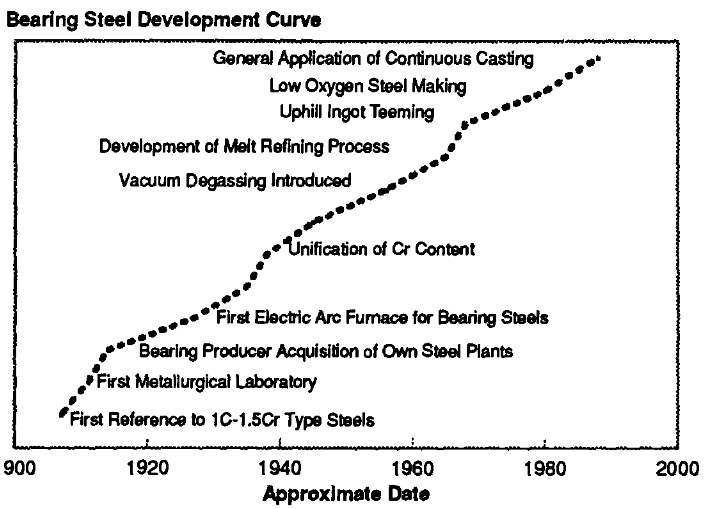
The steel composition most prevalent today, with the composition consistency obtainable in modern secondary metallurgical processes, is nominally closer to 1%C-1.4%Cr. Attempts over the last six years to specify the composition in an ISO standard have so far failed to produce complete agreement on the carbon range but progress is being made. In the interests of unification the ASTM A01-28 subcommittee has agreed to a change of the A295 52100 minimum carbon content from the current 0.98 wt.% to 0.93wt.% but the producers and users of the reduced-cost, billet cast process for the 1C-1.5Cr steel are strongly lobbying for the 0.90 wt.% minimum consistent with the German DIN 17230 100Cr6 specification.

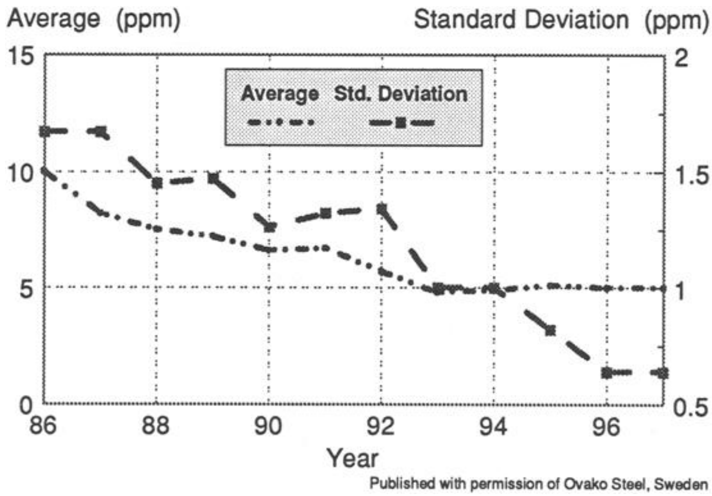
The 1C-1.5Cr type rolling bearing steel grade is manufactured in quantities of around 2 million tons per year and this amount will of course grow with the increased usage of rolling bearings in automobiles, household appliances etc. Considering that metallurgy was recognised as being important in the rolling bearing industry about a century ago, a paper on "Rolling Bearing Life Models and Steel Internal Cleanliness" is very appropriate for the ASTM centennial celebration.

The developments of bearing metallurgy and the introduction of steel refining processes are summarised in Figure 1, where the development of secondary metallurgy techniques for rolling bearing steels in the 1970s significantly reduced the number of failures related to materials' quality. The steel process developments for the 1C-1.5Cr rolling bearing steels are given in more detail in the paper from the SKF Steel Division, Ovako Steel, in these conference proceedings [3].

The steel quality development curve from the Ovako Steel plant, as measured by oxygen content, is characterised by two lines in Figure 2. The low average magnitude of the oxygen content of a heat is important but also the progressive reduction in its standard deviation between heats is a remarkable indicator of improved process quality consistency.

Figure 1: Through hardening rolling bearing steel development events





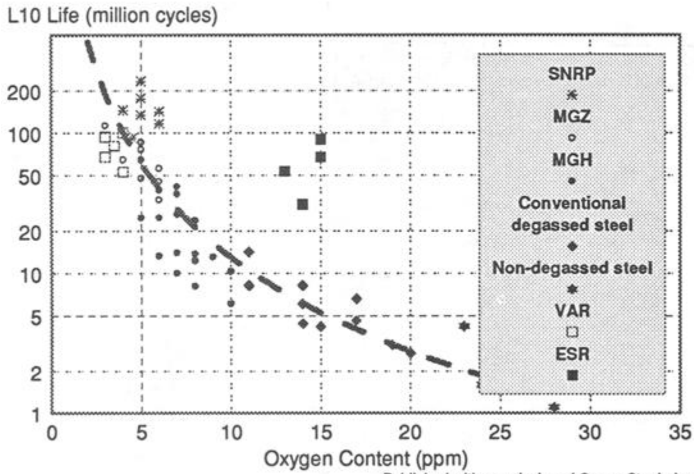
From these plots it follows that more than 90% of the 1C-1.5Cr bearing steel heats currently have oxygen contents of 6 ppm or less.

The internal cleanliness or metallurgical quality of modern bearing steels is characterised by macro and /or micro non-metallic inclusions and by titanium nitro carbides. Metallurgical quality may also be characterised by the total oxygen content, although the determination of such very low oxygen contents requires special care and instrument calibration. A correlation between oxygen content and rolling contact fatigue (RCF) strength has been published numerous times by steel and rolling bearing producers using so-called "accelerated" element tests (thrust washer testing). Figure 3 gives an example of the influence of oxygen content on RCF life, published with the permission of Sanyo Steel, Himeji, Japan.

Figure 3 shows the scatter in L_{10} values commonly found in such accelerated tests, a result of the statistical nature of bearing failures initiated by inclusion distributions. As can be seen, the relationship between steel making, steel quality and bearing life is a well researched and extensively reported topic [4-8]. Indeed, bearing metallurgy was for many years pre-occupied with extending bearing life by making improvements in steel quality. As life became ever more extended, the literature began to be dominated by tests run at stress levels elevated sufficiently to produce conveniently short measurable lives, although the relevance of these high stress level tests to engineering applications of bearings is of course questionable.

For example, the stress level applied in thrust washer testing shown in Figure 3 gives a bearing dynamic capacity (C) and equivalent load (P) ratio C/P less than 2, when in fact normal applications for deep groove ball bearings generally yield values of the order of C/P 10 or even more: i.e. the thrust washer tests are performed at stress levels 1.71 times higher than typical engineering applications of rolling bearings.

Figure 3: Steel processes and 4.9 GPa rolling contact fatigue life testing



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This paper reviews the material requirements for modern bearing applications and the possibilities for meeting them through the improvement of steel cleanliness. Figure 4 illustrates eight typical situations in rolling bearing operation, highlighting for each the type of rolling contact and the critical design factor involved.

The challenge for the design engineer has increased as bearings have become structural load carrying "machine elements", e.g. flanged bearing units in automotive wheel bearings. A methodology for structural fatigue life prediction in rolling bearings, SKF Life Theory, which models the stress distributions as influenced by internal cleanliness effects and introduces a fatigue criterion, such as proposed by Dang Van [9], is described briefly. The precision this model brings to rolling bearing life prediction is demonstrated by comparison with experimental data.

Structural Fatigue and Internal Cleanliness Effects in Soft Annealed 1C-1.5Cr Steels

The vast majority of automotive wheel bearings are angular contact ball bearing designs and many of these are of the integrated flanged type, in which the rings can be bolted to the steering knuckle, brake disc or wheel, thereby supporting both the bearing and the rotating bending structural loads.

Metallurgical design considerations for the outer ring of so-called "type 2 hub (HUB)" bearing units are summarised in Figure 5. This schematic drawing of the cross section with outer ring and balls shows some of the parameters which the designer must consider, not the least important of these being the rotating bearing loads in the flange. It is therefore clear that a high structural strength, in particular a high rotating bending fatigue strength, is required in the HUB steels.

Figure 4: Schematic drawing of rolling bearing material design considerations

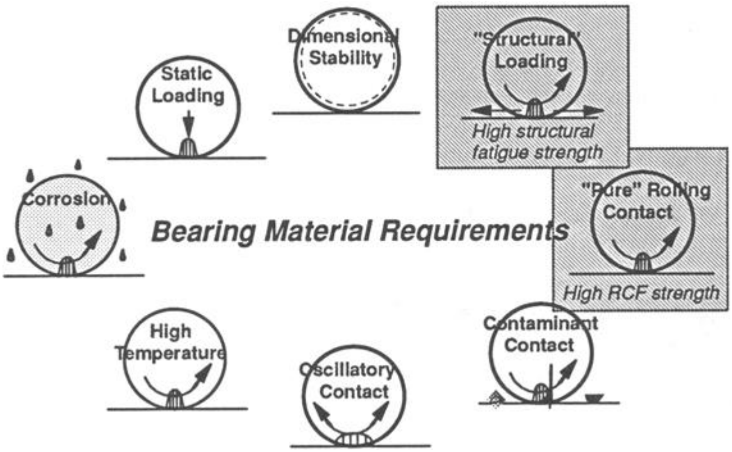
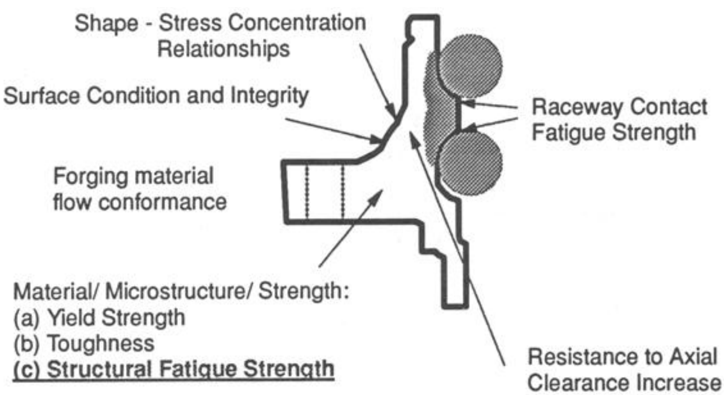
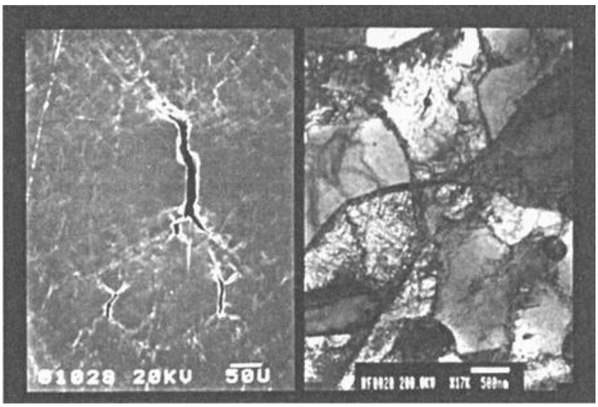


Figure 5: Design parameters for flanged HUB bearing rings



For the HUB application, 1C-1.5Cr steel is used in the soft annealed or quenched and tempered conditions to support the rotating bending structural loads, while the bearing loads are supported by a selective surface induction hardened layer. The flange region, supporting the structural loads, is soft annealed and the microstructure is ferrite with typically of the order of 15 volume percentage of spheroidal cementite. Bending fatigue in soft annealed 1C-1.5Cr takes place by a "classic cell dislocation and persistent slip band" mechanism, as may be seen by reference to Figure 6(a), a SEM photograph of a polished rotating beam specimen surface and 6(b) a transmission electron micrograph from the sub-surface.

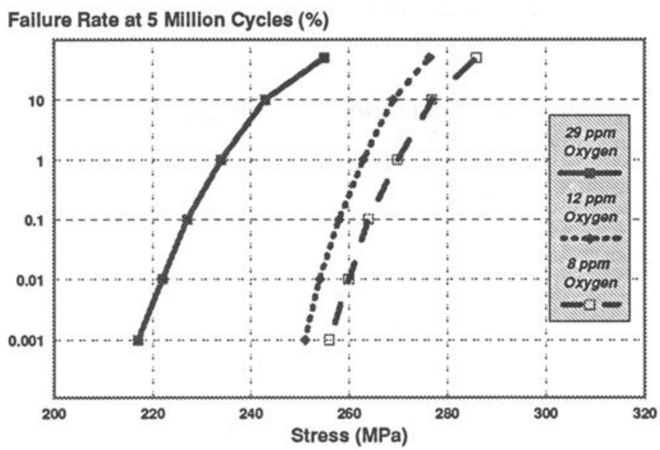
Figure 6: a) SEM photograph of rotating beam specimen surface
b) TEM micrograph from sub-surface



Rotating bending fatigue strength tests on soft annealed 1C-1.5Cr steel with varying levels of total oxygen have shown a measurably increased fatigue strength through reduction of the total oxygen content, see Figure 7. However, before these results can be applied in a design specification, the fatigue strength must be correlated with the matrix fatigue strength, as determined by the carbide size and also by the related ferrite grain size developed during the prior hot forging and applied soft annealing heat treatments.

The multiaxial loading on a HUB requires the application of a new type of fatigue limit criterion such as proposed by Dang Van [9]. Some details of this method in HUB bearing applications are given in the classic paper by Dang Van, Griveau and Message [10]. To summarise their work, such a criterion necessitates structural fatigue test data from rotating bending, push-pull and torsion testing. From these tests a 2-dimensional endurance domain may be constructed in the shear

Figure 7: Five million cycle fatigue strength for soft annealed 1C-1.5Cr steels



stress - hydrostatic pressure plane. If, during a stress cycle, the loading path of a volume element of the material remains within this domain, then that element will not fail by fatigue for a pre-defined life cycle time. By the use of steels with reduced oxygen content, and thereby improved internal cleanliness, the magnitude of this "safe domain" can be increased, thereby allowing a weight reduction in the wheel bearing and a consequent improved fuel economy.

Structural Fatigue And Internal Cleanliness Effects In Martensitic Hardened 1C-1.5Cr Steels

The through cracking resistance of 1C-1.5Cr rolling bearing steels is becoming more of an issue as demands on weight reduction and rotational speeds increase. Structural loads may arise from:

- Hoop stress, due to increased interference fits in combination with ring thickness reduction;
- High rotational speeds, resulting in high centrifugal hoop stresses;
- Reduced outer ring support, resulting in bearing ring bending loads;
- External vibratory loads superimposed on the rolling bearing loads.

In order to test at cycle times typically encountered in rolling bearings, it is necessary to develop ultra high cycle structural fatigue testing technology. As an example, results of 25 kHz strain-controlled push-pull testing, up to 0.5 billion cycles, are shown in Figure 8.

The stress distribution in push-pull is nominally uniform across the specimen section but, nevertheless, as seen in Figure 9, failures tend to be concentrated toward the outer regions, apart from an indication of a narrow peak in the centre segregated regions of the test bars.

Figure 8: High cycle push-pull S-N curve for martensitic hardened "high quality" 1C-1.5Cr steel

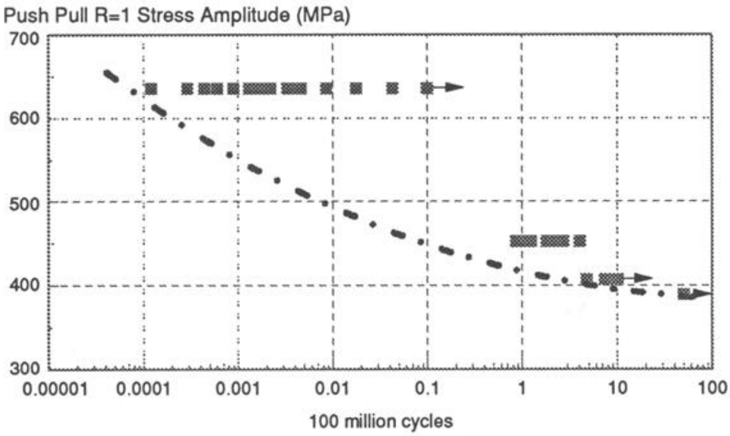
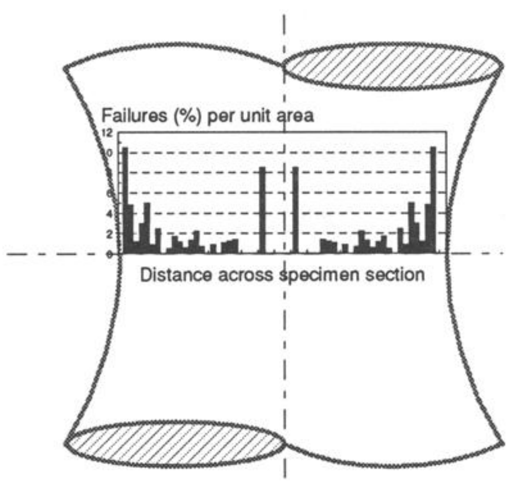


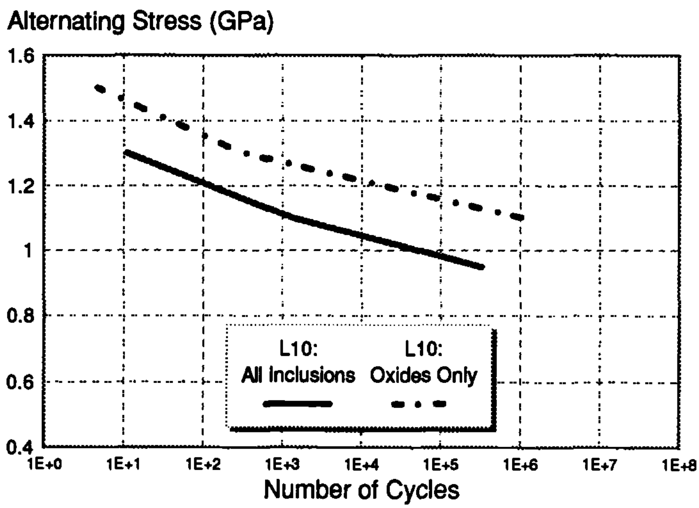
Figure 9: Failure initiation distribution profile for high cycle fatigue push-pull testing of martensitic hardened 1C-1.5Cr bearing steels



In common with rotating bending fatigue testing, push-pull fatigue testing is dominated by fatigue initiation at titanium carbo nitride internal contaminants. The beneficial influence of reduced titanium content in rolling bearings steels has been reported in detail and a previous paper [11] has dealt with the modelling of these effects.

Figure 10 shows the results of such model calculations and illustrates the increased resistance to through fatigue cracking resulting from improved internal cleanliness. Structural fatigue initiation occurs principally at TiC/N internal contaminants when low oxygen steels are tested.

Figure 10: Rotating beam S-N curve - effect of inclusion types



The limited plane strain fracture toughness of through hardened 1C-1.5Cr rolling bearing steels means that the structural fatigue life is determined by the crack initiation time. In the propagation phase, the "stable" fatigue crack growth rate is relatively rapid and, from the previous work, known to be independent of internal cleanliness effects [12].

Rolling Bearing Life Calculations

The design and dimensioning of a rolling bearing requires essentially "ad hoc" engineering calculations for several reasons, including the high stress sensitivity of the life equation, the complexity of the kinematics and the mainly compressive type of stress developed in Hertzian contact. Considerable effort has been put into modelling bearing performance in increasingly realistic terms as opposed to testing at unrealistically high load levels - as already noted - under laboratory "clean" lubrication conditions. Bearings are selected (type and size) such that the percentage (n) of failures in an application within a prescribed number of revolutions L_{na} i.e. the expected life of the bearing, is known. A number of conditions influence the bearing life L_{na} such as surface dents, steel quality, internal stress, etc. and an increasing number of these parameters can now be taken into account in the bearing life prediction models for L_{na}. The major breakthrough in engineering design of rolling bearings came more than 50 years ago, in 1947 and 1952, when Gustaf Lundberg and Arvid Palmgren applied Weibull's probability theory of fatigue to the calculation of rolling bearing life [13-14].

The Lundberg and Palmgren bearing life prediction model was based on the following equation:

$$\ln\left(\frac{1}{S}\right) \approx N^{e} \frac{\tau_o^c}{z_o^h} a z_o l \tag{1}$$

where:

S = Percentage of bearing population surviving
N = Number of cycles survived
e = Weibull exponent (9/8)
τ_o = Maximum orthogonal shear stress amplitude in Hertzian contact [Pa]
z_o = Depth of maximum orthogonal shear stress in Hertzian contact [m]
a = Contact semi-axis in transverse direction [m]
l = Raceway length [m]
c = Lundberg - Palmgren stress exponent (31/3)
h = Lundberg - Palmgren depth-weighting exponent (7/3)

By substitution of τ_o, z_o and a in terms of the applied bearing loads for a defined geometry, the conveniently simple and familiar load-life relationship for rolling bearings is obtained:

$$L_{10} = \left(\frac{C}{P}\right)^p \tag{2}$$

L = No. of cycles survived by (100 - n)% = 90% of all bearings [Mrevs]
C = Bearing dynamic capacity related to geometry and material, [N]
P = Equivalent load, [N]
p = Exponent: equal to 3 for ball bearings and 10/3 for roller bearings.

