MICROCLEANLINESSS STUDIES OF LOW ALLOY AND CARBON STEEL, POWDERS INTENDED FOR POWDER FORGING APPLICATIONS

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Abstract

The results of ten years of experience aimed at continuously improving the quality of powders for forging applications are reviewed. An automated image analysis procedure for inclusion assessment is described which evaluates the inclusion content of samples and defines fragmented inclusion clusters on the basis of a concept of near neighbor separation. A task group approach was used for cause-and-effect analysis to determine the source of different inclusion types and eliminate and/or reduce their incidence. The benefits of this approach are demonstrated by the significant reduction effected in inclusion levels.

Introduction

Powder forging, which is often referred to by the acronym P/F, is a process in which powder metal preforms are hot formed in confined dies. A review of the powder forging process has recently been published in Volume 14 of Metals Handbook (1).

Powder forging is a natural extension of the conventional press and sinter (P/M) process, which has long been recognized as an effective technology for producing a great variety of parts to net or near-net shape. The porosity present in pressed and sintered ferrous P/M materials masks the effect of inclusions on mechanical properties. In contrast, the mechanical properties of near fully dense powder forged materials are strongly influenced by the composition, size distribution, and location of nonmetallic inclusions (2-5). Samples of powder intended for powder forging applications are, therefore, subject to rigorous quality assurance procedures to ensure they meet acceptable levels of microcleanliness (6,7).

The term microcleanliness and the concept of near neighbor separation of inclusions will be explained in this paper, which will summarize the results of ten years of experience aimed at continuously improving the quality of powders for forging applications.

Microcleanliness - The Near Neighbor Concept

Compared with wrought steels, only a limited amount of material flow is present in powder forged components. Inclusion stringers common to wrought steel are, therefore, not found in powder forged materials. Standard inclusion assessment procedures for wrought steels, as reviewed by Vader Voort (8), are therefore not appropriate for powder forged materials.

Inclusions representing a few of the different morphologies encountered in powder forged materials are presented in Figures 1 through 4. The fragmented nature of these inclusions means that their size determination is somewhat more complicated than for a solid inclusion such as the one shown in Figure 5. The maximum Feret's diameter was selected to measure inclusion size. A Feret's diameter is any caliper diameter, as illustrated in Figure 6. Having selected the measurement parameter, the question of where fragmented inclusions, such as the ones in Figures 1 through 4, start and end had to be addressed. In Figures 1 and 2 the individual particles are clustered, and it is possible for the human eye to recognize that they might belong to a single agglomerate. However, such a pattern is more difficult to discern in Figures 3 and 4. It was realized that, in the case of Figures 1 and 2, the human eye is making a pattern recognition judgment based on the interparticle spacing. If an inclusion is within a certain distance from its neighboring particles, it is quite reasonable to consider it a member of that inclusion cluster or agglomerate. This concept of near neighbor separation was adopted for inclusion assessment, and particles within 30 micrometers of one another are considered part of the same inclusion. The concept is illustrated schematically in Figure 7.

Counting and sizing inclusions on the basis of this near neighbor concept results in a higher estimate of the number of inclusions of a given size than would have been obtained if the individual particles of a fragmented inclusion were measured. When reviewing inclusion data, it is important to know on what basis the inclusion size has been determined. Some methods of assessment may make materials appear to have a much lower inclusion content than would be determined by other methods (8,9). While fragmented inclusions may not be as detrimental to mechanical properties as solid exogenous inclusions, sizing fragmented inclusion clusters defined on the basis of near neighbor separation is considered more appropriate than sizing the individual particles that constitute the inclusion cluster. Thirty small inclusions clustered to form an inclusion with a maximum Feret's diameter of 100 micrometres are likely to have a more significant effect on mechanical properties than the same thirty small inclusions randomly distributed throughout the sample.

Inclusion Assessor Procedure

Samples of powders intended for forging applications are repress powder forged under closely controlled laboratory conditions. The 25-mm diameter forging are austenitized and quenched prior to being sectioned longitudinally and mounted for metallographic sample preparation. All metallographic preparation is done on a Struers Abrapol automatic grinder/polisher. Automatic polishing is essential to provide the high quality scratch-free and stain-free surface required for inclusion assessment. Grinding is accomplished using silicon carbide abrasive discs in grit sizes 80 to P1200 (180 µm to 15.3 µm median diameters). This is followed by diamond polishing using 6 µm (Pellon - hard fiber cloth), 3 µm (Dur - hard cloth), and 1 µm (Nap -soft cloth) sprays successively. Ultrasonic cleaning, washing, and drying is performed between each polishing step.

Two cylindrical forging are prepared from each powder sample, and three longitudinal sections are prepared for metallographic examination. An area of at least 120 mm^2 is examined at 100X magnification on each section, giving a total area covered of greater than 360 mm^2 · Inclusions are sized according to their maximum Feret's diameter with the near neighbor concept applied. A record is kept of any inclusions greater than 100 µm or 150 µm and the results presented in terms of two parameters, F_4 and F6, which are defined as follows:

F_4 = the number of inclusions $100 \mu m$ per $100 mm^2$

F_6 = the number of inclusions $150 \mu m$ per $100 mm^2$.

This visual assessment procedure was applied for a number of years to all powders intended for powder forging applications. Periodically, the same samples were assessed by different metallographers to ensure comparability.

Automated Image Analysis

It soon became apparent that the nature of the inclusion assessment test and the large number of samples to be examined were ideal candidates for automated image analysis. Development of an automated image analysis procedure took approximately one year. Samples were evaluated by both the visual assessment and image analysis procedures for a period, before transferring entirely to image analysis just over four years ago. The automated approach is preferred because it is not sensitive to operator subjectivity, and it can be used routinely to obtain a wider range of data on a reproducible basis. For example, the maximum Feret's diameter of any inclusion k 100 ~m is recorded along with a measure of the percentage detection. This latter parameter is an indication of whether the inclusion is solid, such as the one illustrated in Figure 5, or fragmented - and to what extent, such as the ones shown in Figures 1 through 4.

In addition to being classified according to their size and morphology, the inclusions are further subdivided according to their type; e.g., slag, oxide, sulfide, etc.

The automated image analysis procedure has been reviewed previously (10). A Lettz TAS Plus image analyzer equipped with an automatic scanning stage and auto-focus is used as shown in Figure B. In essence, an image analyzer consists of a good quality metallurgical microscope fitted with a video camera which displays the live images on a CRT screen. The video image is assessed in terms of its gray level characteristics, black and white being extremes on the available scale. The basis of the technique is to transform the detected image, on the basis of near neighbor separation, into a form where fragmented inclusions are measured as individual particles without distorting the other particles within the field of view. This is made possible by feature-specific operations carried out under microprocessor control.

The Leitz TAS Plus uses a hexagonal raster to display and analyze the detected image. The sequence of image transformation steps used is illustrated in Figure 9 (a-h) and Figure 10 (a-h) for a "spotty oxide" and a discontinuous sulfide, respectively. This rather complex sequence is necessary to ensure that particles which have been joined on the basis of the prescribed near neighbor separation are not subsequently separated and eliminated from the analysis. A simple dilation/erosion transformation sequence will work for lacy inclusions, but will not correctly classify diffuse "spotty" inclusion (11).

Identification of Inclusion Types and Their Source

Quantifying the level of nonmetallic inclusions is only the first step in the process of continuously improving the microcleanliness of the powders. In order to reduce the level of nonmetallic, we need to identify inclusion types and determine their most likely source. Inclusion chemistry can be determined by energy dispersive X-ray

analysis or wavelength spectroscopy in a scanning electron microscope (SEM). The use of energy dispersive X-ray analysis to identify an inclusion is illustrated in Figure 11 (a-d).

The microcleanliness program is only one facet of an overall quality assurance management system, which employs statistical process control and statistically based design of experiments to ensure product consistency (7,12). A multi-disciplinary approach, using task groups with members drawn from different departments within the company was used for cause-and-effect analysis of the problem. For example, Q.C. Metallurgy, Operating personnel, and the Laboratory Services group worked on identifying inclusions and their source. R&D personnel cooperated with Q.C. Metallurgy and Laboratory Services to determine the effect of inclusions on various mechanical properties.

With a knowledge of inclusion chemistry and morphology, the task groups were able to compare this data with their knowledge of arc furnace steelmaking practice, refractory practice, ladle refractories, tundish refractories, and powder collection and handling procedures throughout the steel atomizing process, illustrated in Figure 12 in order to determine the likely sources of such inclusions. A designed experiment was then carried out to confirm their analysis and pinpoint the inclusion source. Procedures and practices were then modified in order to eliminate and/or reduce the incidence of specific inclusion types. Statistical analysis techniques were used to review the data because it was recognized that while it is possible to produce a few clean lots of powder under ideal circumstances, it is the production of material with low inclusion content over an extended time frame that is the real objective.