In this equation, the survival percentage (probability), taken as S = 90 = (100 - n), is contained in the value of C. This classical Lundberg-Palmgren equation provided the first opportunity to specify the capacity and to define or "guarantee" the performance of rolling bearings. In 1962 this equation was adopted by ISO 281 (1991) as the basis for life ratings of rolling bearings. Later the adjustment constants a_1, a_2 and a_3 were also introduced:

$$L_{na} = a_1 \, a_2 \, a_3 \left(\frac{C}{P}\right)^p \tag{3}$$

a_1 = Different levels of reliability, S = (100 - n)%
a_2 = Material fatigue properties e.g. air melt = 1, VIM = 3 and VIM-VAR = 6
a_3 = Lubrication quality

The ISO basic rating life equation (Lundberg and Palmgren), Equation 2 makes no provision for engineering calculations incorporating details of material structure or cleanliness; the basic rolling bearing life, according to the ISO 281 (1991) equation, depends simply on a basic dynamic load rating (*C*) and an equivalent applied load (*P*). The load rating is based on bearing geometry and standard bearing steel (A295 52100 hardened at least to Rockwell C 58), so that any improvements in life arising from improved material properties can only be described through the factor a_2. However, the increased performance of bearings demanded by the ongoing pressure from customer requirements, forcing the bearing to carry higher loads and exposing it to increasingly high operating temperatures, suggests that a more inclusive approach to life prediction is needed. From the present point of view, effects of steel cleanliness are of particular interest and importance.

Such a materials-oriented, fatigue endurance strength approach to rolling bearing life can only be obtained by modification of the basic Lundberg-Palmgren equation. The Ioannides-Harris [15] equation, for example, introduced a material fatigue limit, τ_u, into equation 1 for the survival probability, at the same time making it a local equation for ΔS, the survival probability for each volume element ΔV:

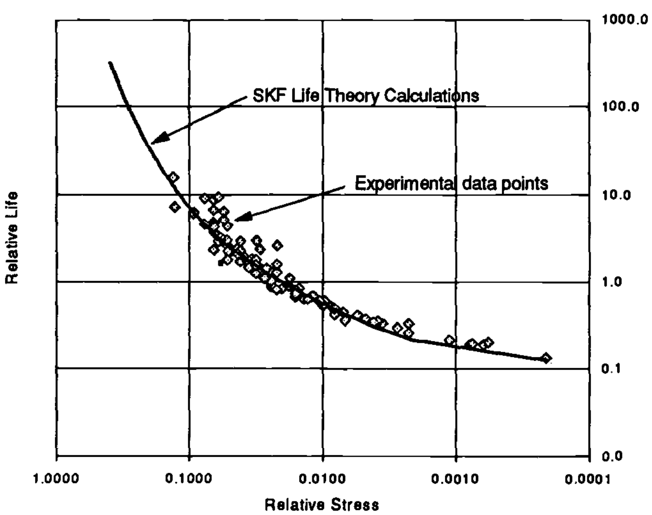
$$\ln\left(\frac{1}{\Delta S}\right) \approx N^e \, \frac{(\tau - \tau_u)^c}{z^h} \, \Delta V \tag{4}$$

ΔS = Survival probability for the discreet volume element ΔV, %
N = Number of cycles [-]
e = Weibull exponent (9/8)
z = Stress weighted average depth, [m]
h = Exponent in life stress equation (7/3)
τ = Maximum shear stress amplitude exceeding the fatigue limit, [Pa]
τ_u = Shear stress fatigue limit, [Pa]

Since *t* is a local shear stress and τ_u is a fatigue limit depending on certain components of the local stress tensor, it is clear that equation 4 contains much more information about the state of the stressed material than does equation 1. For example, the stress field produced in the vicinity of an inclusion clearly affects the survival probability of any volume element falling within it.

The τ_u fatigue limit is influenced not only by metallurgical considerations such as steel composition, heat treatment and internal inclusions but also, through its sensitivity to local stress components, by tribological considerations such as lubrication, lubricant contaminants and surface damage. In view of the microstructural instability noted in the fatigue process in bearings, [16], the τ_u fatigue limit in equation 4 is the stress below which microstructural alterations, which depend on both stress and temperature, may be indefinitely postponed. The model thus accommodates the influence of such stress cycle and temperature dependent microstructural decay through its effect on the stress fatigue limit. The model of equation 4 may thus be seen as combining a fatigue model, in which fatigue damage is dominated by a crack initiation process, with the preceding Lundberg-Palmgren model. The particular choice made for the fatigue criterion, t, can be adapted to the type of contact situation, such as those shown in Figure 4. Typically, a maximum shear stress amplitude is used. The characteristic improvement in life prediction made possible by the SKF Life Theory can be seen with reference to Figure 11, where relative experimental and calculated bearing life values are plotted against the stress level used in the bearing test.

Figure 11: SKF life theory calculated - compared to measured rolling bearing life



Rolling Contact Fatigue Life Calculations and Steel Quality

The τ_u value in the Ioannides-Harris equation is influenced by the stress concentration effects of internal inclusions, i.e. the fatigue limit is effectively reduced due to the stress concentrations found around an inclusion:

$$\tau_u \approx \sigma_{u,incl} \tag{5}$$

$\sigma_{u,incl}$ = Stress fatigue limit - inclusion (MPa)

Since from Equation 4, the risk of fatigue depends on the difference between the fatigue stress criterion, τ, and the fatigue limit, τ_u, from a physical point of view any modification of τ arising from the presence of inclusions may be taken as an equal but opposite modification of τ_u.

The Murakami [17] semi-empirical fatigue limit expression assumes that, for a given matrix material, the stress concentration effect of an inclusion depends only on its projected area. Thus:

$$\sigma_{u,incl} = \frac{a\,(HV+b)}{\left(\sqrt{InclusionEquivalentArea}\right)^{\frac{1}{6}}} \tag{6}$$

HV	=	Vickers hardness (matrix), [kg mm^{-2}]
a	=	Constant from Murakami (matrix), [m$^{7/6}$ s^{-2}]
b	=	Constant from Murakami (matrix), [kg mm^{-2}]

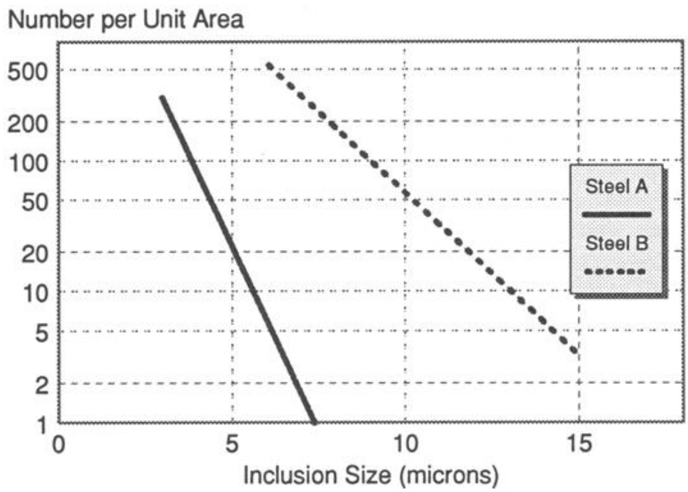
Taking the depth-weighting exponent, h, to be zero, the fatigue risk ΔR_{incl} introduced by each inclusion may be written as:

$$\Delta R_{incl} = (\sigma_{matrix} - \sigma_{u,incl})^c \, V_{incl} \tag{7}$$

σ_{matrix}	=	Matrix fatigue criterion, [MPa]
$\sigma_{u,incl}$	=	Stress fatigue limit at inclusions, [MPa]
c	=	Lundberg - Palmgren exponent, (31/3)
V_{incl}	=	Volume at risk, [m^3]

Thus the increased failure risk due to internal inclusions is modelled through the use of this reduced fatigue limit within the effective volume V_{incl}. A multi-level, multi-integration approach [18] is applied for micro-inclusion size distributions by suitably adjusting the fatigue limit in the Ioannides-Harris equation. The fatigue risk ΔR_{incl} is then integrated over the effective volume V_{incl} to evaluate the survival probability S in Equation 4. In order to demonstrate this approach, the micro-inclusion distributions shown in Figure 12 for steels A and B were modelled. The micro-inclusion size distributions were obtained by application of the new Swedish standard SS 11 11 16.

Figure 12: Typical micro-inclusion size distribution used in bearing life calculation



The relative lives modelled for 6309 deep groove ball bearing tested, at 3.3 and 4.2 GPa maximum Hertzian contact pressures, are shown in Figures 13 and 14. Both the relative life improvement for steels with reduced micro inclusion contents and the longer testing times with reduced rolling contact stresses are quite obvious.

Figure 13: Calculated results of stochastic dispersion of ball bearing life for 3.3 GP Hertzian contact stress bearing

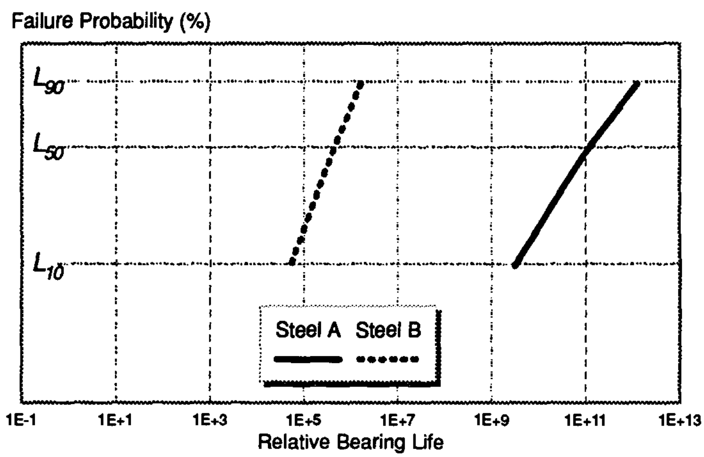
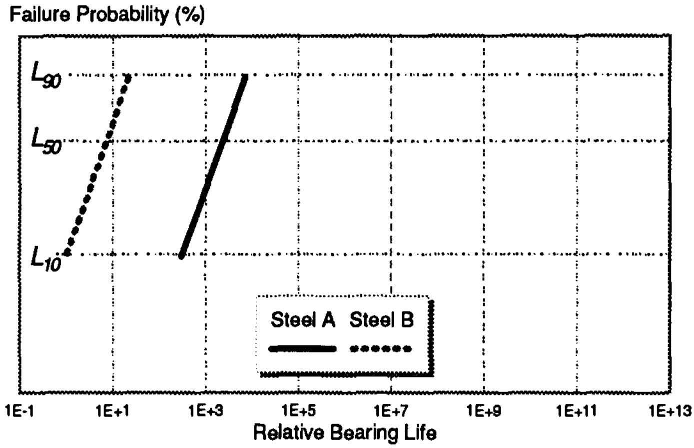


Figure 14: Calculated results of stochastic dispersion of ball bearing life for 4.2 GPa Hertzian contact stress bearing



In addition to modelling of such internal cleanliness effects, the methodology can also be applied to demonstrate the influence on rolling bearing life of more complicated stress distributions, for example biaxial stress fields σ_x and σ_y. Clearly, both tensile and compressive internal stress effects can be included. The calculated effect on relative life for a 200 MPa *tensile uniaxial* stress and a 400 MPa *compressive hydrostatic* stress is shown in Figures 15 and 16 respectively.

Figure 15: Calculated results of stochastic dispersion of ball bearing life for 4.2 GPa Hertzian contact stress with the addition of 200 MPa tensile uniaxial stress.

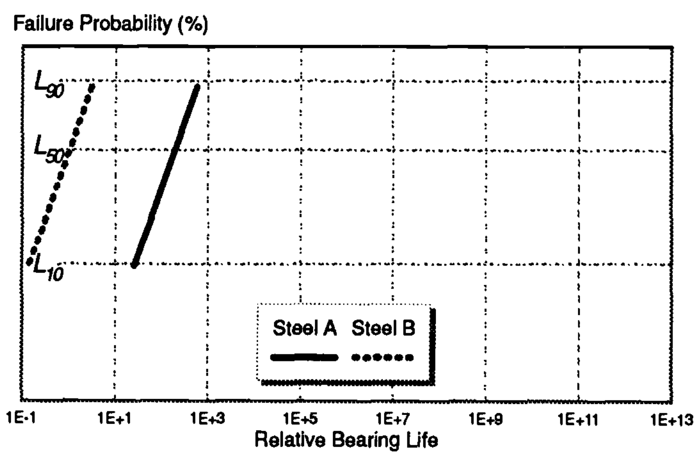
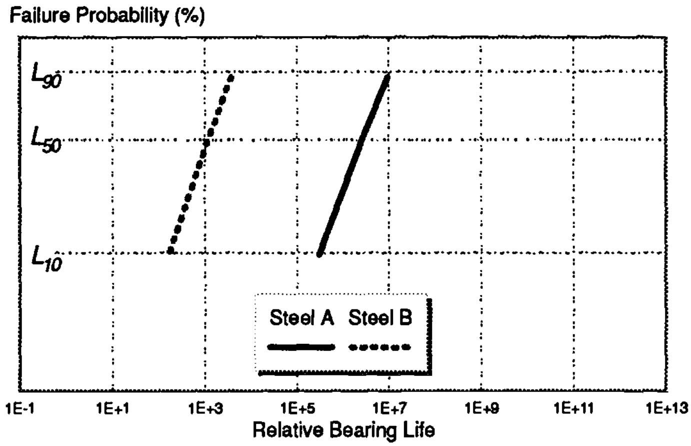


Figure 16: Calculated results of stochastic dispersion of ball bearing life for 4.2 GPa Hertzian contact stress with the addition of 400 MPa compressive hydrostatic stress.



Discussion

The use of the same steel composition, based on 1C-1.5Cr, for almost a century is a credit to the designers who made the original selection. The bearing industry is littered with attempts to change drastically the 1C-1.5Cr through-hardening steel composition but these to date have failed, largely due:-

- Exceptional product properties,
- Ease of spheroidisation and machinability,
- Relative robustness in the hardening properties,
- Degree of industrial standardisation,
- Global availability.

However the metallurgical quality, in terms of internal cleanliness has greatly improved in the last century, opening up the possibility for significantly increased reliability and bearing down sizing over the years. On the other hand, as has been discussed in the Introduction to this paper, the testing and verification of the effects on bearing performance of such reduced total oxygen contents and the related decrease in oxide inclusion contents is problematic for the discerning user and designer alike. In place of expensive and time-consuming endurance testing, a more generally applicable theoretical model of bearing life would lead to a deeper understanding of cleanliness effects, resulting in greater economy in the required verification procedures.

The development of suitable bearing service life models also gives an opportunity for the rolling bearing designer to specify the steel quality requirements related to the engineering demands. The application of value engineering in steel quality specification and related design decisions is an intelligent technique for avoiding under- or over- specification of internal cleanliness requirements. Clearly, value engineering methodology can only be applied if the bearing service life models have sufficient generality and precision in modelling the various metallurgical properties, such as internal cleanliness.

The accuracy of the SKF Life Theory has been demonstrated in this paper and it is reassuring to mention that the model is capable of reproducing the well known increased scatter with longer bearing lives or the reduced scatter with increasing test loads. The reduced scatter at higher bearing loads arises because the stressed volume under risk of initiating failure is larger and thus makes a more representative sampling of the entire inclusion distribution. In contrast at lower loads the risk of fracture originates from fewer larger inclusions which may not find themselves in the risk volume. The risk of failure and the associated fatigue lives therefore show higher dispersion. This means that a given Weibull exponent *e*, in the present model represents the scatter due to local variation of the microstructure and the properties of the inclusions, other than their size which has been explicitly included. Reduction of the load diminishes the effective composite *e* exponent.

The development of the SKF Life Theory and related use of the shear stress fatigue limit τ_u is the ideal opportunity for physical metallurgy (relationship of microstructure to mechanical properties) to interface with tribological aspects of rolling bearing theory. This paper has shown step-wise how internal steel cleanliness can be modelled with a precision which may exceed fatigue testing capabilities, given present-day demands on bearings. The methodology described here can also be extended to evaluate the influence of the numerous other microstructural characteristics of heat treated 1C-1.5Cr rolling bearing steel.

Conclusions

Internal cleanliness effects can be included in structural fatigue models for rolling bearings. Here the SKF Life Theory has been extended to include micro-inclusion effects by calculation of the local reduction in fatigue limit. A similar approach is also possible to include effects of:

- Microstructure, as influenced by heat treatment;
- Residual stress, as influenced by heat treatment and manufacturing.

Considerable progress has been made in improving the consistency of internal cleanliness of bearing steels. The methodology described here provides a basis for defining internal cleanliness specifications for rolling bearing steels, as related to:

- Engineering design optimisation,
- Process optimisation and related manufacturing economy,
- Speed in product development.

Acknowledgements

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IMPROVING PRODUCTION, CONTROL AND PROPERTIES OF BEARING STEELS INTENDED FOR DEMANDING APPLICATIONS

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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.

KEY WORDS: bearing steel, fatigue, non-metallic inclusions, ultrasonic

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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.

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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.

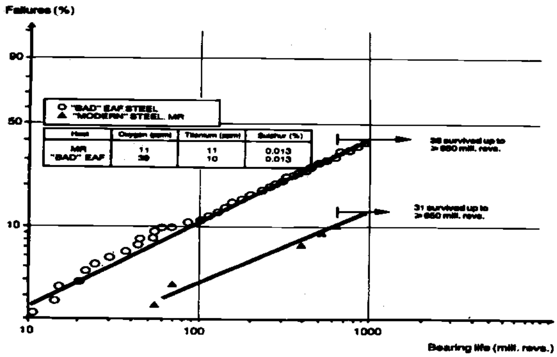

FIG. 1--*Fatigue life of ball bearings produced at the same time based on two-slag practice ('dirty') and ladle furnace produced ('clean') bearing steels.*

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.

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.

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 20 major bearing steel producers in Western Europe, the United States and Asia.

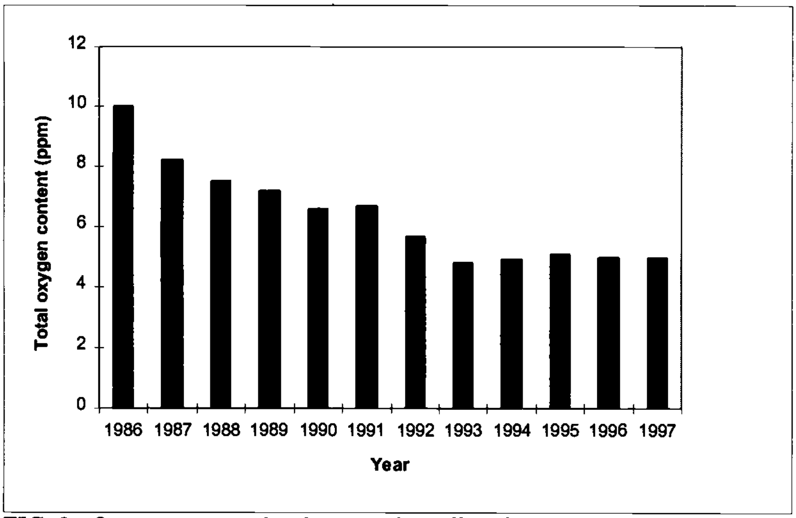

FIG. 2--*Oxygen content development for rolling bearing steel production.*

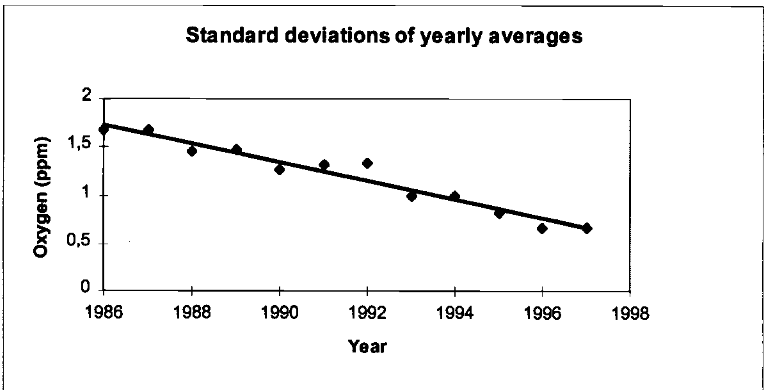

FIG. 3--*Oxygen content standard deviation for rolling bearing steel production.*

For each producer material from at least three heats, all produced later than 1995, were examined. The producers which consistently attain very low oxygen contents all have very long traditions in bearing steel production. 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.

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.