	Typical Composition (Wt. Z)		
Alloy	**Mn**	**Ni**	**Mo**
Ancorsteel 4600V	0.10/0.25	1.75/1.90	0.50/0.60
Ancotsteel 2000	0.25/0.35	0.40/0.50	0.55/0.65
Ancotsteel 1000P/F	0.10/0.25	--	--

Table 1: Typical chemical composition of the powders examined.

The Results of the Microcleanliness Program

The benefits of themicrocleanliness program are first illustrated for Ancorsteel 4600V; water atomized low alloy steel powder with a typical chemical analysis given in Table 1. Ancorsteel 4600V was the first powder evaluated by the microcleanliness program. In Figure 13, the square root of the parameter F_4 is plotted against the cumulative percentage frequency of observation for a given plotted value on a probability scale. The data illustrates the improvement in cleanliness achieved between 1981 and 1987. In Figure 13, the F_4 data represents all inclusion types. This form of data analysis is also applied to the various inclusion families - slags, oxides, sulfides, etc. Figure 14 shows F_4 oxides plotted on the same basis, and again illustrates the improvement effected between 1981 and 1987. Figure 15 summarizes the F_6 data for Ancorsteel 4600V and shows the improvement made from 1981 to 1987.

The experience gained with Ancorsteel 4600V was applied to another water atomized, low alloy steel powder - Ancorsteel 2000. A comparison between the years 1985 and 1987 for F_4 and F_6 inclusions is presented in Figures 16 and 17. Sulfides are the predominant inclusion type in Ancorsteel 2000, and it is the sulfide content that causes the higher F_4 and F_6 values compared with Ancorsteel 4600V.

The water atomized iron powder, Ancorsteel 1000P/F, has benefited from the application of

statistical process control concepts throughout its development. The average F_4 and F_6 values for Ancorsteel 1000P/F are 0.66 and 0.20, respectively.

The Effect of Inclusion Content on Rolling Contact Fatigue Performance

In addition to the program aimed at reducing the inclusion content of powders, a program was run concurrently to determine the influence of inclusions on the mechanical properties of powder forged materials. The rolling contact fatigue (RCF) test was performed on samples produced from Ancorsteel 4600V and Ancorsteel 2000 low alloy powders. The RCF test is an accelerated test aimed at ranking materials according to their potential performance in bearing applications. RCF testing was carried out according to the procedure described by Glover (13). Sample preparation procedures have been detailed elsewhere (1). The rolling contact fatigue testers used are shown in Figure 18, and a detail of the specimen arrangement is given in Figure 19. The results of the RCF testing on the Ancorsteel 4600V and Ancorsteel 2000 samples are summarized in Figures 20 and 21. The results of testing bearing quality wrought AISI 52100 are included in Figure 22 for comparison. Samples of the Ancorsteel 4600V and Ancorsteel 2000 were purposely selected from material produced with a high inclusion content for comparison with forging quality powder.

The bands in Figures 20 through 22 cover the range of results observed for a material of a given category. The data has been plotted on Weibull probability paper, where the percentage failure rate according to a Weibull distribution is plotted against cycles-to-failure on a logarithmic scale. This data representation results in straight-line plots, which enable the L10 values to be determined (90% of samples would have lives which exceed the L10 cycles-to-failure value). A shift in the L10 point to a higher cycles-to-failure represents an improvement. An increase in the slope of the line represents greater consistency.

The results show that the samples produced from the forging quality Ancorsteel 4600V compare favorably with the bearing quality AISI 52100. The other samples performed in good correlation with their inclusion content - see Table 2.

Alloy	Sample	F4	F6
Ancorsteel 2000	1 (Low)	6.7	2.8
	2 (High)	11.4	2.2
Ancorsteel 4600V	3 (Low)	0.8	0.0
	4 (High)	5.0	2.5
	5 (High)	6.8	2.1
	6 (High)	~15 – 20	~6.0

Table 2: Inclusion data for the low alloy powders used to prepare the powder forged samples for rolling contact fatigue testing.

Summary

Over ten years of experience in producing powders for powder forging applications have been reviewed, and an automated image analysis procedure for inclusion assessment has been described. The procedure incorporates the use of a near neighbor concept for sizing fragmented and discontinuous inclusions. This concept forms the basis of a standard test method for determining the nonmetallic inclusion level of low alloy powder forged steel parts, which is currently being developed through the auspices of ASTM sub-committee B.09.11.

The benefit of the microcleanliness program in reducing the level of inclusions in both Ancorsteel 4600V and Ancorsteel 2000 low alloy powders has been clearly demonstrated. Ancorsteel 1000P/F had the benefit of the knowledge gained from the low alloy program. Statistical process control was applied to this product throughout its development as part of an overall quality assurance system.

References

W.B. James, M.J. McDermott, and R.A. Powell, "Powder Forging", Forming and Forging, Volume 14, 9th edition of Metals Handbook, ASM International, 1988, p. 188.

G. Bockstiegel and C.-A. Blande, "The Influence of Slag Inclusions and Pores on Impact Strength and Fatigue Strength of Powder Forged Iron and Steel", Powder Metallurgy International, Volume 8 (No. 4), 1976, p. 155.

R. Koos and G. Bockstiegel, "The Influence of Heat Treatment, Inclusions, and Porosity on the Machinability of Powder Forged Steel", Progress in Powder Metallurgy, Volume 37, 1981, p. 145.

B.L. Ferguson, H.A. Kuhn, and A. Lawley, "Fatigue of Iron Base P/M Forgings", Modern Developments in Powder Metallurgy, Volume 9, 1977, p. 51.

G.T. Brown and J.A. Steed, "The Fatigue Performance of Some Connecting Rods Made by Powder Forging", Powder Metallurgy, Volume 16 (No. 32), 1973, p. 405.

W.B. James, "Quality Assurance Procedures for Powder Forged Materials", SAE Technical Paper 830364, Society of Automotive Engineers, 1983.

P. Ulf Gummeson and D.J. Kasputis, "SPC in Steel Powder Manufacturing", The International Journal of Powder Metallurgy, Volume 23 (No. 4), 1987, p. 261.

G.F. Vander Voort, "Inclusion Measurement", Metallography as a Quality Control Tool, Editors J.L. McCall and P.M. French, Plenum Press, 1980, p. 1.

C.-A. Blande, H. Soderhjelm, R.J. Causton, and T.F. Murphy, "Automatic Counting of Nonmetallic Inclusions in Ferrous Powders for Powder Forging", Powder Metallurgy, Volume 29 (No. 2), 1986, p. 125.

R.J. Causton, T.F. Murphy, C.-A. Blande, and H. Soderhjelm, "Nonmetallic Inclusion Assessment of Powder Forged Steels Using an Automatic Image Analysis System", Horizons of Powder Metallurgy, Part II, Verlag Schmidt, 1986, p. 727.

W.B. James, "Automated Counting of Inclusions in Powder Forged Steels", Modern Developments in Powder Metallurgy, Volume 14, 1981, p. 541.

F.J. Semel, D.J. Kasputis, and P. Ulf Gummeson, "Statistical Process Control in Iron Powder Production and New Product Development", SAE Technical Paper 880144, Society of Automotive Engineers, 1988.