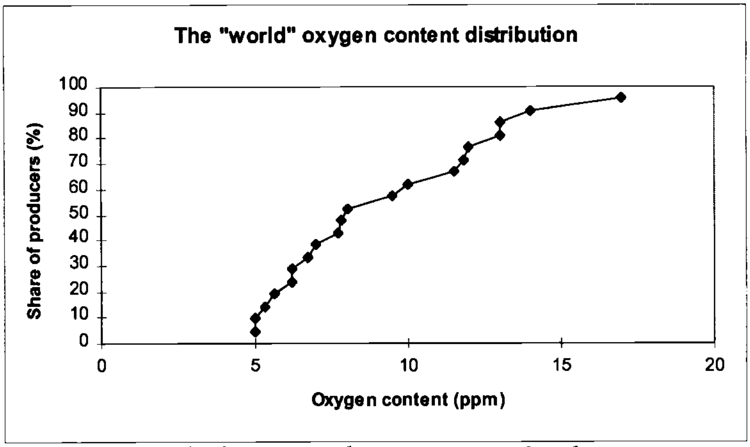

FIG. 4--*The 'world' bearing steel oxygen content distribution.*

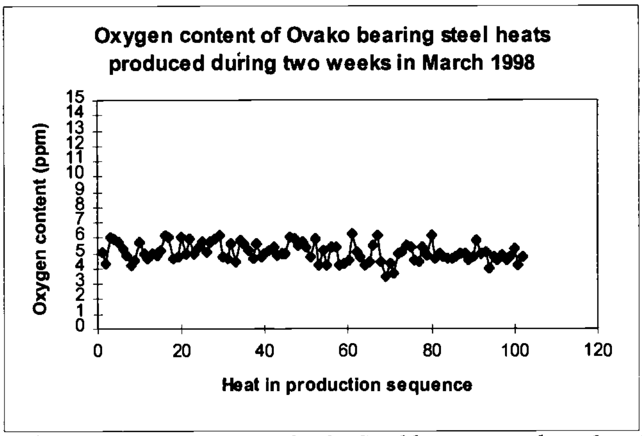

FIG. 5--*Oxygen content in Ovako Steel bearing steel produced during two weeks in March 1998.*

The same trend as earlier described remains: Know-how and tradition in bearing steel production is a more important factor to the quality attained than the equipment available for casting.

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 1998.

Titanium Content

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

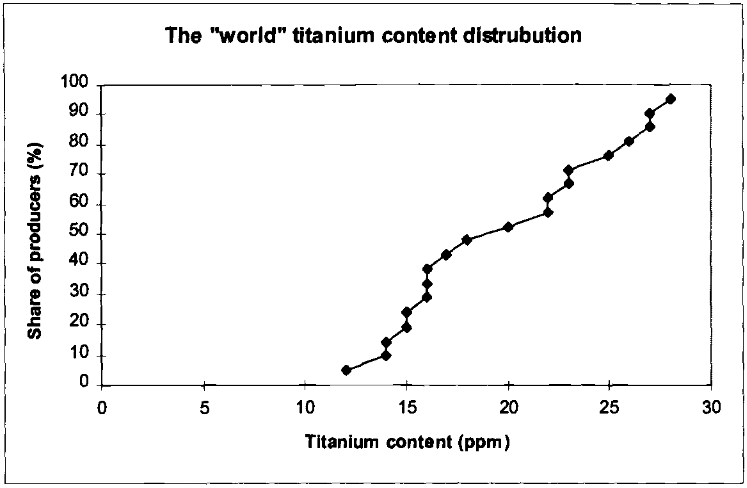

FIG. 6--*The 'world' titanium content distribution in bearing steels.*

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 practice.

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.

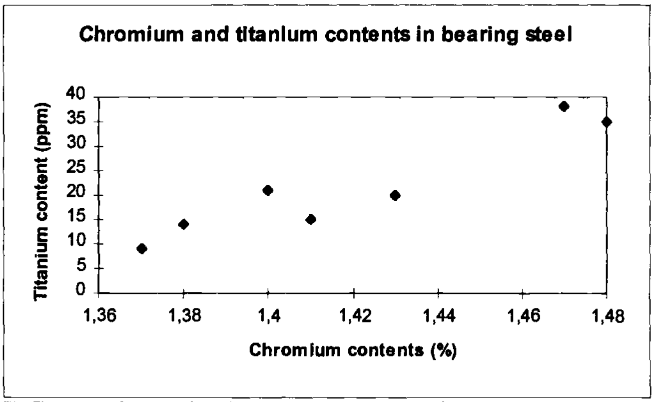

FIG. 7--*Relationship between titanium and chromium content for bearing steel produced using high purity ferrochromium.*

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

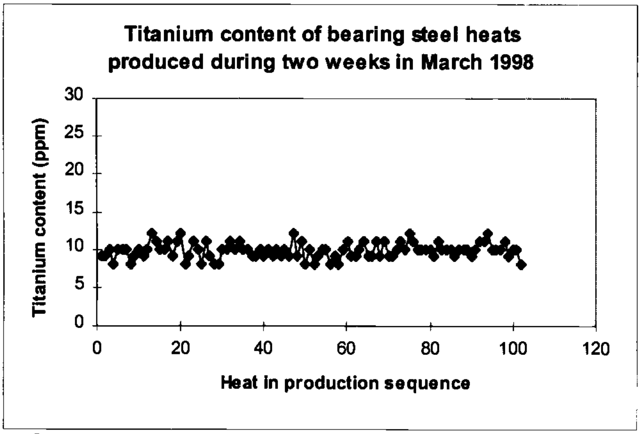

FIG. 8--*Titanium content in Ovako Steel bearing steel produced during two weeks in March 1998.*

Sulfur

The sulfur content can be regulated to the desired level in the ladle furnace, so basically the sulfur level is determined by the customer specification. Today, quite low sulfur 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 standardized 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 normalized 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 E 588).

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.

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

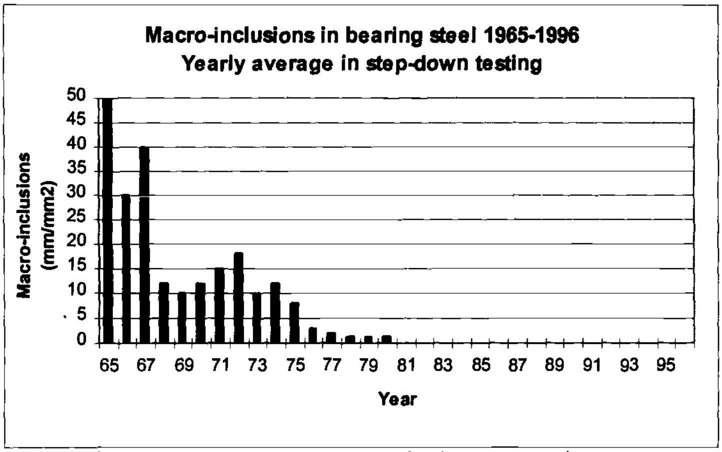

FIG. 9--*Yearly step-down testing averages for bearing steels produced at Ovako Steel.*

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

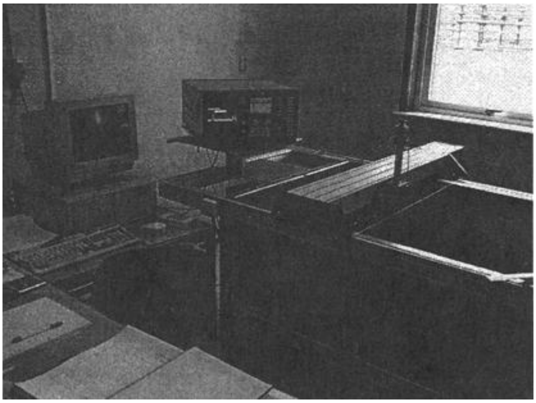

FIG. 10--*Ultrasonic scanning equipment.*

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.

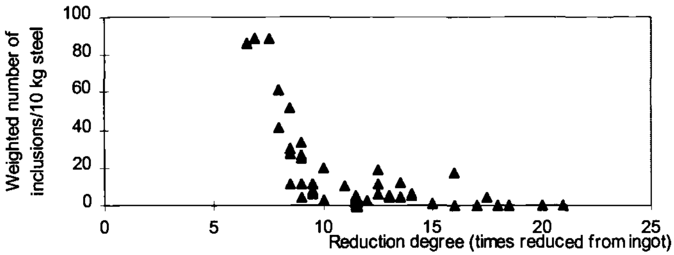

FIG. 11--*Reduction degree and ultrasonic scanning result on bearing steel billets.*

To get a comparative number, inclusions are classified into three size groups, weight factors are applied to each group, and the result is normalized 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 realized 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.

The data given in Fig. 11 derive from tests made on high-carbon bearing steels.

However, data cumulated for other low alloyed steel grades, as carburizing steels, strongly suggest that the same relationship is valid for all low alloyed steels.

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 analyzing the composition of a large number of oxide inclusions detected, pin-pointed and scanning electron microscope analyzed.

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.

Micro Inclusions

The world-wide standard for micro-inclusion rating today is the ASTM Standard Test Method for Determining the Inclusion content of Steel (E 45) with the sampling and evaluation rules of ASTM Standard Specification for High-Carbon Anti-Friction Bearing Steel (A 295).

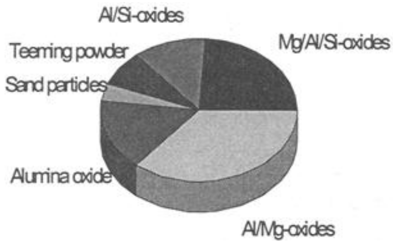

FIG. 12--*Chemical composition of inclusions found by UL-scanning in one single heat.*

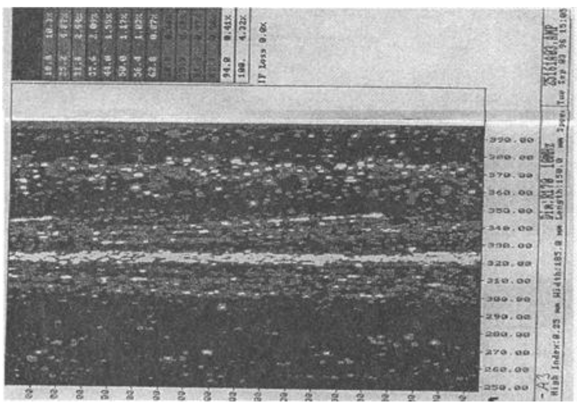

FIG. 13--*Steel sample with central porosity, segregation and large inclusions.*

Even with the recent upgrades of the E 45 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 today rate zero and thus provide no meaningful development information.

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 standardized in Sweden.

This method does provide meaningful correlation to chemical composition results.

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.

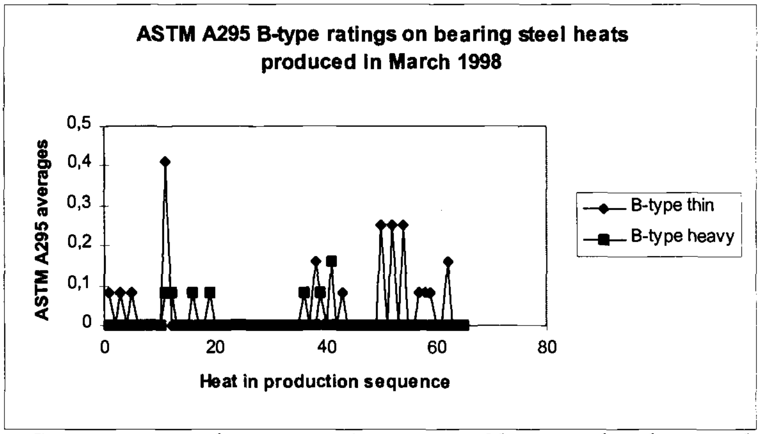

FIG. 14--*B-type oxide ratings on bearings steel heats produced in March 1998.*

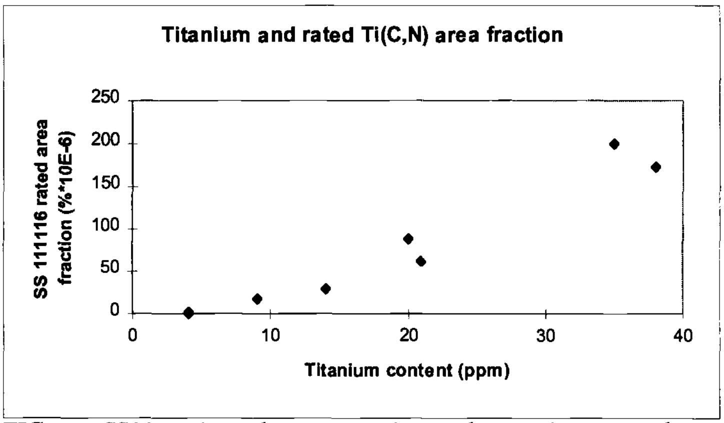

FIG. 15--*SS111116 rated titaniumcarbonitride area fraction and titanium content.*

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.

By combining the high resolution technology in ultrasonic testing with advanced sampling procedures new routes can be opened.

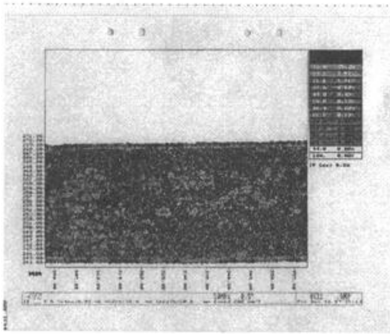

Fig. 16--*Example of scan at 50 MHz on low carbon, medium oxygen content steel.*

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.

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 analyze the content, size distribution and composition of non-metallic inclusions.

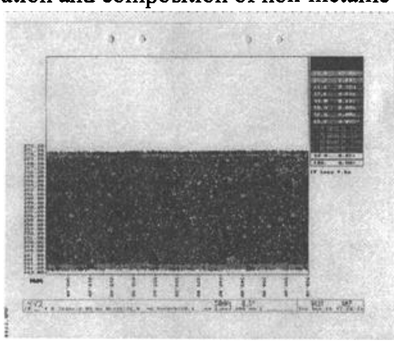

FIG. 17--*50 MHz frequency scan of a sample taken just prior to teeming (rolled, reduced 4x).*

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.

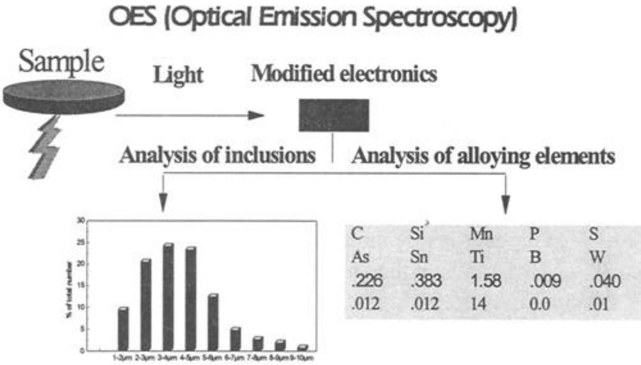

FIG. 18--*Signal capturing procedure for OES inclusion definition.*

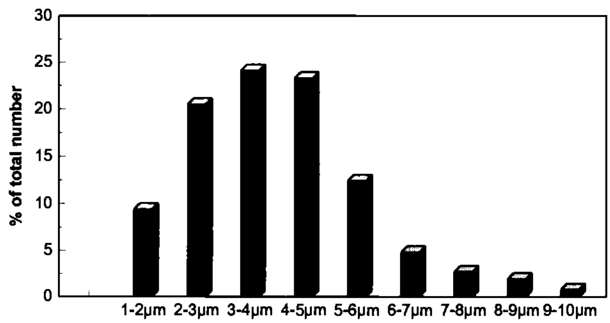

FIG. 19--*Size distribution of OES detected alumina oxide inclusions.*

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 and still cause a fatigue failure at a certain stress level. Finally, one fatigue initiation source may be carbide clusters. This has been seen in heavily segregated bearing materials 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 analyzed.

Different Oxide Types

Two heats produced by the electric arc furnace route were fatigue tested at a single stress level of 950 MPa. Aluminum 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.

TABLE 1--*Chemical composition in weight % (* in PPM).*

Heat	C	Si	Mn	P	S	Cr	Ni	Mo	Cu	Al	Ti*	O*	Ca*	N*
D7788	0.99	0.30	0.37	0.013	0.003	1.39	0.11	0.03	0.14	0.028	10	3.5	2	58
G1578	1.04	0.22	0.32	0.012	0.006	1.43	0.14	0.06	0.17	0.028	12	6.0	1	100

The majority of the fatigue failures were caused by oxide inclusions, approximately 85% in both cases, and the remainder were initiated by titanium carbonitrides. Surprisingly, the heat with the lower oxygen content had the lower fatigue life. This is shown in Fig. 20 with the L50 values which is the number of cycles 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 Fig. 21, were similar.

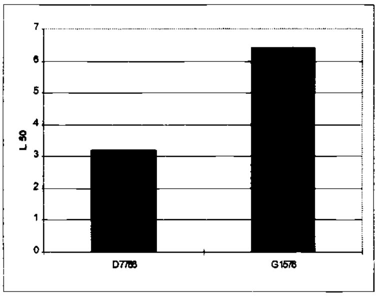

FIG. 20--*L50 life in 10^6 cycles for heat D7788 and G1578 tested at a stress level of 950 MPa.*

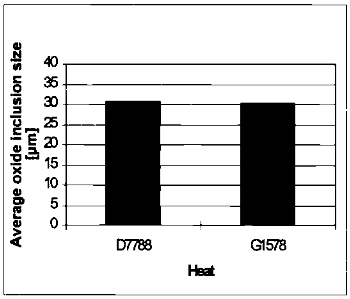

FIG. 21--*Average size of oxide inclusion initiating fatigue.*

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.

Analyzing the failure initiating inclusions in-situ quantitatively presented a problem. This mainly due to the fact that oxides as well as carbonitrides very often are associated with sulfides. As only the 'top' of the inclusions are available for analysis, presence of sulfide will interfere with the analysis of the initiating, 'core' inclusion.

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 Fig. 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%.

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.

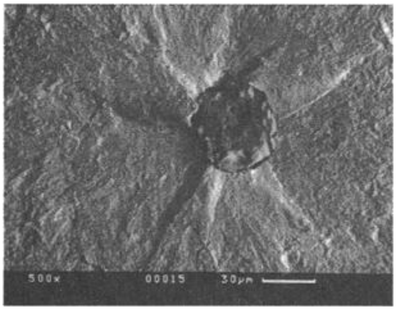

FIG. 22--*Cracked oxide inclusion.*

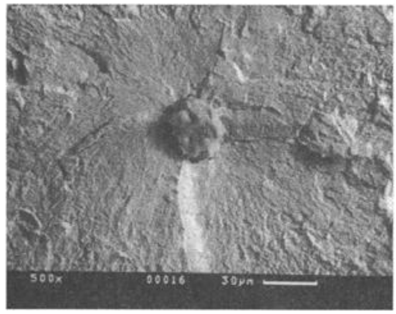

FIG. 23--*Solid oxide inclusion.*

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. Aluminum 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.

TABLE 2--*Chemical composition in weight% (* in PPM).*

Heat	C	Si	Mn	P	S	Cr	Ni	Mo	Cu	Al	Ti*	O*	Ca*	N*
G1578	1.04	0.22	0.32	0.012	0.006	1.43	0.14	0.06	0.17	0.028	12	6.0	1	100
G1581	1.01	0.24	0.28	0.012	0.007	1.39	0.14	0.05	0.21	0.030	11	5.0	1	118

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 were in both cases titanium carbonitrides. The fatigue life expressed as L50 values were 6.4 and 4.0 for heat G1578 and G1581 respectively. From the composition one would not expect a big difference between the two heats.

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 Fig. 24. The size of the titanium carbonitrides is much smaller compared to oxides. Fig. 25 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.

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.

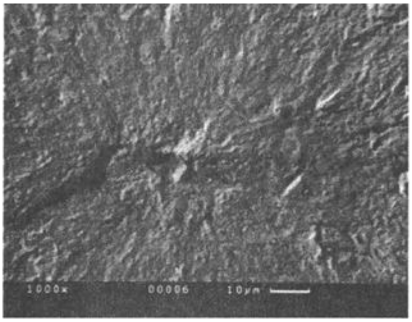

FIG. 24--*Titanium carbonitride causing a fatigue failure in heat G1581.*

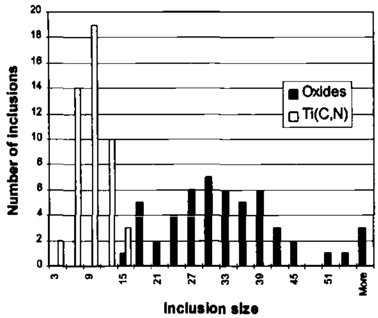

FIG. 25--*Size distribution, oxides and carbonitrides initiating fatigue.*

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.