D. Glover, "A Ball/Rod Rolling Contact Fatigue Tester", Rolling Contact Fatigue Testing of Bearing Steels, American Society for Testing Materials, STP771, Editor J. Hoo, 1982, p. 107.
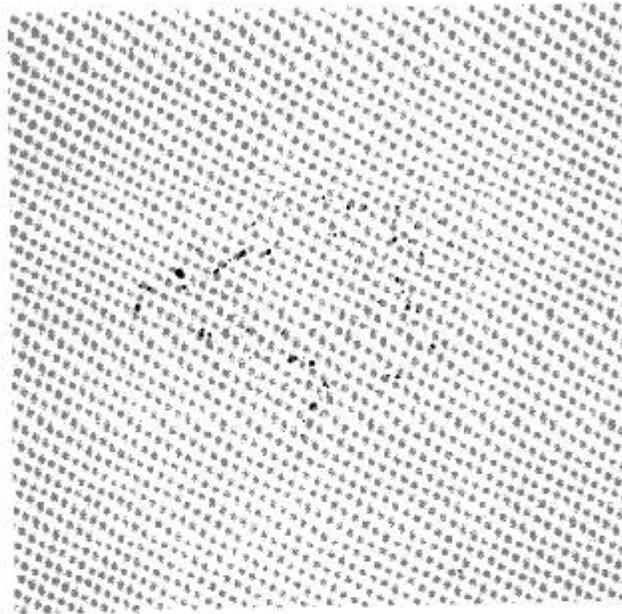
Figure 1: "Spotty oxide" inclusion. Magnification 500X.
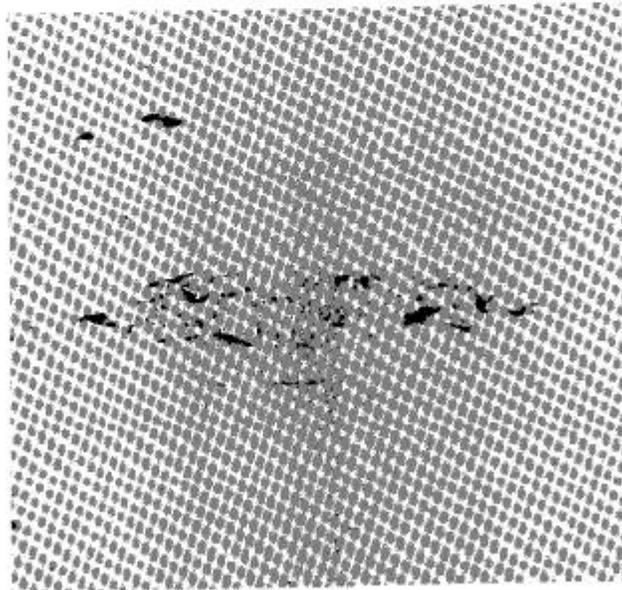
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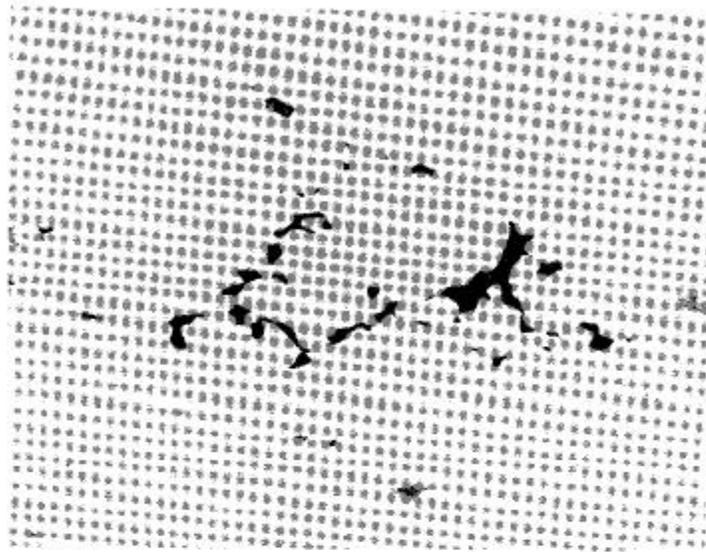
Figure 3: Discontinuous slag inclusion. Magnification 500X.

Figure 4: Discontinuous sulphide inclusion. Magnification 500X.
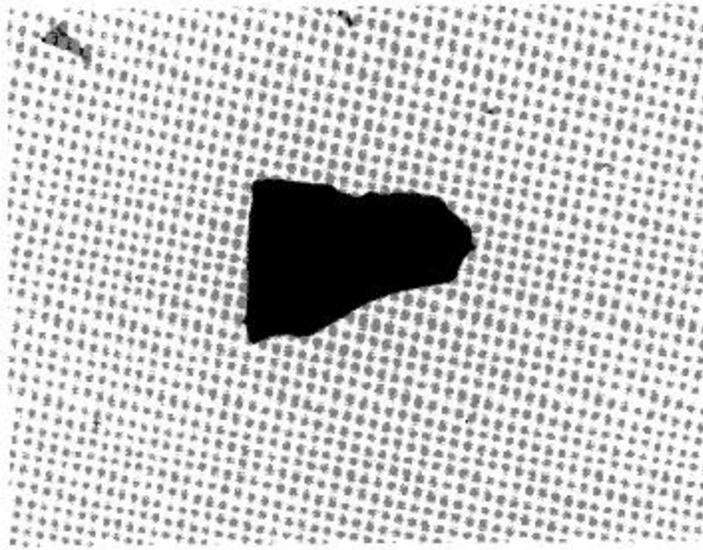
Figure 5: Solid exogenous inclusion. Magnification 500X.
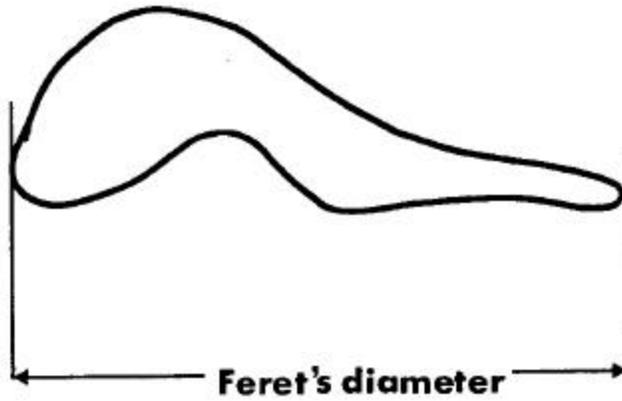
Feret's diameter