TABLE 3--*Chemical composition in weight% (* in PPM).*

Heat	C	Si	Mn	P	S	Cr	Ni	Mo	Cu	Al	Ti*	O*	Ca*	N*
RM	1.06	0.20	0.31	0.006	0.0002	1.35	0.08	0.06	0.10	0.030	25	4.3	1	34

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

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 20µm, see Fig. 27. The fatigue life expressed as a L50 value was only 0.02 million cycles.

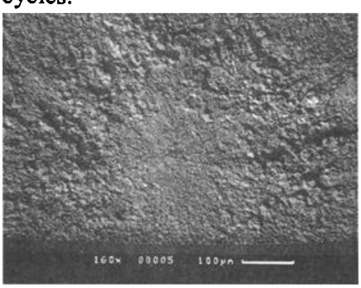

FIG. 26--*Titanium carbonitride causing fatigue in heat RM.*

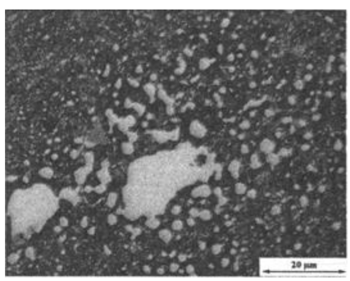

FIG. 27--*Carbide cluster in hardened sample of heat RM.*

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 remelting 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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MICROSTRUCTURAL FEATURES OF IMPLANT QUALITY 316L STAINLESS STEEL

REFERENCE: Disegi, J. A. and Zardiackas, L. D., **"Microstructural Features of Implant Quality 316L Stainless Steel,"** *Advances in the Production and Use of Steel with Improved Internal Cleanliness, ASTM STP 1361*, J. K. Mahaney, Jr., Ed., American Society for Testing and Materials, West Conshohocken, PA, 1999.

ABSTRACT: ASTM F 55 material specification was issued in 1965 to cover the metallurgical requirements of Type 316/316L stainless steel bar and wire for the manufacture of surgical implants. Eventually, it was recognized that austenitic implant stainless compositions with well defined nonmetallic inclusion limits would be desirable. The low carbon 316L alloy with specified inclusion limits emerged as the most widely used implant quality stainless material over the last 30 years. Microstructural requirements include a fully austenitic microstructure that is free of delta ferrite when examined at 100X magnification and an ASTM grain size of five or finer. The importance of low sulfur content, effect of secondary phases, intergranular corrosion resistance, and the compositional requirement %Cr + 3.3 X %Mo $\geq$ 26 are highlighted. Various clinical factors are discussed relative to the microstructural features of implant quality 316L.

KEYWORDS: metals (for surgical implants), stainless steel, surgical implants, microstructure, inclusion content, corrosion

During World War II there was considerable interest in identifying satisfactory stainless steel implant materials to repair bone fractures associated with wartime injuries. Murray and Fink recommended Type 302 stainless steel [1] to the U.S. Army and Navy in 1943. After the war, Peterson (1947) reviewed the clinical performance [2] of plates and screws used for fracture treatment by the Army. He concluded that 18-8 SMo (18% chromium + 8% nickel + 2% molybdenum) provided the best combination of

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properties. In the early 50's, Blunt et al. evaluated various metals that were implanted in dogs [3] and Type 316 appeared to be the best choice. This landmark effort set the stage for the widespread use of Type 316/316L for surgical implant applications. ASTM Standard Specification for Stainless Steel Bar and Wire for Surgical Implants (F 55-65T) was eventually published to define the metallurgical requirements for Type 316 (Grade 1) and Type 316L (Grade 2). ASTM Standard Specification for Stainless Steel Sheet and Strip for Surgical Implants (F 56) was published one year later.

The air-melted implant compositions performed reasonably well but it was recognized that premium-melted material offer an advantage. Remelted material was capable of providing improved homogeneity, controlled microcleanliness, and better corrosion resistance. ASTM F 138 Standard Specification for Stainless Steel Bar and Wire for Surgical Implants (Special Quality) was published in 1971 while ASTM F 139 Standard Specification for Stainless Steel Sheet and Strip (Special Quality) was issued in 1976. Both standards defined maximum microcleanliness ratings for Type A, B, C, and D nonmetallic inclusions. The low carbon 316L composition with specified inclusion limits emerged as the most widely used implant stainless material over the last 30 years. This review paper will highlight the composition refinements and microstructure features that have evolved over the years for implant quality 316L stainless steel.

Composition

The chemical requirements for implant quality and commercial quality 316L stainless steel are compiled in Table 1.

TABLE 1 -- *Comparison between implant quality and commercial quality 316L.*

Element	Chemical Requirements (%)[A] ASTM F 138-97 (Implant)	ASTM A 276-96 (Commercial)
C	0.030	0.030
Mn	2.00	2.00
P	0.025	0.045
S	0.010	0.030
Si	0.75	1.00
Cr	17.00-19.00[B]	16.00-18.00
Ni	13.00-15.00	10.00-14.00
Mo	2.25-3.00[B]	2.00-3.00
N	0.10	0.10
Cu	0.50	...
Fe	Balance	(Balance)

[A] Maximum, unless otherwise noted.
[B] %Cr + 3.3 X %Mo $\geq$ 26.

Lower phosphorus content provides somewhat better ductility especially for the majority of surgical implants that are moderately or highly cold worked. The reduction in maximum sulfur content from 0.030% to 0.010% has a favorable effect on the volume fraction of sulfide inclusions. The importance of manganese sulfide (MnS) control in implant quality 316L will be discussed in the next section. Reduced silicon content is responsible for a decrease in silicate type inclusions and provides better austenite stability. Higher chromium content has a favorable effect on corrosion resistance but has a negative influence on austenite stability. The nominal nickel content in implant quality 316L is significantly higher than in commercial quality and is primarily responsible for maintaining a completely austenitic microstructure. High nickel content minimizes the tendency to form delta ferrite (δ-ferrite) and cold working response is decreased because of the inverse relationship with nickel content. The minimum 2.25% molybdenum content is in agreement with the molybdenum requirements in ISO 5832-1 Implants for Surgery- Metallic Materials-Part 1: Wrought Stainless Steel. The compositional requirement $\%Cr + 3.3 \times \%Mo \geq 26$ conforms to the ISO standard and will be described in more detail in the section dealing with clinical factors. The limitation on copper content is a secondary method of controlling tramp elements that may be present in revert material.

Inclusion Limits

ASTM F 138/F 139 standards establish inclusion limits for implant quality bar, wire, sheet, and strip when evaluated according to Method A of ASTM E 45 Standard Test Methods for Determining the Inclusion Content of Steel. Microcleanliness limits are shown in Table 2.

TABLE 2 -- *Inclusion limits for implant quality 316L stainless.*

Inclusion Type	Maximum Limits	
	Thin	Heavy
A (Sulfide)	1.5	1.0
B (Alumina)	1.5	1.0
C (Silicate)	1.5	1.0
D (Globular Oxide)	1.5	1.0

Bar and wire inclusion ratings are usually performed on billet or bar samples while sheet and strip are rated at an intermediate hot rolled stage. In the past, some implant device manufacturers specified double vacuum melting such as vacuum induction melt (VIM) + vacuum arc remelt (VAR) to ensure that the inclusion limits would be met. The implant quality 316L suppliers developed a significant database over the years that verified that double vacuum melting was not required to meet the microinclusion limits. Alternate melting practices were established for implant quality 316L stainless which included electric arc + argon oxygen decarburization (AOD) refining + VAR in the United States. European melt practice was similar but included electric arc + AOD or vacuum ladle refining + electroslag remelt (ESR). VAR practice tends to minimize Type D globular oxides because oxygen content is reduced during vacuum remelting. The volume fraction of Type A sulfides are usually lower in ESR ingots because the slag layer can be formulated to desulfurize the arc melted 316L electrode.

Manganese Sulfide

Metallographic etching for the determination of δ-ferrite will reveal fine sulfides that may not be observed during examination of polished samples. These sulfides may be mistaken for δ-ferrite when examined at low optical magnification. Scanning electron microscopy (SEM)/wavelength dispersive spectroscopy (WDS) can be used to identify MnS inclusions that may be inadvertently classified as δ-ferrite during routine microstructural examination (Fig.1).

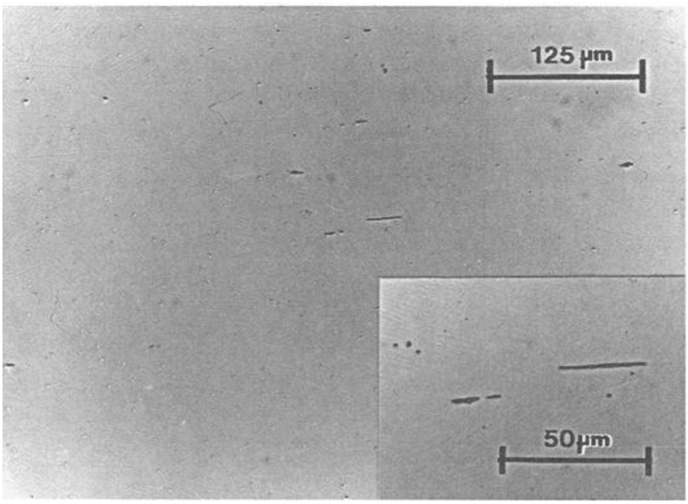

FIG.1-- *MnS inclusions in implant quality 316L stainless steel after etching with Kallings # 2 (200X and 500X).*

The certified heat analysis of the material lot examined in Fig.1 was 0.003% sulfur content. Microprobe analysis confirmed that the suspected indications were enriched in manganese and sulfur while elements that would be found at elevated levels in δ-ferrite such as chromium, iron, and molybdenum were at reduced concentrations. The elements associated with δ-ferrite are not totally absent due to to volume excitation of the matrix around the MnS inclusion.

Industry standards specify a maximum 0.010% sulfur content for ASTM F 138/ F 139 implant material. The sulfur content is relatively low but the implant quality 316L specialty steel producers always supply less than 0.005% sulfur content and routinely meet maximum 0.002% sulfur content.

Secondary Phases

Carbides

Carbides of the M_6C type have been observed [4] in the higher carbon Type 316 composition after prolonged heating in the 800-1200°F sensitization range. M_6C tends to precipitate intergranularly and has an adverse effect on intergranular corrosion resistance. This observation provided technical justification for the eventual widespread use of low carbon 316L rather than higher carbon 316 for surgical implants. Each lot of implant quality 316L must be capable of passing Practice E of ASTM A262 Practices for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels.

Delta Ferrite

ASTM F 138/F 139 standards specify that implant quality 316L microstructures must not contain δ-ferrite when examined at 100X magnification. Longitudinal specimens may be etched using Kallings # 2 (2 g copper chloride/40 mL hydrochloric acid/60 mL ethanol) to reveal the presence of δ-ferrite. An electrolytic potassium hydroxide (KOH) etch which consists of 45 g potassium hydroxide/60 ml water may also be used at 2.5 volts to confirm δ-ferrite which will be visible as a blue stain. Delta ferrite is considered an objectionable secondary phase in implant quality 316L because of increased magnetic permeability when compared to the austenitic matrix.

The specialty steel producers may also use weld metal constitution diagrams such as Delong or Schaeffler to estimate the amount of δ-ferrite in each remelted ingot. Numerous calculations over the years have indicated that δ-ferrite will typically not be observed in the microstructrue of homogeneous remelted material when the calculated δ-ferrite content is less than 1.5% based on chromium and nickel equivalents. The calculated δ-ferrite values represent a preliminary estimate since finish mill products must be metallographically examined to verify that δ-ferrite is not present in the microstructure.

Sigma

The presence of sigma phase (σ-phase) may be determined using electrolytic KOH etchant and will be visible as a red-brown stain. Sigma phase is frequently described [4] as an FeCr or Fe(Cr, Mo) intermetallic compound that can be promoted by high molybdenum content and the presence of δ-ferrite in the microstructure. This intermetallic phase creates an undesirable embrittlement effect and has been observed in 317L stainless weld wire that contains 3.00-4.00% molybdenum. For these reasons, the ASTM F 138 specification which originally specified a range of 2.00-4.00% molybdenum content was changed to 2.00-3.00% molybdenum content in the 1982 revision to minimize the possibility of σ-phase in the microstructure.

Chi

Chi phase (χ-phase) is a complex intermetallic phase that forms in alloys containing a substantial amount of molybdenum but this phase has generally not been observed in implant quality 316L material. The authors are aware of one instance where χ-phase was positively identified in implant quality 316L. It was postulated that improper thermal handling and molybdenum segregation were probably responsible for the formation of χ-phase.

Grain Size

Implant quality bar, wire, sheet, and strip must meet a grain size of five or finer according to prevailing ASTM standards. A fine grain size is desirable to provide a good combination of tensile and fatigue properties. The specialty stainless suppliers measure and certify the ASTM grain size after the last annealing operation. However, the majority of 316L implant stainless is ordered in the moderate or highly cold worked condition. The implant standards specify that the grain size should be measured on a transverse sample if the material is rated in the cold worked condition. This compensates somewhat for the grain elongation that occurs in the longitudinal plane during unidirectional cold working. Implant device manufacturers may also specify restrictions on grain size uniformity. For example, a grain size of ASTM 7.5 or finer may be acceptable as long as the material does not exhibit duplex grains that differ by more than 2.5 ASTM grain size ratings. A photomicrograph of moderately cold worked implant quality 316L with a grain size of ASTM 7.0 is shown in Fig. 2.

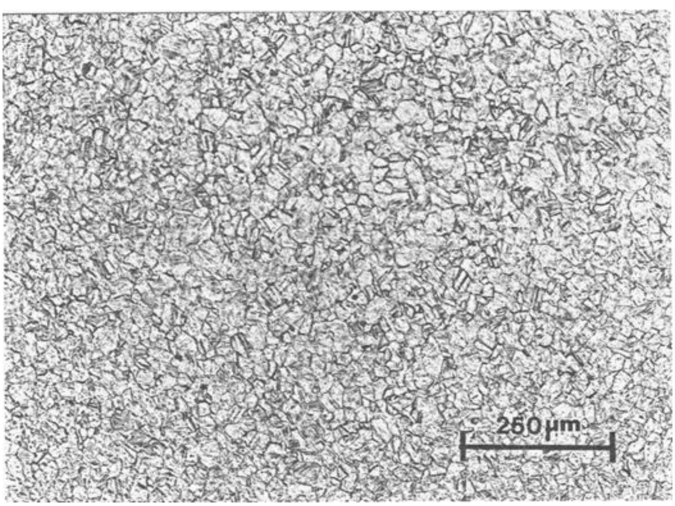

FIG. 2 -- *Etched transverse microstructure of cold worked implant quality 316L stainless steel (100X).*

ASTM E112 Test Methods for Determining Average Grain Size specifies that the grain size for cold worked round specimens should be measured in the transverse and longitudinal directions. The average grain size calculated from the transverse and longitudinal measurements should be reported as the final grain size rating.

Longitudinal transverse, and planar measurements are required for cold worked sheet and flat products according to ASTM E 112. Final grain size is reported from calculations of the measurements obtained for the three orientations.

Grain size requirements for cold worked implant quality 316L stainless steel as defined in ASTM F 138 and F 139 should be revised in the future to coincide with the procedures specified in ASTM E 112.

Medical device manufacturers commonly recertify lots of 316L implant stainless steel as part of their Quality Assurance program. Some small diameter wire products may be ordered in the extra hard condition with an ultimate tensile strength around 1400 MPa. It is not possible to accurately measure austenite grain size in material with this amount of microstructural distortion. In these instances, the recertification laboratory will normally document the grain size rating certified by the supplier after the last annealing operation.

Clinical Factors

The compositional requirement %Cr + 3.3 X %Mo $\geq$ 26 was derived from a study published by Steinemann in 1980 [5] which determined that adequate 316L pitting resistance in the body could only be assured if this relationship was satisfied. The increased chromium and molybdenum content of implant quality 316L provide improved corrosion properties in a chloride containing physiological environment. Calculations indicate that implant quality 316L compositions with a nominal 17.50% chromium content must contain at least 2.60% molybdenum to meet this compositional formula.

Sulfur content greater than 0.005% can create surface blemishes on electropolished 316L implants. The MnS present at the surface of the implant will be preferentially attacked during electropolishing and cosmetic features may be compromised. Uniform surface appearance is not as problematic when bead blasting or shot peening are used to produce a textured surface before electropolishing. Typical implant quality 316L compositions rarely exceed 0.002% sulfur content and electropolishing problems will be minimized regardless of whether a smooth or matte implant surface finish is desired.

Stainless grades such as 316L which contain molybdenum will not become magnetic during cold working because the stabilized austenitic microstructure is resistant to strain-induced martensite phase transformation at room temperature. An increase in magnetic permeability due to cold working or the presence of a secondary magnetic phase such as δ-ferrite are not desirable since patients with implanted devices may be subjected to magnetic resonance imaging (MRI) procedures. Microstructures must be completely nonmagnetic to avoid implant heating or movement of the implant during MRI. Magnetic permeability measurements [6] have confirmed that negligible magnetic response is obtained for highly cold worked (extra hard) 316L stainless implants.

Biocompatibility properties have not been the focus of this review. However, ASTM F 138 material has been successfully used for human implants for nearly 30 years. The alloy has a well characterized level of local biological response and is specified as one of the control materials in ASTM F 981 Standard Practice for Assessment of Compatibility of Biomaterials for Surgical Implants with Respect to Effect of Materials on Muscle and Bone.

Summary

Premium melting methods for implant quality 316L stainless steel include vacuum arc remelting or electroslag remelting to provide good compositional uniformity and a low volume fraction of sulfide, alumina, silicate, and globular oxide inclusions.

Important alloy requirements for implant quality 316L stainless steel include low residual sulfur plus high chromium and molybdenum for enhanced corrosion resistance and a balanced nickel content to provide a fully austenitic microstructure. Implant quality 316L stainless steel microstructural features include no delta ferrite, an absence of carbide and intermetallic secondary phases, an ASTM grain size of five or finer, and a nonmagnetic structure in the cold worked condition.

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Malcolm Blair[1], *Raymond W. Monroe*[2], *and John A. Griffin*[3]

A REVIEW AND UPDATE OF ADVANCEMENTS IN CLEAN CAST STEEL TECHNOLOGY

REFERENCE: Blair, M., Monroe, R.W., and Griffin, J.A., "**A Review and Update of Advancements in Clean Cast Steel Technology,**" *Advances In The Production and Use of Steel With Improved Internal Cleanliness, ASTM STP 1361*, J.K. Mahaney, Jr., Ed., American Society for Testing and Materials, West Conshohocken, PA, 1999.

ABSTRACT: The Steel Founders' Society of America Quality Assurance Task Force identified oxide macroinclusions as a universal problem experienced by users of steel castings. SFSA along with the Department of Commerce and the Department of Energy have sponsored research directed at reducing the occurrence of macroinclusions in steel castings. The Clean Cast Steel Technology program has investigated melting practice, pouring practice, gating practice, ladle treatment, and special devices such as filtration and analog simulation of mold pouring and filling.

In-plant trials have demonstrated a dramatic improvement in casting quality with submerged pouring of steel castings. Research is currently underway in optimizing foundry melting practice to reduce macroinclusions. A 30-50% reduction in macroinclusion occurrence has been observed in production castings at the foundries participating in the trials. Analog simulation and in-plant trials of pouring practices have demonstrated that poor gating practice can increase air entrainment and oxide inclusions. Ladle treatments such as calcium wire injection has been demonstrated in plant trials to significantly reduce oxide defects in steel castings. Experiments have been conducted at participating foundries to examine the benefits of filtration on casting quality. Filtration has been shown to reduce rework and scrap by 70% in some cases.

KEYWORDS: castings, steel, macroinclusions, reoxidation, submerged pouring, filtration, calcium wire injection, analog modeling

Introduction

Steel castings are crucial basic components used in agriculture, chemical processing, aircraft, road and rail transportation, steam and nuclear power production, and a host of other applications. Oxide macroinclusions in cast steel components

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represent a technological barrier to economical production, wider usage of castings, and product competitiveness in the U.S. steel casting industry.

Oxide macroinclusions have long been a major cost factor in the production of steel castings. These macroinclusions are typically composed of reoxidation products. Surface macroinclusion removal, weld repair, and reinspection add 20% to the cost of production. Subsurface inclusions not found and repaired in the foundry can cause costly disruptions in machining operations and result in poor component performance in service. Automated high-speed machining lines offer producers the opportunity to economically increase productivity and quality. These lines take rough components and machine them to final form without the variations and delays associated with operator-run standalone machining.

Surface and internal discontinuities must be reduced or eliminated to realize the full potential of machining operations including high speed machining lines. Macroinclusions are a major source of steel casting discontinuities. The reduction of macroinclusions in steel castings would therefore be an important step forward for the steel foundry industry.

Research has been conducted in several areas of steel foundry production where improvements in techniques would reduce the severity of surface macroinclusions. The areas included pouring, melting practice, ladle treatments, analog modeling of steel pouring and filling, and metal filtration. The following sections review and discuss the results from the research in each area.

Background

Oxygen can come from several sources including air, refractories, molding sand, or oxidizing slag, and react with steel to produce reoxidation products. There is substantial evidence that reoxidation is a major cause of oxide macroinclusions. Reoxidation is considered the reaction of elements in steel with oxygen after the steel has been deoxidized. This oxygen may come from the air, reactions with slag or refractories, or the reduction of water in air, refractories, or the mold. The formation of deoxidation and reoxidation products is schematically illustrated in Figure 1 [*3*]. Deoxidation products are small, typically 10 μm or smaller in diameter, primarily composed of alumina (Al_2O_3) in aluminum deoxidized carbon and low alloy steels, and dispersed throughout the casting. In reoxidation, oxygen locally depletes the aluminum and reacts with other oxidizable elements in the steel. Reoxidation products are typically macroscopic in size and float to the cope surface of the casting before solidification. Microstructurally, the products contain alumina particles that lie in multiphase globules formed by the oxidation of aluminum followed by silicon, manganese and iron [*1-3*]. The oxide matrix in which the alumina particles lie is typically rich in manganese and silicon, but in some cases may contain iron oxide. The presence of iron oxide may cause carbon monoxide gas holes to be formed within the inclusion as the iron oxide is reduced by carbon during solidification. Reoxidation is identified by the composition and volume of inclusions, which is related to the amount and composition of the deoxidizers. Control of reoxidation can be accomplished by limiting the exposure of the metal to oxygen in all forms since deoxidizer additions are necessary to produce sound steel castings.

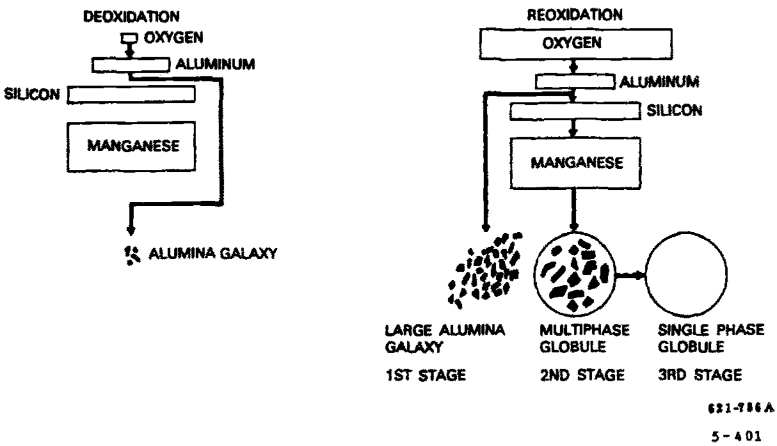

FIG. 1 – *Stylized formation of inclusions in cast steel deoxidized with aluminum.*

Oxidation of molten steel in air occurs very rapidly [*3*]. The oxygen concentration in steel shot reduced by hydrogen increased from near zero to 0.08% in about 0.25 seconds when exposed to air. The rate of reaction is probably limited only by the ability of oxygen to diffuse through the nitrogen rich layer surrounding the steel to replace oxygen already reacted at the liquid metal surface. In a foundry environment, a major exposure of molten steel to air occurs when pouring the steel into molds through

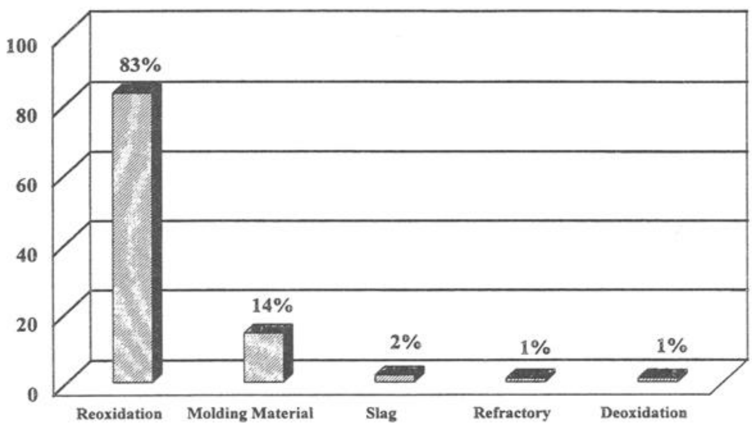

FIG. 2 -- *Distribution of macroinclusion sources for carbon and low alloy steel castings.*

air and when air is entrained in the pouring stream.

In many molten metal transfer operations, the large reoxidation products have time to float from the liquid. However, pouring into a casting cavity forms products that are likely to be trapped at or under the cope surface. In continuous casting, proper shrouding to exclude exposure to air has been found to reduce oxide inclusions by 90%. It has been estimated that in excess of 60% of steel inclusions is a result of reoxidation during casting [*1,3,4*].

Research sponsored by the Steel Founders' Society of America has shown that reoxidation of metal during pouring and mold erosion is the principal causes of macroscopic nonmetallic inclusion in steel castings. Approximately 425 defects from carbon and low alloy steels were removed and examined by optical and scanning electron microscopic techniques to determine the nature and form of oxide nonmetallic materials present. Castings came from a wide variety of practices including acid and basic melting furnaces, large and small pouring ladles, high silica and high alumina refractory ladle linings, and molds prepared from green sand and by resin bonding techniques [*5*].

The results of the examination of defects removed from carbon and low alloy castings are illustrated in Figure 2. Approximately 80% of the casting defects consisted principally of reoxidation products, and 15% consisted of eroded sand. Slag, refractory, and deoxidation products were relatively minor contributors.

The results of an examination of 100 ceroxide defects cut from high alloy and stainless castings are summarized in Figure 3 [*6*]. Approximately 50% of the defects

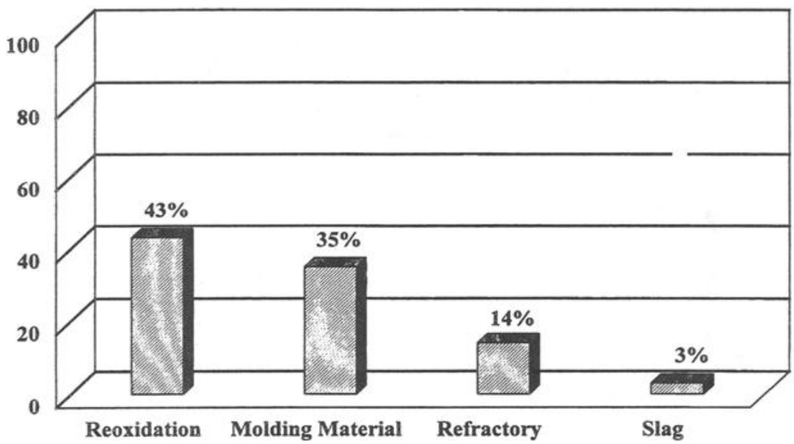

FIG. 3 -- *Distribution of macroinclusion sources for high alloy steel castings.*

consisted principally of reoxidation products. Approximately 35% were composed principally of eroded molding materials, and 12% consisted of ladle refractories. Slag carry over amounted to less than 5% of the total observed.

The incidence of these inclusions must be reduced to lower the labor and cost involved in producing castings and permit casting use on automated high speed machining lines. Achieving this goal requires that methods be devised to deliver clean metal, free of reoxidation products to the mold cavity. This must be accomplished by pouring with minimal exposure to atmospheric oxygen.

Advancements in Pouring Steel Castings

Shrouding and submerged pouring to eliminate air exposure has been successfully used in continuous casting of steel but has had in the past very limited success in reducing oxide inclusions in static castings. A submerged shroud pouring trial was conducted to determine if recent advances in shrouding technology would allow submerged pouring to be used in a steel foundry. The casting selected had a history of containing reoxidation macroinclusions on the surfaces especially in the diameter transitions.

The metal was melted in a thirty-ton basic electric arc furnace and the molds were formed from no-bake bonded silica sand. The shroud was produced from fused silica and used a clay-graphite gasket and toggle clamps to seal the shroud to the ladle. A total of sixty molds were poured conventionally. The remaining twenty-six molds were poured using the shroud.

Very little splash was associated with the submerged pour operation. After shakeout, the castings were heated treated and blasted and the surface quality of the castings was visually rated by counting the number of one-inch diameter circles needed to cover the surface macroinclusions.

Initial observation of the castings from both heats poured using the submerged shroud did not detect any macroinclusions on the casting surface. Macroinclusions were visible on almost all of the conventionally poured castings. A graph illustrating the cleanliness rating for all castings poured in both heats is shown in Figure 4. The conventionally poured castings contained an average of 9.35 one-inch diameter circles of macroinclusions with a sample standard deviation of 4.3. The submerge poured castings

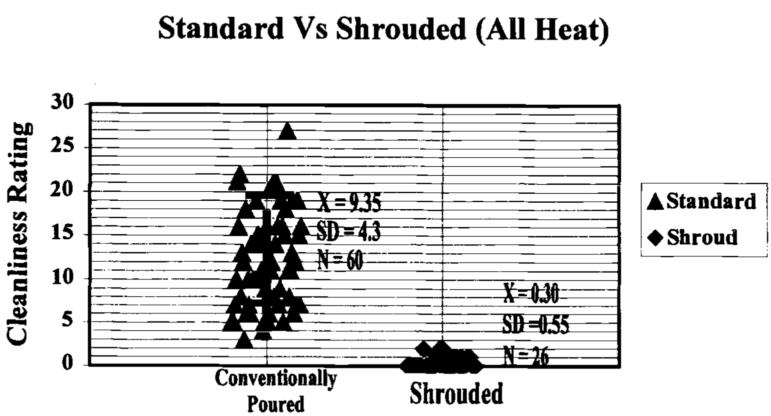

FIG. 4 -- *Cleanliness ratings for all shrouded and conventionally poured castings.*

contained an average of 0.30 one-inch diameter circles of macroinclusions with a standard deviation of 0.55.

Casual observation of the cleanliness ratings showed that submerge poured castings were dramatically cleaner than the conventionally poured castings. T-test analysis confirms that there is a 99.99% probability that submerge poured castings had a lower average and a smaller standard deviation.

Advancements in Foundry Practice

A long-term trial has been conducted at three foundries to determine if causation between some variable or variables in the melting practice and pouring practice and average heat casting quality can be determined. Information on casting quality, metal and casting processing was collected over the length of the trial. After statistical analysis and discussions with foundry personnel, variables that significantly affected heat quality were selected. Additional heats were poured at the foundries with the selected variables at the optimum levels to verify the improvement in heat quality. The goal was to gain a better understanding of the influence of melting and pouring practice on casting quality and, subsequently, determine how to reduce the heat-to-heat variation.

There were several requirements that the foundry had to meet in order to participate in the trial. The foundry was requested to provide at least five to ten defects of interest to determine if the defects were reoxidation products. The foundry had to have a casting that has a quality problem and had a reasonable production rate. Documentation from a minimum of 30 heats was needed for a trial. The 30 heat criteria provided enough data for a good statistical analysis. The foundries also had to qualitatively rate the castings for surface quality. The method used to measure casting quality was largely left up to the foundry.

The participating foundries were asked to supply as much information as possible along with any other documentation, such as the heat sheets, that might be available. The foundries for the most part did not record any data that was not already available somewhere in the foundry.

After the data for the heats were collected, the variables from each heat were keyed into a statistical analysis program. Normality tests were conducted on the casting quality data to ensure statistical analysis techniques could be used. The data was analyzed by two different methods using the average casting quality for the heat as the dependent variable. In the first method, each independent variable was analyzed individually using simple regression and then ranked according to the highest R^2 and the highest statistical significance. In the second method, a t-test was used to detect statistically significant differences of the variables between the cleanest and dirtiest heats. Although the order may change slightly, both methods typically pick the same independent variables as having a significant influence on heat quality.

There were usually 10-12 variables that had at least a moderate correlation with overall heat quality. The list of variables was then reviewed with foundry personnel to evaluate the results. Typically, the list was narrowed down to four or five variables that strongly affected heat quality, were directly affected by the melting or pouring practice, and have a reasonable engineering solution for influencing heat quality. A subsequent trial was conducted at the participating foundries to verify the influence of the variables selected.

One acid practice foundry and two basic practice foundries were involved in the trials.

The acid practice foundry melted 1025 carbon steel in a 20 ton, acid electric arc furnace. Thirty-five heats were collected over the span of about 12 months. Approximately 150 variables were recorded or calculated for each heat and stored in an analysis spreadsheet. The acid practice foundry selected six variables from a list of thirty-four that had a reasonable probability of influencing the quality of the heat [7]. These variables were metallostatic head pressure (lower is better), metal pouring temperature (higher is better), residual silicon after oxygen blow (higher is better), residual manganese after oxygen blow (higher is better), amount of slag thinner added to the ladle (higher is better), and furnace tapping rate (higher is better). An additional 24 heats were poured with the six variables set at levels to produce clean heats. The average heat quality rating for the 24 heats decreased from about 32.6 inches to an average of 18.1 inches, a decrease of about 44%. Subsequent analysis by the foundry indicated that three variables had the strongest influence on casting quality: metal head height, pouring temperature, and silicon level after the oxygen blow.

The first basic practice foundry melted 1025 carbon steel in a ten ton, basic electric arc furnace. Twenty-five heats were used in the initial analysis. Approximately 80 variables were recorded or calculated for each heat and stored in an analysis spreadsheet. Initial t-test analysis of the basic practice foundry data indicated that ten variables influenced heat quality. From this list, six of the variables involved carbon and manganese concentrations in the metal. After discussions with the foundry, the variables of interest were determined to be the percent concentration of carbon and manganese at meltdown. An L4 designed experiment was run to evaluate the effect of meltdown manganese and carbon concentrations on heat quality. Eight heats were poured in the L4 with two heats at each condition (low carbon, low manganese | low carbon, high manganese | high carbon, low manganese | high carbon, high manganese).

Subsequent analysis of the L4 revealed that meltdown carbon did not significantly affect heat quality. The manganese concentration at meltdown was, however, important with low meltdown manganese producing cleaner heats compared to high meltdown manganese. The heat quality improved from an average of about 4 to less than 2, an improvement of about 50%. It should be noted that silicon concentration was not measured at meltdown at this foundry.

Similar to what was observed in the acid practice foundry, the clean heats had a higher manganese compared to the dirty heats. More manganese was available for reaction with oxygen in the clean heats. This was an indication that there was less oxygen available to react with the manganese.

The second basic practice foundry melted 1025 carbon steel in a 25-ton electric arc furnace. Thirty-one heats were evaluated over the span of about two months. Approximately 140 variables were recorded or calculated for each heat and stored in a spreadsheet. Initial review of the data showed that one heat had an inclusion surface area count four times higher than the next largest area count. The data also failed all tests for normality. This heat was removed from the analysis. The normality for the remaining heats was adequate to proceed with further analysis.

Initial analysis of the foundry data indicated that four variables influenced heat quality. In both tests, the percent manganese recovered from the slag after tapping had the strongest correlation to casting quality (higher is better), followed by the angle of the oxygen lance (higher is better), number of heats on the ladle lining (higher is better), and tap rate (higher is better). The initial results were discussed with foundry personnel to determine the next course of action.

A second set of castings was poured at the foundry with four variables set at their optimum level. This test was performed to demonstrate that these variables do in fact improve metal and casting quality. Calcium carbide was added to the slag after tapping and more slag was tapped with the metal to increase manganese recovery from the slag. Tap rate was increased as much as possible and the oxygen lance angle was controlled. Additionally, the silicon and manganese concentrations at meltdown were increased. The number of heats on a ladle lining is not a practical variable to control.

Seven additional heats were poured under these conditions and rated identically to the previous heats. The average area of cope surface inclusions decreased by about 51% from 75.4 to 37.2 inches squared. As important, the variation in casting quality decreased by 76% from 53.8 to 13 inches squared.

All three foundries showed significant improvement in casting quality by manipulating the melting practice. Improvements ranged from 40-50%.

TABLE 1 -- *Important variables in producing clean heats from all foundries.*

Variable	Condition
Acid Practice Foundry	
Metallostatic Head Pressure	Lower is Better
Pouring Temperature	Lower is Better
Residual Silicon After Decarburization	Higher is Better
Residual Manganese After Decarburization	Higher is Better
Amount of Slag Thinner Added to Ladle	Higher is Better
Furnace Tapping Rate	Higher is Better
First Basic Practice Foundry	
Carbon Concentration After Meltdown	Lower is Better
Manganese Concentration After Meltdown	Lower is Better
Amount of Manganese Added to Ladle	Higher is Better
Final Manganese Concentration	Higher is Better
Ladle Aluminum Concentration	Lower is Better
Amount of Carbon Blown From Furnace	Lower is Better
Second Basic Practice Foundry	
Manganese Recovery From Slag	Higher is Better
Oxygen Lance Angle	Higher is Better
Number of Heat on Ladle Lining	Higher is Better
Furnace Tapping Rate	Higher is Better

Table 1 lists the important variables in producing clean heats from all of the foundries. These variables can be grouped into two separate categories. The first category is the pouring and filling practice. Metallostatic head pressure and tapping rate were found important in three of the four foundries with lower head pressure and faster tapping rate improving casting quality. Lower head pressures reduce the velocity of the metal exiting the nozzle and throttling of the ladle. The first basic practice did not have head height as a variable since the entire heat was used to pour the selected casting. High metal velocities and flaring during throttling have been shown to increase air entrainment and reoxidation of the metal [*8*]. Higher tapping rates also reduce the amount of oxygen exposed to the metal.

The second category concerns the concentration of oxidizable elements contained in the steel. Silicon, manganese, and aluminum concentrations were important factors in all three foundries. These elements can be indicators of the oxygen concentration contained in the steel. Carbon is typically used as an indicator of oxygen concentration in the steel bath. However, authors have shown that other oxidizable element can also influence the oxygen content of the steel and the availability of the oxygen to form reoxidation inclusions in steel castings [*8,9*]. By using the oxidizable elements as indicators and manipulating the melting practice to optimize the concentration of these elements, improvements in casting quality can be observed.

Clean heats can consistently be produced if the oxygen concentration in the steel bath can be minimized through improved melting practice and reducing exposure of the steel to atmospheric oxygen during pouring and filling.

Advancements in Ladle Treatments

At the 1993 Steel Founders' Society of America Technical and Operating Conference, Robert Shepherd presented a paper comparing acid and basic melting practices at Harrison Steel Castings Co. [*10*]. The trial lasted over a year and produced over 900 basic heats of various grades of carbon and low alloy steel. Shepherd determined that basic refractory practice reduced dirt defects in the castings. Calcium wire injection was found to significantly reduce dirt defects (32%) in the acid practice.

This data was reanalyzed to try to determine the relative effect of melt practice and other factors on casting quality. The objective of this analysis was to determine the effect of calcium wire injection, refractory practice, and casting type on heat to heat average casting quality and variation in casting quality. This was done to sort out melting and metal handling from pouring and gating practices.

Data on four different casting designs were selected that met two criteria: 1) the castings were produced with and without calcium wire injection and with acid and basic refractory and 2) a relatively large number of heats and castings were produced for each casting design to allow statistical analysis. Heats where only one casting was poured were deleted from the data set. This was done to ensure metal quality from the heat was characterized by a minimum of two castings. Casting design was identified by the casting pour weight. A total of 222 heats and 505 castings were used in the analysis. Casting quality was determined by measuring the length of weld excavation needed to remove surface macroinclusions.

A three-factor two-level analysis of variance (ANOVA) was performed on the data using average casting quality as the dependent variable. When the analysis was based on average casting quality, casting type was by far the largest contributor at 83%, followed by calcium wire injection and refractory type both at 2.5%.

The grand mean for all heats was 8.17 inches of dirt. Injecting calcium wire reduced the dirt inches on average from 9.66 to 6.68 inches for all castings. Dirt inches for casting type varied from 1.78 inches in the 6900 casting to 21.31 inches in the 8900 casting. Switching from acid to basic refractory reduced dirt inches from an average of 9.67 inches to 6.67 inches.

The interaction of calcium wire injection and refractory practice is illustrated in Figure 5. Calcium wire injection with acid refractory practice produced castings with average casting quality similar to basic refractory with calcium injection. The graph also shows that basic practice is cleaner than acid practice without wire injection. Calcium wire injection with a basic practice does not significantly improve average casting

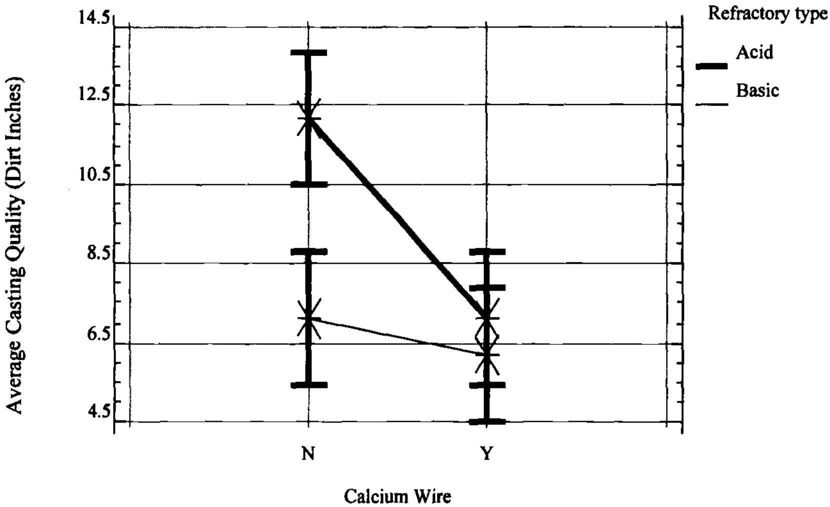

FIG. 5 -- *Interaction of calcium wire injection and refractory type.*

quality. This is consistent with the findings of Shepherd [*10*]. Similar results with calcium wire injection with acid or basic suggests that other factors such as molding, pouring, or gating limit the ability to produce cleaner castings.