Figure 6: Schematic illustration of Feret's diameter.

>30μm

maximum Feret's diameter
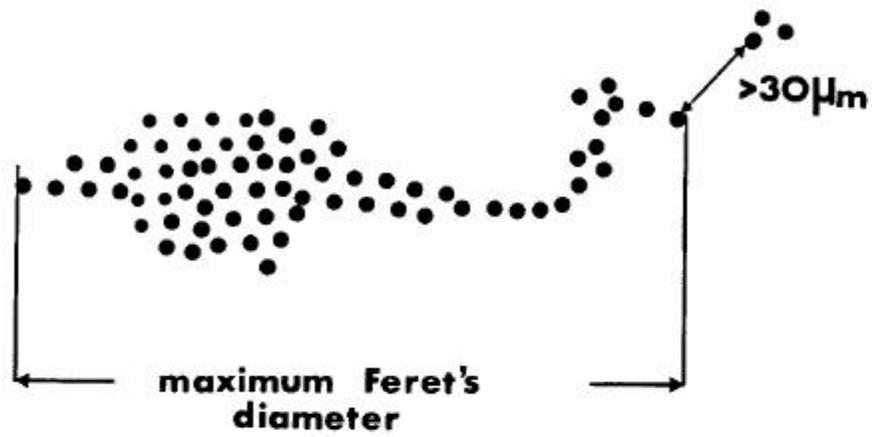
Figure 7: Schematic illustration of the "near neighbor" concept and maximum Feret's diameter.

Television Camera (Plumbicon)

Automated stage

Viewing and analyzing CRT

Light pen

Keyboard   Keypad
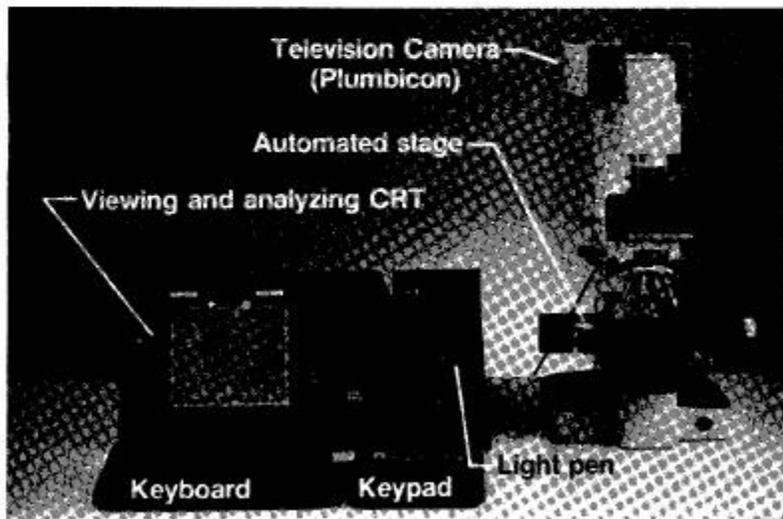
Figure 8: Leitz TAS Plus image analyzer.