The variation in casting quality is also important. Another ANOVA was run using standard deviation as the dependent variable. This shows how consistent the process is and which factors makes it more or less consistent. Standard deviation was used as an indication of casting quality variation. Refractory was the largest contributor to casting quality at 37% followed by calcium wire injection at 8%. Casting type did not influence the amount of variation in casting quality. The remaining casting quality variation (55%) cannot be explained by furnace refractory, calcium wire injection, or casting design. This unexplained variation might be contained in melting practice variations. As average casting quality improves, reducing casting quality variation will become more important.

The grand mean for variation in casting quality was 4.85 inches of excavation. This compares to 8.17 inches for average casting quality. Calcium wire injection reduced variation from 5.68 to 4.03 inches. Changing furnace refractory from acid to basic had the largest influence by reduced quality variation from 6.42 to 3.29 dirt inches.

There was no interaction between furnace refractory and calcium wire injection. Calcium wire injection reduced the variation in both acid and basic melting. The lowest variation was basic refractory with calcium wire injection and the greatest variation was no calcium wire and acid refractory.

Casting type, which includes gating, inspection standards, casting shape, etc., was the most important factor for average casting quality. Improvement in casting quality was similar for use of basic instead of acid refractories, or use of calcium wire with acid refractory. The combination of basic refractory and calcium wire produced the best average heat quality. Acid refractory with calcium wire produced average heat quality similar to basic refractory with calcium wire. No explanation was found for the variation in casting quality from heat to heat.

Casting type had no effect on casting quality variation. Largest reduction in quality variation was due to basic melting. Calcium wire injection also significantly reduced variation from heat to heat in both acid and basic melting. Reduction in the number of outliers is an opportunity to reduce the average and variation of cope surface macroinclusions.

Advancements in Analog Modeling of Steel Pouring and Fill

Water modeling experiments have been conducted on pouring techniques for minimizing air entrainment. The use of water to predict the amount of air entrained during the metal pouring operation has a solid theoretical foundation because water and molten steel have a similar kinematic viscosity. Air entrainment when pouring water in a full-scale model is expected to be similar to pouring steel because the similarity in viscosity causes the Reynolds number, Froude number, and the Z parameter for the two liquids. Air entrainment rates from water model experiments have correlated with cleanliness ratings of castings poured under similar conditions.

The modeling experiments described explore techniques for minimizing air entrainment during pouring of metal. A literature study was used in developing the water modeling apparatus, designing the experimental matrices, and conducting trials using bottom pour ladles.

The "mold" into which water was poured consisted of an acrylic box cavity with inside dimensions of 18" x 18" x 36". This box was used to simulate the cavity of a conventional sand mold and had several holes machined to allow various commercial sprues and gating systems to extend through the top while keeping an air tight seal to eliminate air leakages.

Water was poured directly from a ladle into the pouring cup and downsprue for experimental simplicity. During pouring, water from the ladle and air entrained with the water entered the mold box. The water collected in the mold box caused some air displacement from the box. The entrained air and the displaced air exited the box through vents and were collected in a sealed bag. After a pouring experiment was completed, the mold was isolated from the gas collection system by a valve.

The total volume of air in the sealed bag was determined by pumping the air out through a precision wet test meter. The total volume of air collected minus the displaced air (determined from the weight of water poured) provided a direct measurement of the volume of entrained air.

A 20-inch square by 26-inch deep acrylic bottom pour ladle was used for all of the bottom pour trials. The effective volume of the ladle above the stopper-nozzle contact line was about 4 cubic feet providing an equivalent steel capacity of about 2000 pounds. The ladle was constructed to allow various nozzle and stopper combinations to be easily interchanged.

All of the water modeling trials were setup to be analyzed using Analysis of Variance (ANOVA). All of the data arrays were orthagonal (balanced), full factorial, and triplicate experiments were made for each condition. The data was statistically analyzed using commercial statistical software.

A series of steel casting trials have been conducted in a steel foundry and companion water modeling experiments conducted to correlate the casting cleanliness with the amount of air entrained in water models. A 4530 lb casting was poured using two gating systems. All castings were poured through a 2-inch diameter nozzle and all variables held as constant as possible.

Casting quality with the original gating system averaged 16.2 linear inches of dirt with a standard deviation of 5.2. Casting quality with the new gating design averaged 34.9 inches of dirt with a standard deviation of 13.9, an increase of about 115%.

A water modeling trial was conducted to determine if a gating system simulation would produce air entrainment volumes that correlated with cleanliness ratings in the castings. If successful, the trials would help confirm the connection between air entrainment and casting cleanliness and, on the ability of laboratory scale water model experiments, to predict reactions on the casting floor.

The variables in the water modeling trial included the gating system design ("original" vs "new") and stopper opening (fully open vs throttled). The experimental matrix was an orthagonal, full factorial L4 designed experiment. The bottom pour ladle was filled at the beginning of each experiment. Approximately one cubic foot of water was poured through a 2" diameter nozzle and stopper head supplied by the foundry.

The variation sources include the main variables and two level interactions. The sum of squares determines the influence of a particular factor on the dependent variable being measured. The F-ratio compares the variation caused by the factor to the variation caused by noise. A high F-ratio gives confidence that the factor does significantly influence the dependent variable.

The ANOVA analysis indicates that the stopper opening is the most important factor, and it contributes about 73% to the total air entrained. Gating design contributed about 22% to the total air entrained. The interaction between stopper opening and gating design was not a significant factor and residual accounted for about 5%.

Statistical analysis of the data indicated that the new gating design increased the air entrainment rate by about 46%. This increase was less than the 115% increase in dirt observed in castings poured with the new gating system. However, the overwhelming influence of throttling may have skewed the analysis. On a large casting, throttling was probably not necessary throughout the pour. The air entrained with fully open nozzle presents an increase of about 94% with the new gating design compared to the "original" gating design. This is more in line with the cleanliness ratings of the castings.

The "new" gating design had two modifications compared to the "original" design that could have increased air entrainment. The runner diameters were increased and would not be completely filled by the single downsprue. This would reduce the hydraulic losses or backpressure in the downsprue. The pouring stream would drop further before impact with the pool in the sprue and hence would have a higher jet velocity. Air detrainment would also be much less likely with lower backpressure. The additional 90° vertical elbow could also increase entrainment by creating a waterfall effect.

The water modeling air entrainment data followed the same trend as observed in the foundry casting cleanliness measurements. The foundry observed a sharp increase (about 115%) in casting inclusions with the "new" gating system compared to the "original" gating system. The water model showed an increase in air entrainment of about 50% when using a nozzle similar to the one used at the foundry. However, comparing the entrainment rates for the fully open stopper condition (a more realistic condition) showed approximately a 94% increase in entrained air when changing from the original gating system to the new gating system. This is close to the increase in casting inclusions observed at the foundry.

Advancements in Steel Filtration

A trial was conducted at a participating foundry to evaluate the effect of filtration on the cleanliness and repair time associated with the production of a stainless steel manifold [*11*]. Cleaning room and weld repair time were used as indicators of casting cleanliness. The purpose of this trial was to determine the filter performance, the cleanliness of the resulting castings, and the potential economic benefits of a large steel casting. The castings poured are near the upper limit weight range of steel parts that can be successfully poured through filters.

All of the parts were cast from 17-4 PH stainless poured at 2900-2950 °F. The steel was melted in a MgO/Al_2O_3 lined induction furnace, and the molds were formed with furan-bonded silica sand. The casting weight was approximately 900 lbs. Four ceramic 3" x 3" x 1", 15 ppi, foam filters were used on each casting. A tile downsprue with sand runners formed the gating system. The gating system for the unfiltered castings was similar except that no filters were placed in the filter prints.

The castings were cleaned, examined, and given a first stage weld repair after shakeout. The castings were then liquid penetrant tested and radiographed and then given a second stage weld repair. The effectiveness of filtration was measured by reductions in both the cleaning room and weld repair time. The first and second stage times associated with both cleaning and weld repair were added together.

A one way ANOVA analysis was performed to determine if there was a significant difference in cleanliness between the unfiltered and filtered castings. There was a high probability that filtration did affect cleaning room time. Filtration reduced cleaning room time from about 26 hours for unfiltered castings to 10 hours for filtered castings. Assuming a cleaning room cost of $33/hour and subtracting the cost of the filters, filtration results in savings of approximately $530 per casting.

The effect of filtration on weld repair time was also very significant. Filtration reduced the average weld repair time from 11 hours to 4 hours for a 65% reduction. Using a cost of $37 per hour for weld repair, this translates into savings of about $260 per casting.

Filtration had a very significant effect on cleaning room and weld repair time. Cleaning room time was reduced on average from 25 to 10 hours or about 60%. Weld repair time was reduced on average from 11 to 4 hours or about 65%. Filtration reduced total cleaning room and weld repair time by about 62% which translates to a savings of about $790 per casting.

Conclusions

Oxide macroinclusions have long been a major cost factor in the production of steel castings. These macroinclusions are typically composed of reoxidation products. Surface macroinclusion removal, weld repair, and reinspection add 20% to the cost of production. Subsurface inclusions not found and repaired in the foundry can cause costly disruptions in machining operations and result in poor component performance in service. Research has been conducted in several areas of steel foundry production where improvements in techniques would reduce the severity of surface macroinclusions. The areas included pouring, melting practice, ladle treatments, analog modeling of steel pouring and filling, and metal filtration.

Shrouding and submerged pouring to eliminate air exposure has been successfully used in continuous casting of steel. A submerged shroud pouring trial was conducted to determine if recent advances in shrouding technology would allow submerged pouring to be used in a steel foundry. The conventionally poured castings contained an average of 9.35 one-inch diameter circles of macroinclusions with a standard deviation of 4.3. Submerge poured casting contained an average of 0.30 one-inch diameter circles of macroinclusions with a standard deviation of 0.55. Cleanliness ratings show that submerge poured castings were dramatically cleaner than the conventionally poured castings.

A long-term trial has been conducted at three foundries to determine if causation between some variable or variables in the melting practice and pouring practice and average heat casting quality can be determined. The goal was to gain a better understanding of the influence of melting and pouring practice on casting quality and, subsequently, determine how to reduce the heat-to-heat variation. All three foundries showed improvement ranging from 40-50% in casting quality by manipulating the melting practice. Clean heats can consistently be produced if the oxygen concentration in the steel bath can be minimized through improved melting practice and reduced exposure of the steel to atmospheric oxygen during pouring and filling.

In 1993, Robert Shepherd presented a paper comparing acid and basic melting practices at Harrison Steel Castings Co. [*10*]. The trial lasted over a year and produced over 900 basic heats of various grades of carbon and low alloy steel. Shepherd determined that basic refractory practice reduced dirt defects in the castings. Calcium wire injection was found to significantly reduce dirt defects (32%) in the acid practice. This data was reanalyzed to try to determine the relative effect of melt practice and other factors on casting quality. The objective of this analysis was to determine the effect of calcium wire injection, refractory practice, and casting type on heat to heat average casting quality and variation in casting quality.

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A trial was conducted at a participating foundry to evaluate the effect of filtration on the cleanliness and repair time associated with the production of a stainless steel manifold [*11*]. Cleaning room and weld repair time were used as indicators of casting cleanliness. The purpose of this trial was to determine the filter performance, the cleanliness of the resulting castings, and the potential economic benefits of a large steel casting. The castings poured are near the upper limit weight range of steel parts that can be successfully poured through filters.

Filtration had a very significant effect on cleaning room and weld repair time. Filtration reduced total cleaning room and weld repair time by about 62% which translates to a savings of about $790 per casting. Cleaning room time was reduced on average from 25 to 10 hours or about 60%. Weld repair time was reduced on average from 11 to 4 hours or about 65%. Total cleaning room and weld repair time reductions saved the foundry about $1000 per casting.

These trials demonstrate that significant improvements in casting quality through the reduction of surface macroinclusions can be made with the use of current technologies. The foundry must identify the problem area to address and then use the technology available to eradicate the problem.

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CLEAN STEEL TECHNOLOGY - FUNDAMENTAL TO THE DEVELOPMENT OF HIGH PERFORMANCE STEELS

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ABSTRACT: The use of clean steel technology (low sulfur with calcium treatment for inclusion shape control) is a fundamental building block in the development of high performance plate steels. A brief review will be presented of the benefits of calcium treatment and its effect on non-metallic inclusions (sulfides and oxides) and reducing sulfur levels. During the past thirty years the requirements for low sulfur levels have been reduced from 0.010% maximum to 0.001% maximum. The effects of clean steel practices on specific properties will be reviewed including tensile ductility, Charpy V-notch and fracture toughness, fatigue crack propagation and hydrogen-induced-cracking resistance. Traditional low sulfur plate steel applications have included pressure vessels, offshore platforms, plastic injection molds and line-pipe skelp. More recent applications will be discussed including bridge steels, high strength structural steels to 130 ksi (897 MPa) minimum yield strength, 9% nickel steels for cryogenic applications, and military armor.

KEYWORDS: clean steel, low sulfur, inclusions, toughness, fatigue, steel properties

Over the last three decades, the cleanliness of structural steels has been improved enormously. The challenge to improve cleanliness has been presented by a variety of applications which have required improvement in mechanical and other properties. The level of sulfur has been the principal focus to improving the cleanliness and thus the performance of steels. In the 1970's, 0.010% maximum sulfur was felt to be clean plate steel. Today, 0.001% maximum sulfur level is required for an increasing number of applications.

There has been a significant amount of effort in inclusion control over the past 30 years. Lamellar tearing problems in welded structures in the 1960's was a major initial factor in demanding cleaner plate steels. This also resulted in a better

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understanding of the influence of nonmetallic inclusions on steel performance. Furthermore, the benefits of low sulfur and low inclusion contents were found to improve toughness, ductility and fatigue properties of steels and thus the resistance to failure in service. These improved performing steels are being utilized in a wide range of applications. Most often it is the applications that have pushed the steelmaking improvements towards cleaner steels.

Steel Inclusions

When inclusion control in steels is considered, the primary concerns are indigenous non-metallic inclusions, which precipitate as discrete phases during the solidification of molten steel, e.g., sulfides and oxides. These inclusions are influenced by the steelmaking techniques that are used in the steelmaker's melt shop, as well as other processing that is subsequently used. Some of these influences will be discussed in the following text. Although oxides and sulfides are of particular interest in steel, other inclusions can also play an important role. In steels that are not aluminum killed, silicates are an important concern. Also when nitride forming elements are used in alloying, there can be a significant influence of nitride inclusions. For purposes of this review, only aluminum killed steels will be considered and the importance of controlling aluminum oxide and manganese sulfide inclusions will be emphasized.

Characterizing these inclusions is an important part of identifying the influences of different steelmaking practices. Various metallographic methods have been used in these characterizations. Quantifying the inclusion content differences between steels is also of concern. Identifying the influence of these inclusions upon mechanical and other properties is also of interest.

The non-metallic inclusions in plate steel are significantly influenced by the steelmaking processes that are used. This is particularly the case with sulfide inclusions. Our experience has been related to electric arc furnace (EAF) produced steels and, therefore, the discussion will be directed toward this particular operation. In EAF steelmaking, the sulfur levels can be reduced through double slag practices and until approximately 20 years ago this was the primary method used to achieve the lowest sulfur levels for structural steels. With the development of ladle metallurgy practices, sulfur levels could be reduced outside the EAF through various techniques. One of the most popular methods that has been used is calcium treatment (CaT). It is possible to achieve very low sulfur steels (maximum sulfur levels as low as 0.001%S) in conjunction with other desulfurization techniques within or outside the EAF using CaT.

After molten steel has been refined through a ladle metallurgy station, it is teemed into ingots or cast using continuous casting processes. Both casting processes influence the inclusion content in the final product. In ingot casting, due to the long solidification period, there can be segregation of sulfur at the top of ingot and near the centerline. However, with lower sulfur steels, there is less of a concern for this problem. In continuous cast slabs, there can be problems with reoxidation inclusions collecting near the top quarterline of the slab. Through use of various argon shrouding techniques, reoxidation inclusions are minimized in both casting methods.

Calcium Treatment

The primary emphasis of this paper is on calcium treatment of plate steels. We have a great deal of experience using this practice over the past 20 years. Because calcium has a boiling point below steelmaking temperatures, it has been found important to use a powder injection or wire feeding method to add calcium compounds to the molten steel. Both processes involve adding calcium compounds to the bottom of the ladle and stirring the bath with argon. Calcium has a very strong affinity for both sulfur and oxygen and therefore the benefits of both lower sulfur and oxygen content are achievable. The recognition that calcium has some limited solubility in molten steel was the key discovery, which led to the use of calcium treatment of production steels [1]. With some calcium in liquid solution, the chemical reactions with sulfur and oxygen can be performed more efficiently, removing them from the molten steel, with the sulfide and oxide phases being absorbed by the slag cover.

The effectiveness of a calcium treatment may be influenced by a number of parameters. For example, these may include the amount and chemistry of the calcium compounds, effectiveness of deoxidation prior to treatment, various injection and stirring parameters, molten steel temperature and chemistry, tapping practices, ladle refractory composition, teeming and casting practices. Any of these may have an influence on the efficiency of calcium treatment and thus on the inclusion structure of the calcium treated steel.

The benefits of calcium treatment are best described through reviewing how manganese sulfides (MnS) and alumina inclusion clusters form in a steel [1]. In conventional steels, MnS inclusions have a low melting point and are among the last to precipitate in the solidifying steel and thus tend to accumulate in the interdendritic areas of the cast structure. By the addition of calcium, CaS phases form and rise out of the steel and are absorbed by the slag. The remaining calcium sulfide phases have melting points closer to those of steel and thus are more evenly distributed throughout the steel.

Alumina inclusion clusters form in conventional steels almost immediately after the addition of aluminum for deoxidation. These are very high melting point inclusions and they begin to rise in the molten steel, growing, contacting each other, and forming extensive three dimensional arrays. Upon calcium treatment, calcium combines with alumina inclusions in a fluxing reaction, which forms much lower melting point individual, liquid, complex inclusions, which rise more easily out of the molten steel. The more calcium present in the inclusion, the lower the melting point of the $CaO{\cdot}nAl_2O_3$ inclusions (Ca-aluminates) that will form. The composition of these calcium aluminates becomes a "telltale" signature of the efficiency of the calcium treatment practice.

The remaining inclusions in calcium treated steels tend to be duplex, calcium modified inclusions which resist deformation on hot rolling. The calcium modification of the sulfide phase makes them harder at hot rolling temperatures in comparison to the steel matrix. This is the basic building block for inclusion shape control. However, even more subtle differences can be detected in the inclusion structure of these steels; these are a direct result of the efficiency of calcium treatment.

Inclusion Shape Control

It is very important to have a number of techniques available to characterize the inclusion content and distribution in a steel. Each method can have a separate contribution and therefore improve the overall understanding of the inclusion structure. Light optical microscopy is still a very valuable tool for this evaluation. In fact, at times it is the most important. Figure 1 exhibits the typical inclusions in a conventional higher sulfur steel. MnS inclusions elongate both individually and as a cluster and are pancaked during the hot rolling process. The alumina inclusions do not individually deform, but the clusters do and further contribute to mechanical property reduction and anisotropy. Calcium treatment assists in lowering sulfur content and provides inclusion shape control and the removal of alumina inclusions as demonstrated in Figure 2. These enhancements can also be shown through use of fractographic evaluation of various mechanical test specimens from the sample steels.

Quantitative analysis of inclusions that are in a steel can be attempted through evaluation of mounted metallographic samples through use of manual techniques or using quantitative image analysis. The availability of microprobe analysis is also vital to understanding inclusions in steel. This is very important in identifying inclusions and provides important support in determining the efficiency of the steelmaking practice.

In studies of calcium treated steels, six classes of inclusions have been identified [2]. These are summarized in Figure 3. Although more than one class of inclusion is normally present in any particular steel, generally a trend is noted in an evaluation and it is very useful in establishing a semi-quantitative level of effectiveness of the calcium treatment process.