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Steps of Image Transformation as Applied to:

Figure 9
Spotty Oxide

Figure 10
Discontinuous
Sulphide
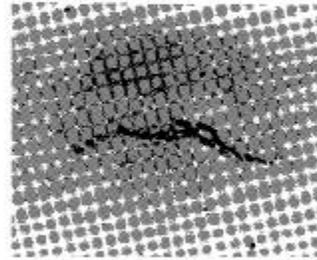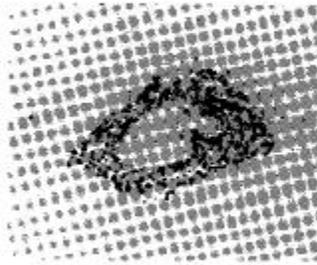
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Image as Detected
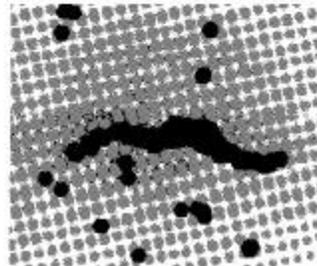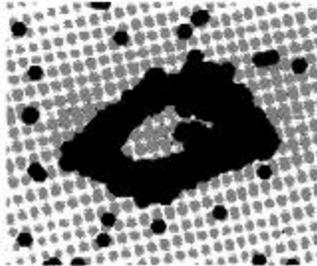
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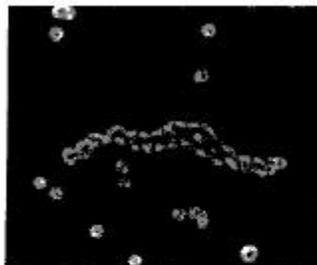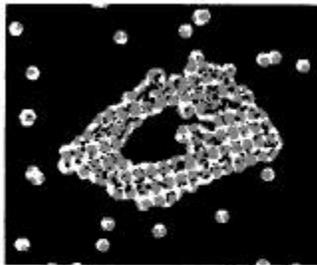
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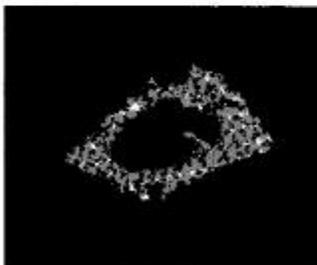
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Negation Dilated
Equivalent Number
of Times to
Figures 9b & 10b

(Figures 9 & 10 continued on next page)
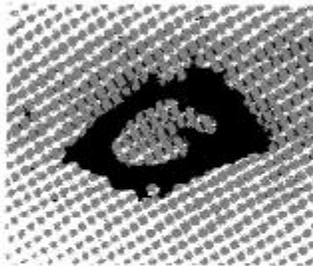
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Figures 9d & 10d
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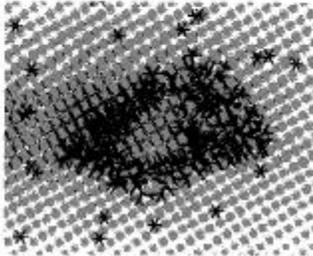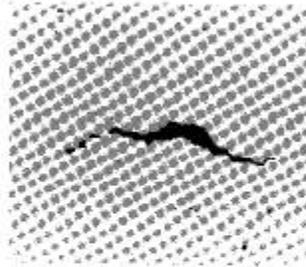
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Line Skeleton of
Figures 9b & 10b
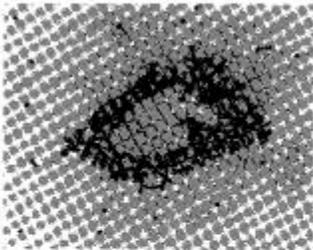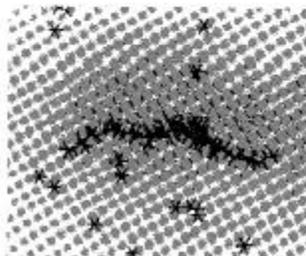
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Figures 9f & 10f
with End Points
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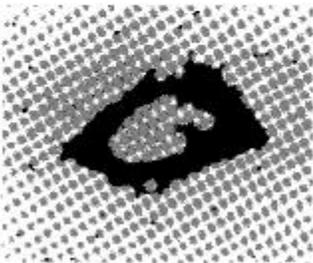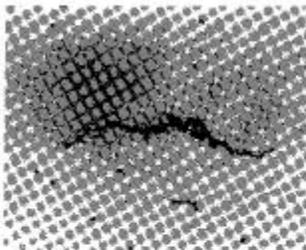
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Combination of
Figures 9e & 9g
and
Figures 10e & 10g
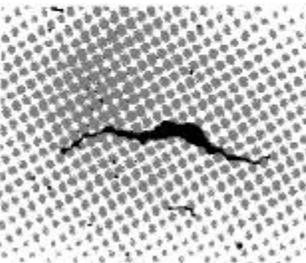
Note: During individual particle analysis all holes are filled.