Through metallographic studies of the calcium treated steels, it has been established that the presence of elongated inclusions (Classes C, E or F) and clusters of inclusions (Class D and F) are indicators of a poorer level of calcium treatment and of inclusion shape control. The optimum inclusions, Classes A and B, have unique chemistry. Figure 4 demonstrates the bull's-eye appearance of Class B and the intermingled sulfide and aluminate phases of Class A. The Ca-aluminate in Class A inclusions tend to have the highest Ca content of any Ca modified inclusions [2]. The intermingled nature of the Class A indicates that the sulfide and aluminate phases solidified at the same time, while the Class B aluminate phase formed first and became the nucleus for the sulfide phase. The presence of magnesium is a result of pick-up from refractory systems.

In early studies of calcium treated steels, the measurement of calcium content was used as an important tool in indicating quality of the steel. However, calcium measurement is only detected if it is present in inclusions, since there is no calcium in solid solution in steel. Therefore, if there is a very high level of inclusions in the steels that are calcium treated, then the measured calcium level may be high. If methods are used whereby inclusion content in general is reduced, these could show a lower calcium content. Therefore, calcium level alone is not always a good measure of effectiveness of calcium treatment [2].

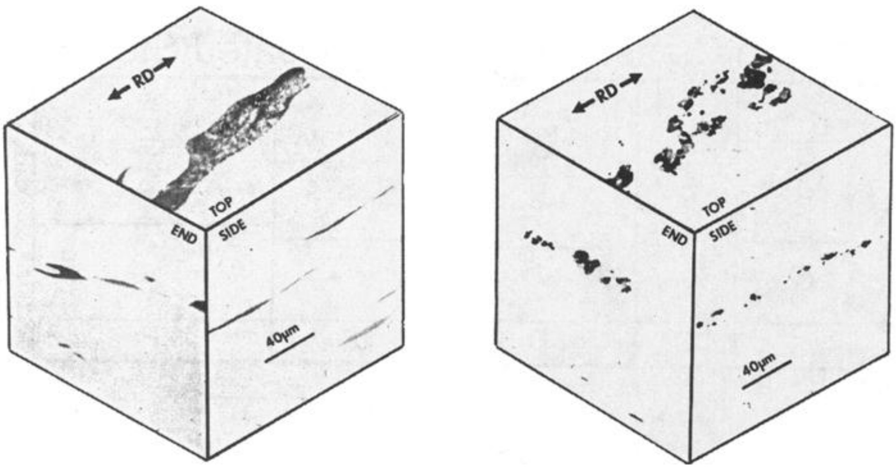

FIG. 1 -- *Composite of light photomicrographs showing Type II MnS inclusions (left) and Al_2O_3 inclusion clusters (right) in conventionally produced (CON) steel.*

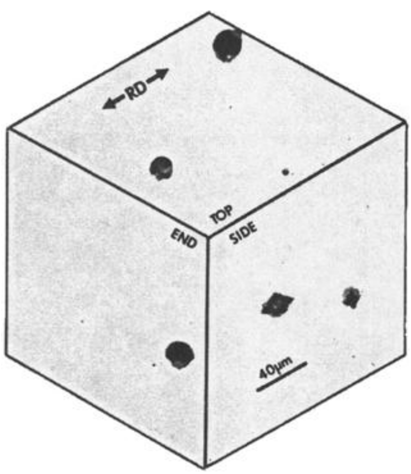

FIG. 2 -- *Composite of light photomicrographs displaying typical calcium modified inclusions in a CaT steel.*

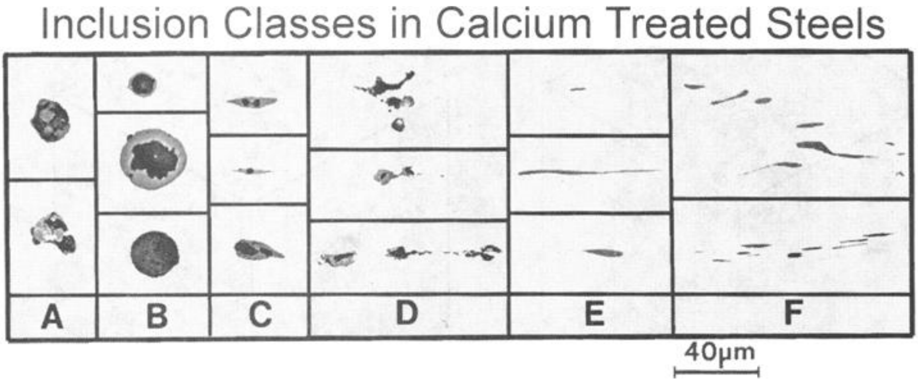

FIG. 3 -- *Six inclusion classes identified in calcium treated steels [2].*

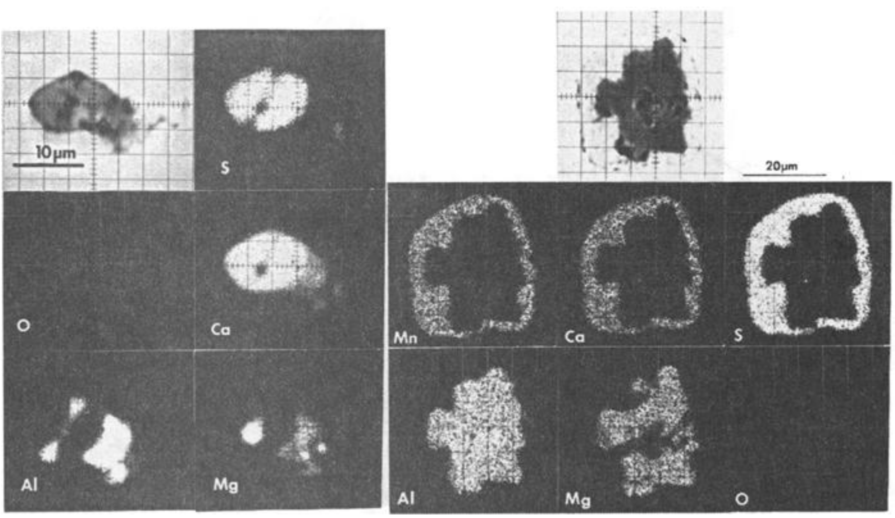

FIG. 4 -- *Energy dispersive x-ray maps of Class A and Class B inclusions displaying intermingled nature of Class A (left) and bull's-eye appearance of Class B (right).*

Effects on Mechanical Properties

Cleanliness and inclusion content can have a significant effect on mechanical properties depending on the property and the testing orientation. In any study of mechanical properties of steels with varying inclusion contents, it is very important to look at a number of testing orientations. Figure 5 provides the orientations that can be used. These orientations range from three testing orientations for tensile testing and up to six orientations for Charpy-V-notch (CVN) and fracture toughness, and fatigue crack growth rate testing. The effect of improved cleanliness on these properties is demonstrated in Figure 6. This figure summarizes the comparison testing of two A588, 3" (76 mm) thick plate steels, one conventionally produced (CON) with a 0.020% sulfur level and the other calcium treated (CaT) with a 0.003% sulfur level. Of particular note is the major differences in the through-thickness (S,ST,SL) testing orientations for all types of testing. The CaT steel also showed significantly improved upper shelf toughness in all testing orientations whether measured by CVN impact, dynamic tear (DT) or J-Integral elastic-plastic (J_{Ic}, T - tearing modulus) fracture mechanics tests. Fatigue crack propagation (FCP) threshold results require analysis for closure-correction to show the inclusion effects at very low crack growth rates. These results are discussed in more detail elsewhere [3].

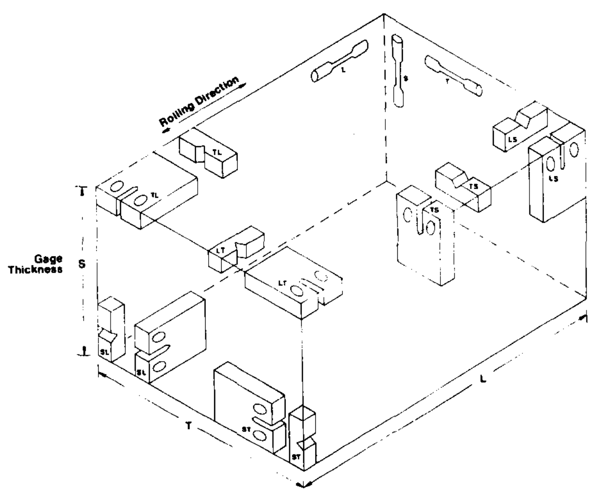

FIG. 5 -- *Schematic drawing showing the specimen orientations and designations per ASTM E399. Actual test location varies with test program.*

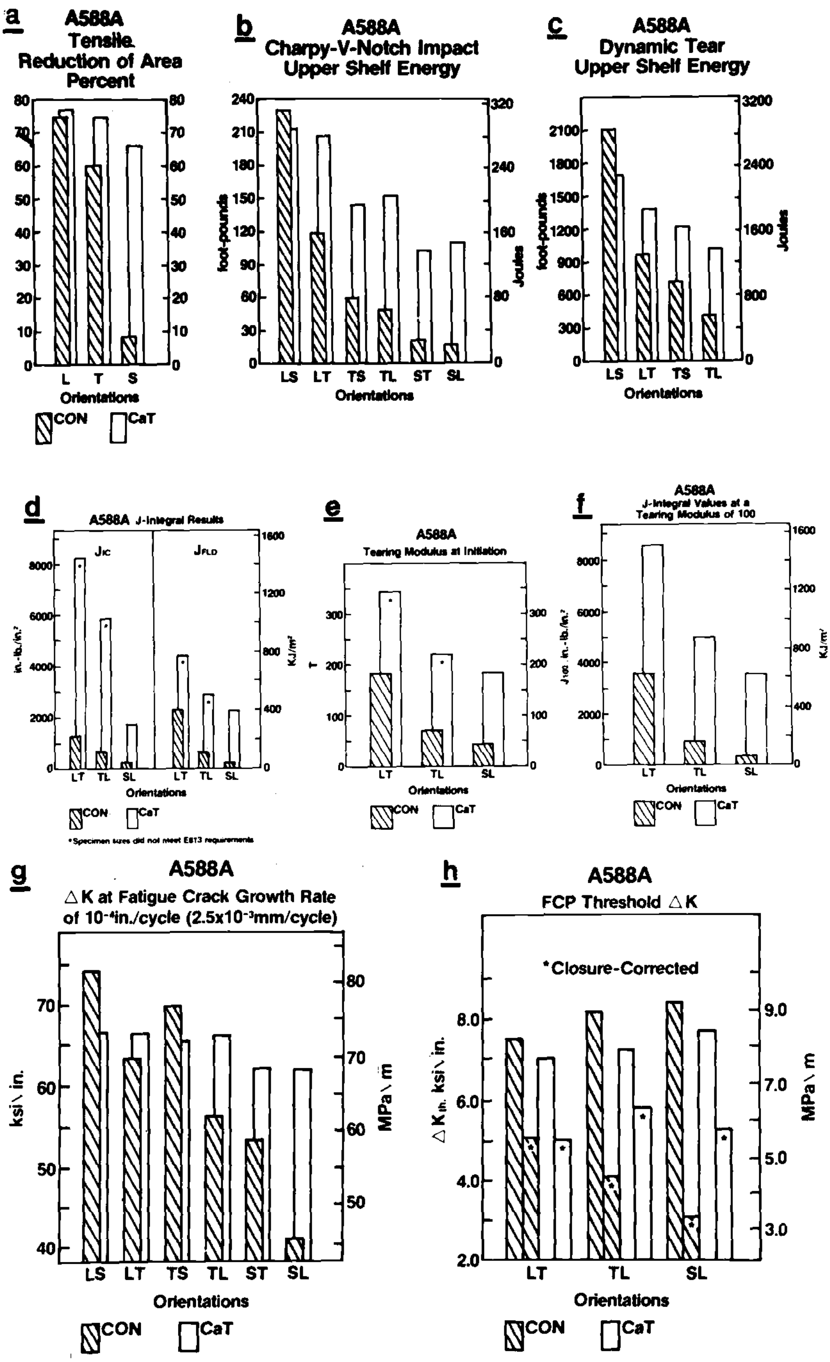

FIG. 6 -- *Summary bar graphs of comparison of CON and CaT Quality ASTM A588A.*

The higher inclusion content in the CON steel certainly influences the results shown in Figure 6. Of further importance is the clustering of the elongated and pancaked sulfide and oxide inclusions in the CON steel. Fractographic analysis of any of the tensile, toughness or FCP test specimens, particularly for the through-thickness orientations (S,ST,SL) dramatically reveals these clustered inclusions, as shown in Figure 7. The clustering of inclusions in clean steels can also lead to a degradation in properties. Figure 8 demonstrates this in hydrogen-induced-cracking (HIC) testing in A516 normalized, calcium treated carbon steel. HIC-testing involves exposing standard size samples to an acidic, hydrogen charging solution. Cracking initiates at inclusions and propagates along inclusion clusters [4].

Inclusion clusters can provide improvements in test results in certain orientations. For example, the LS orientation gave higher results for the CON steel in Figure 6 for CVN, DT and FCP tests because the elongated and pancaked clusters of inclusions act to blunt and deflect the cracking during the test.

High Performance Steels

In today's markets for steel plate there is a growing demand for improved steels to meet more challenging requirements. Clean steel practices have previously been required for lamellar tearing resistance for offshore platforms and machinery, improved toughness in line-pipe skelp and pressure vessels and better ultrasonic cleanliness in plastic injection molds. New high performance steels are being developed starting with very clean steel as the fundamental building block. A brief discussion of several examples is detailed in the following section. Table 1 gives the typical chemistries and minimum yield strengths of the steels to be discussed, as well as those referred to previously.

TABLE 1--*Example chemistries of steels discussed.*

ASTM Grade	C	Mn	Cu	Ni	Cr	Mo	Other	Y.S.
A588	0.15	1.11	0.29	0.19	0.59	0.05	0.06 V	50(345)
A516	0.22	0.92	0.08	0.05	0.06	0.01	...	38(262)
A709-HPS 70W	0.10	1.20	0.32	0.31	0.54	0.07	0.06 V	70(483)
A553	0.04	0.60	0.10	9.10	0.06	0.01	...	85(586)
A656-80	0.05	1.33	0.13	0.13	0.09	0.29	0.09 Cb	80(552)
LQ-130*	0.17	1.45	0.11	0.11	0.45	0.45	...	130(897)
HY-80*	0.16	0.31	0.12	3.12	1.57	0.54	...	80(552)

* LQ-130 - Bethlehem Lukens Plate grade in development
HY-80 - U.S.Navy specification MIL-S-16216

Y.S. - minimum yield strength, ksi (MPa)

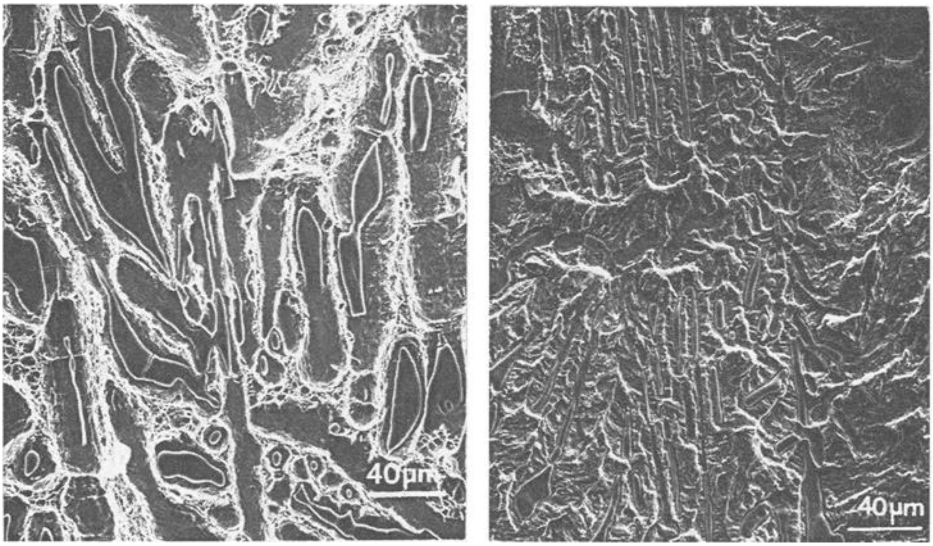

FIG. 7 -- *Scanning electron microscope fractographics exhibiting MnS inclusion clusters on SL oriented CVN specimen (left) and SL oriented FCP sample (right).*

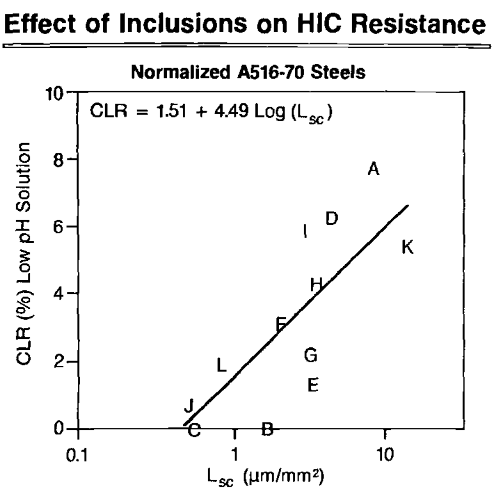

FIG. 8 -- *HIC resistance measured by crack length ratio (CLR) of normalized A516 related to inclusion stringer and cluster length factor L_{SC} determined metallographically for twelve plates.*

Bridge Steels

Traditional bridge steels are produced to ASTM A709, which has grades with yield strength level minimums of from 36 to 100 ksi (248-689 MPa). The toughness and welding practices for these steels have been based on the capabilities of these steels produced with 0.050% maximum sulfur levels and carbon contents at the higher ranges of mill experience. The Federal Highway Administration funded a research effort with the steel industry to develop high performance bridge steels with improved weldability and toughness. The result of this effort was a grade designated A709 HPS-70W with the nominal chemistry shown in Table 1 [5]. This quenched and tempered, weathering steel is produced with a sulfur level maximum of 0.005%. The carbon content has also been significantly reduced from a 0.17% maximum to 0.105% maximum. Bridges have been fabricated of this new grade and benefits in weldability realized. The improvements in the CVN toughness are summarized in Figure 9. These enhancements may make possible more aggressive design approaches in the future.

9% Nickel Steels for Cryogenic Vessels

Storage of liquified gases utilizes a variety of steels depending on the working temperature required. A553, 9% nickel steel is used at very low temperatures and is tested for CVN toughness at -320°F (-196° C). Over the past 20 years there has been continuing pressure to increase the required CVN toughness levels for improved design safety. This has required modifications to the melted chemistry. These changes and the resultant improvements in CVN impact properties are exhibited in Figure 10. Currently, sulfur maximum levels of 0.001% and low carbon and higher Ni levels are required for the most stringent CVN specifications.

High Strength As-rolled Steels

Steels used in the fabrication of construction and mining equipment have been increasing in strength to provide weight reduction and improved service performance. Traditionally higher strength levels required quenching and tempering (Q&T) heat treatments. However, advances in controlled-rolling technology have allowed development of steels to 80 ksi (552 MPa) minimum yield strength. ASTM A656 Grade 80 is the most popular plate steel grade for this application. Although A656-80 has a 0.035% maximum specified sulfur level, it has been traditionally been produced to 0.010% maximum sulfur to provide optimum toughness and lamellar tearing resistance. Lukens recently installed a Steckel mill, which produces plate in coiled form [6]. The nature of the coiled production route is that there is considerable rolling in one direction and thus a significant potential for developing property directionality. Thus, to provide the highest level of transverse CVN toughness, tighter controls on cleanliness are required. We are now producing this grade with 0.005% maximum sulfur. The results of this control are shown in Figure 11. The modest difference between longitudinal and transverse CVN results for a slab with a 24:1 reduction is a testament to the benefits of a lower sulfur level and inclusion shape control.

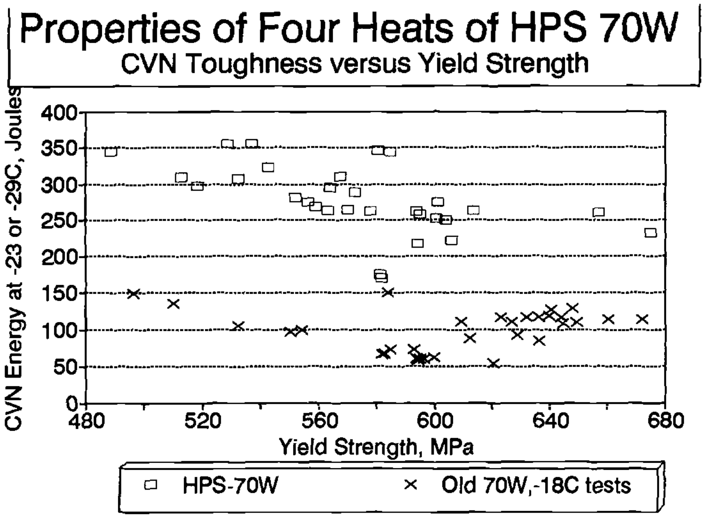

FIG. 9 -- *CVN data versus yield strength for ASTM A709-HPS 70W compared to traditional grade A709-70W.*

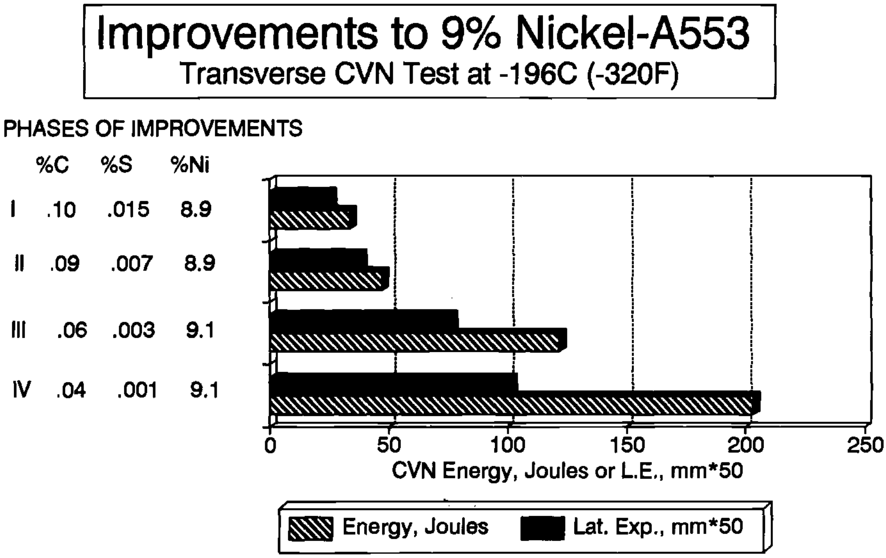

FIG. 10 -- *Improvements made to A553 steel and enhancements to CVN properties.*

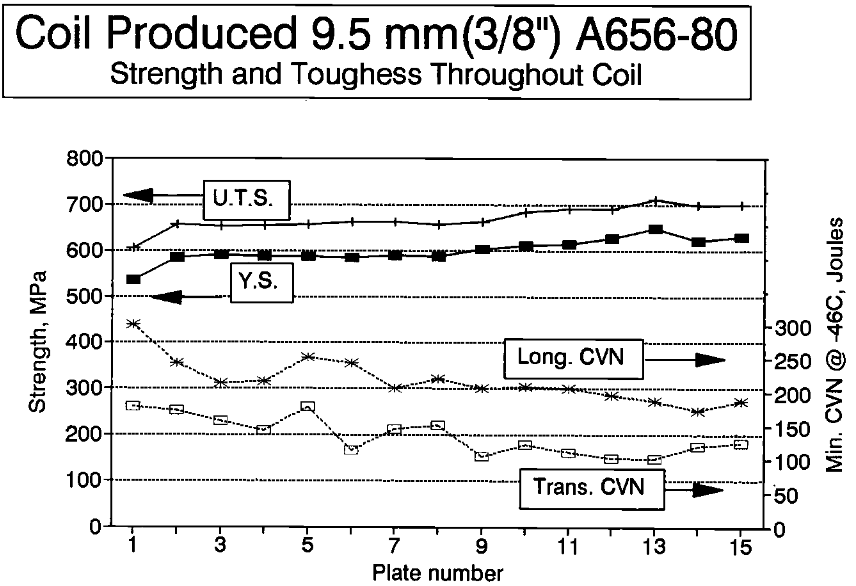

FIG. 11 -- *Strength and CVN results throughout coil of Steckel mill rolled A656-80.*

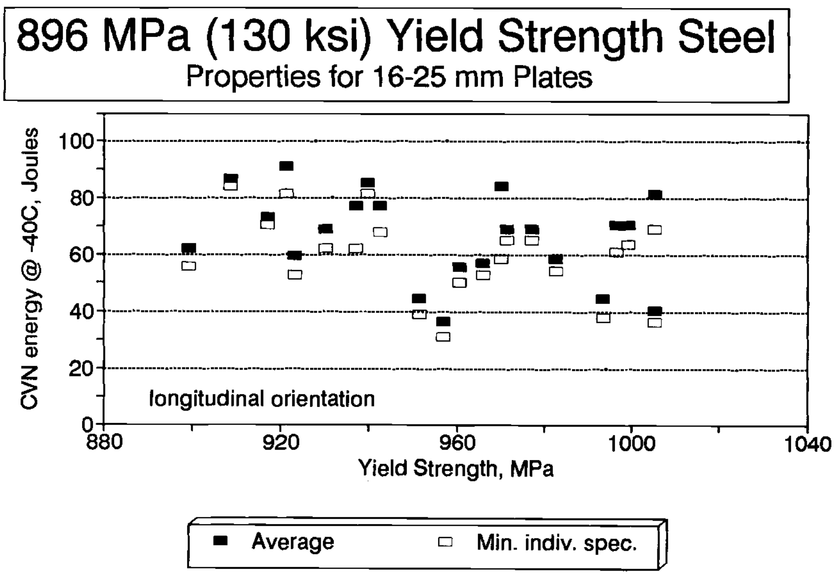

FIG. 12 -- *Summary of CVN and tensile data for Bethlehem Lukens Plate LQ-130 steel.*

Higher Strength Heat Treated Steels

With the increasing use of the A656-80 type steels there has been an accompanying demand for Q&T steels above the traditional 100 ksi (690 MPa) minimum yield strength level. Currently this demand has come for 130 ksi (896 MPa) minimum yield strength. Bethlehem Lukens Plate grade LQ-130 was developed for this application. To meet the CVN impact toughness requirements of equipment manufacturers, we have found it is necessary to produce this grade to a 0.001% maximum sulfur level. Figure 12 provides a summary of some of the latest results of this development.

U.S. Navy Armor Steels

The Navy specification for 80 ksi (552 Ma) minimum yield strength armor plate, MIL-S-16216 (HY-80), was developed in the 1950's for use on submarines. Specification requirements added over the years included CVN impact toughness testing at -120°F (-84 °C). However, the Navy was concerned whether the CVN test adequately represented the toughness behavior for applications where explosive events must be survived. The Naval Research Laboratory developed the dynamic tear test at -40 °F (-40 °C) as a more reliable quality control test for this challenging application. To meet this rigorous test, more control of the steelmaking process was required. Thus the latest specification requires a 0.008% maximum sulfur level with calcium treatment for inclusion shape control. The benefit of this change is displayed in Figure 13.

HIC-Tested A516 Steels

As discussed previously, normalized A516 steels for process vessels, in sour or hydrogen sulfide service require excellent cleanliness to pass specified HIC testing. This application continues to be one of the most demanding for clean steel production practices. Depending on requirements, either 0.002% or 0.001% maximum sulfur are dictated. The ability to consistently meet these levels is demonstrated by the distribution of sulfur levels shown in Figure 14 for the latest 100 heats produced for this application.

Summary

The preceding provided a review of the clean steel technology developed to allow the development of today's high performance steels. Low sulfur steels with inclusion shape control have been found to provide improved ductility, toughness, fatigue properties, as well as other behavior such as in HIC-testing. Today the need for these steels is required not only for special situations, but also for everyday structural applications such as bridges and construction equipment. We expect this demand will continue into the next century.

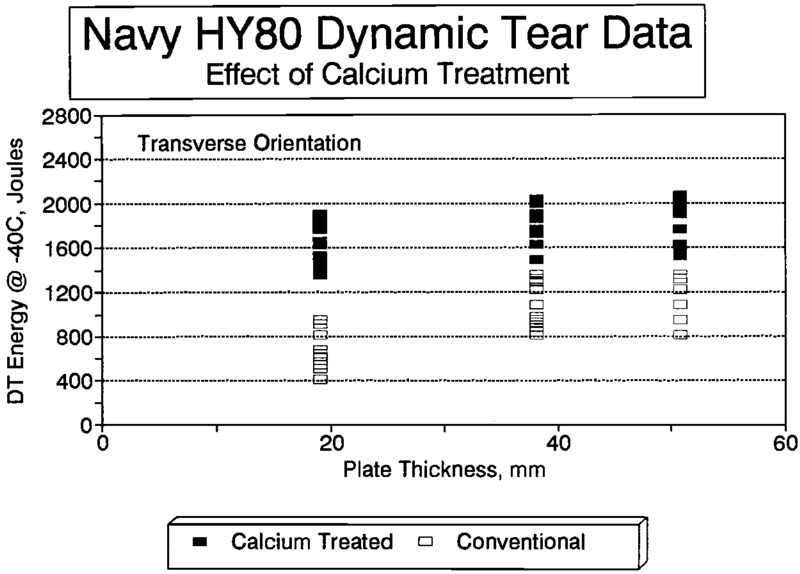

FIG. 13 -- *Benefits of using calcium treatment in HY-80 for improved DT results.*

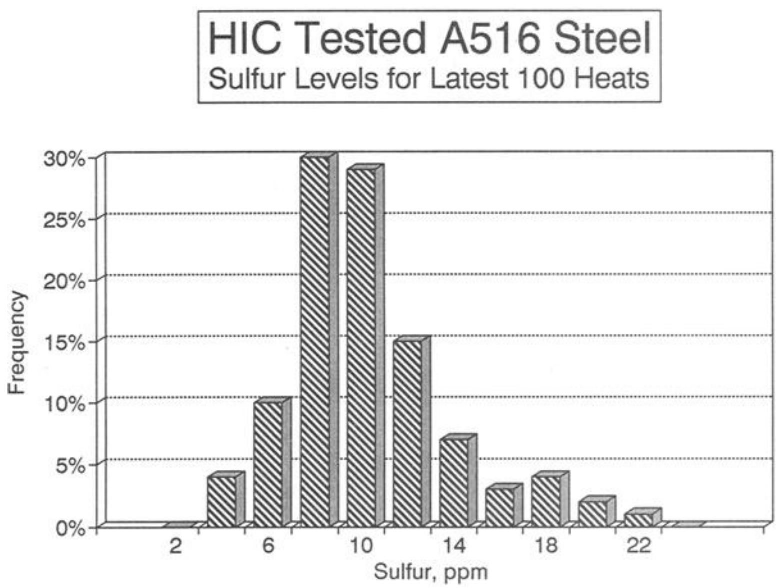

FIG. 14 -- *Distribution of sulfur levels for latest production of HIC-Tested A516. Testing according to latest ASTM standards.*

Acknowledgments

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SULFUR CONTENT OF CARBON STEEL PLATE MATERIAL FOR DISHED END MANUFACTURE BY COLD SPINNING*

REFERENCE: Dutta, T., Chandawale, R.G., and Vanchinath, S.A. **"Sulfur Content of Carbon Steel Plate Material for Dished End Manufacture Cold Spinning,"** *Advances in the Production and Use of Steel with Improved Internal Cleanliness*, ASTM STP 1361, J.K. Mahaney, Jr., Ed., American Society for Testing and Materials, West Conshohocken, PA, 1999.

ABSTRACT: Over many years SA 516 Gr.70 plate material is being used for the construction of boilers and pressure vessels and has become a standard of the industry. However a typical failure of this material during dished end manufacture has troubled the manufacturer on and off. Many times lamellar separation takes place along the central line of the thickness visible at the edges of the dished ends after cold spinning.

In this present study we have carried out a customized shear test to ascertain the effect of sulfur content on the susceptibility of the SA 516 Gr.70 plate material to fail by shear along the plane of segregation.

This study indicated that the presence of central segregation is a critical factor to induce reduction in the shear strength. As the sulfur content goes down the segregation line disappears and the shear strength also increases. At 0.01% maximum sulfur best results are obtained. It was supported by the field observation of failed dished ends, where failures are observed with plates with typical sulfur content of 0.02% and above.

KEYWORDS: cold spinning, lamellar separation, segregation, shear strength

One of the most popular carbon steel plate material specification for boiler and pressure vessel application is SA 516 Gr.70, Pressure Vessel Plates, Carbon Steel, for Moderate- and Lower-Temperature Service (specified chemical analysis is given in Table 1 for reference). Over the years it has become the standard of the industry. But on and off the manufacturers are plagued by the occurrence of lamellar separation during the cold spinning operation of the dished ends. These separation becomes visible at the edges or the near the edges when subjected to ultrasonic examination. These results into rework or altogether scrapping of the dished ends. The failure was somewhat controlled by carrying out ultrasonic testing as per A435, Straight-Beam Ultrasonic Examination of Steel Plates or A578, Straight-Beam Ultrasonic Examination of Plain and Clad Steel Plates for Special Applications, of the plate raw material before

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* This paper was not presented at the Symposium.

cold spinning of the dished ends and ensuring sound raw material without lamination. But even then these measures didn't altogether eliminate the occasional failure of dished ends by lamellar separation.

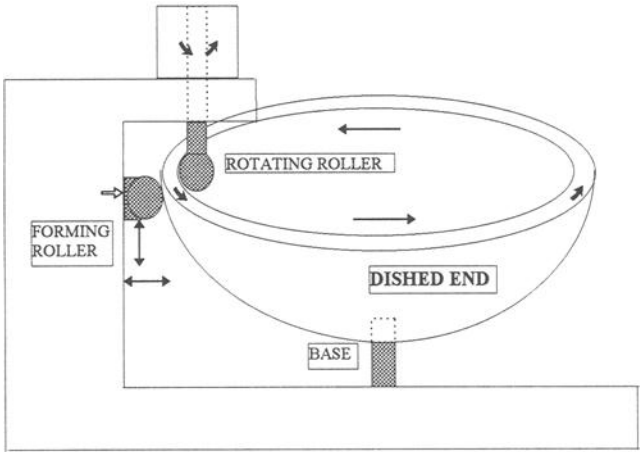

FIG.1 *Showing a typical spinning arrangement for a dished end.*

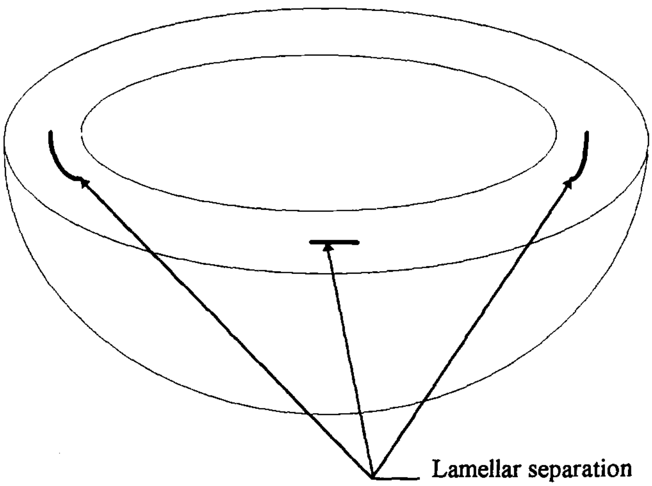

FIG.2 *Showing lamellar separation in a dished end.*

Cold spinning is a very widely used process of forming of dished ends. With this technique good dimensional tolerance can be achieved with low cost. But it demands high structural integrity of the raw material because of the severity of the shear stresses that are induced between the adjacent layers during the spinning operation.

FIG.1 shows a typical cold spinning arrangement and FIG.2 shows the schematic figure of lamellar separation.

Steel plates made through the conventional route of Open-hearth/ Basic Oxygen/ LD Process ➡ Ingot / Continuous cast slab ➡ Hot rolling, has a tendency to have centrally segregated band of low melting point composition primarily containing sulfides[1]. To reduce this segregation modern techniques of reduction in sulfur in the ladle, induction stirring etc. are adopted. For a purchaser the parameter he can relate to while specifying a composition is sulfur content and that must be controlled to a maximum, to minimize the chances of lamellar separation during cold spinning of dished ends.

TABLE 1 *Showing the specified chemical analysis of SA 516 Gr.70*

Carbon	Manganese	Silicon	Sulfur	Phosphorus
0.28 max.	0.85 - 1.20	0.15 - 0.40	0.035 max.	0.035 max.

EXPERIMENTAL METHOD

Since shear is the prevalent mode of failure during cold spinning it was decided to design a shear test to study the effect of central segregation on the shear strength of the plate material along the plane of segregation. Figure 3 shows the shear test specimen used for the experiment.

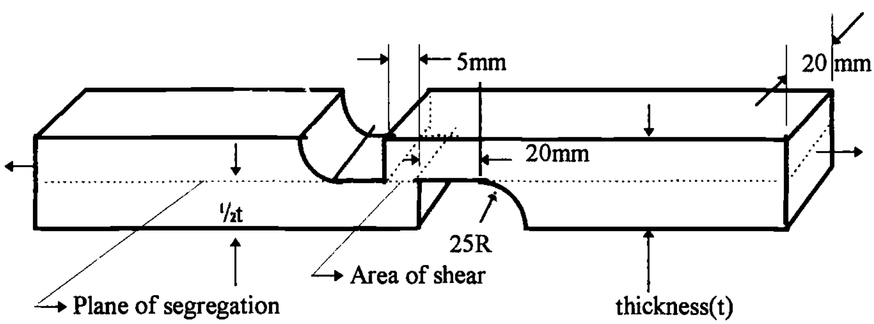

FIG.3 *Showing the sketch of the shear specimen.*

Specimens were made from heats of different sulfur contents between 0.009% to 0.020%. Before carrying out shear test the specimens were macro etched with 10% nital to reveal the segregation line, if any. The specimens were subjected to tensile load in their longitudinal axis in an universal tensile testing machine, to induce shear stresses in the area marked as 'Area of shear' in FIG.3. The shear strength was calculated by dividing the breaking load by the area of shear. In all cases the fracture took place along the plane of shear.

RESULTS

The observations are tabulated in Table 2 and Figure 4 shows the effect of %sulfur on shear strength.

TABLE 2 *Showing the experiment results*

SR NO	HEAT NO.	PLATE NO.	% S	SHEAR STRENGTH kg/mm^2	REMARKS
1	6731	0109-1	0.009	44.29	No segregation
2	3088	0111-1	0.010	44.30	No segregation
3	41008	32865-2	0.014	34.79	segregation visible
4	41008	32865-2	0.014	39.15	segregation visible
5	41008	37872-1	0.014	38.87	No segregation
6	41008	37872-1	0.014	42.08	No segregation
7	44650	57320-3	0.020	33.75	segregation visible
8	44650	57320-3	0.020	37.78	segregation visible

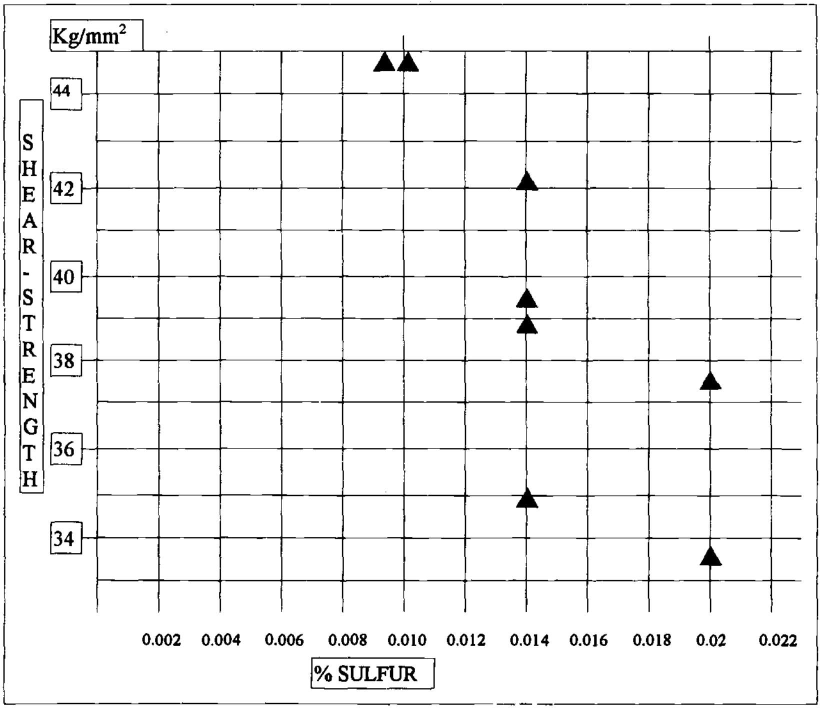

FIG.4 *Showing the effect of %sulfur on the shear strength.*

CONCLUSION

From the experiment it became evident that the drop in shear strength has got a direct relationship with the increase in the sulfur content of the steel and the presence of segregation line. Though, as expected, at the same sulfur content the shear strength varied due to the presence or absence of segregation line. At sufficiently low % sulfur (0.01 max.) the segregation line disappeared and the shear strength also was highest. This is in line with our actual experience with carbon steel dished ends, failed due to lamellar separation during cold spinning; the failure rate which was as high as 5-10%, dropped to nil after introduction of the control on %sulfur within 0.01 for dished end manufacture.

REFERENCES

[1] American Society of Metals, 1984, *Metals Handbook® Desk Edition,* ASM, Metals Park, Ohio, pp 22-7.