Figure 11: Inclusion identification via energy dispersive X-ray analysis.
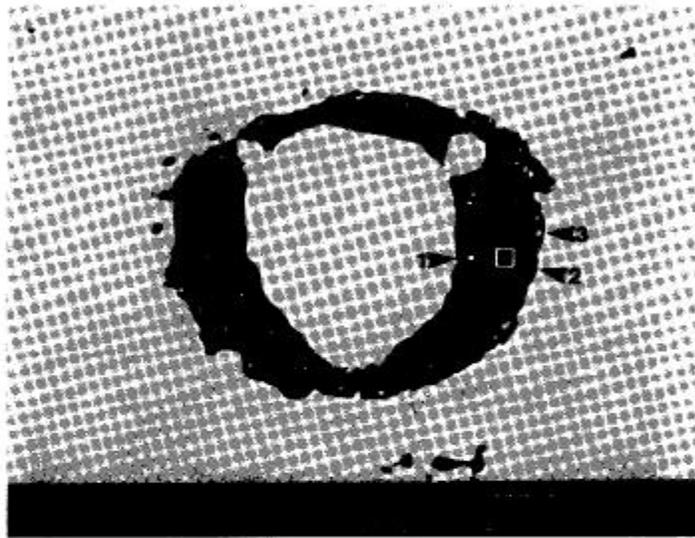
11 a) Secondary electron image of inclusion indicating three locations subjected to energy dispersive X-ray analysis.
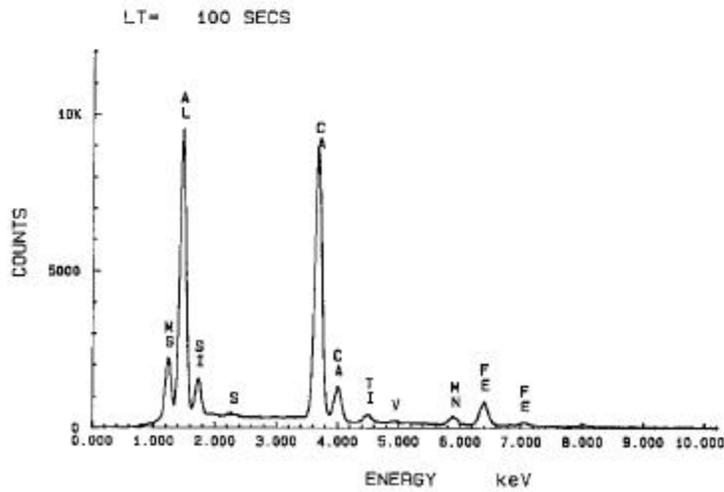
11 b) Energy dispersive X-ray spectra for region 1 of the inclusion in 11 a).

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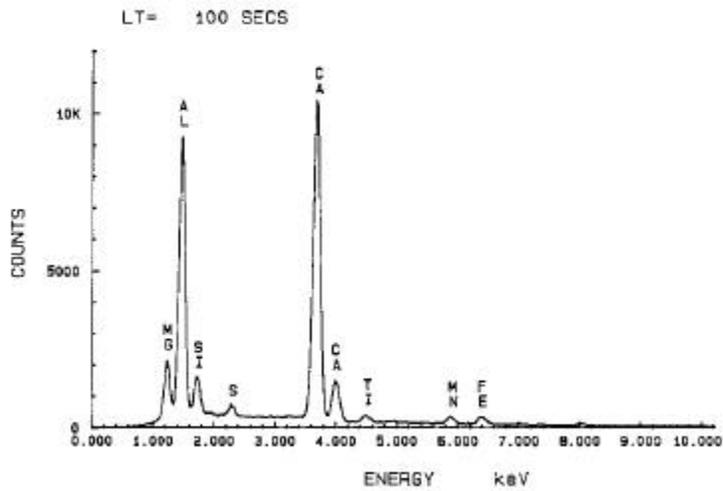
11 c) Energy dispersive X-ray spectra for region 2 of the inclusion in 11 a).

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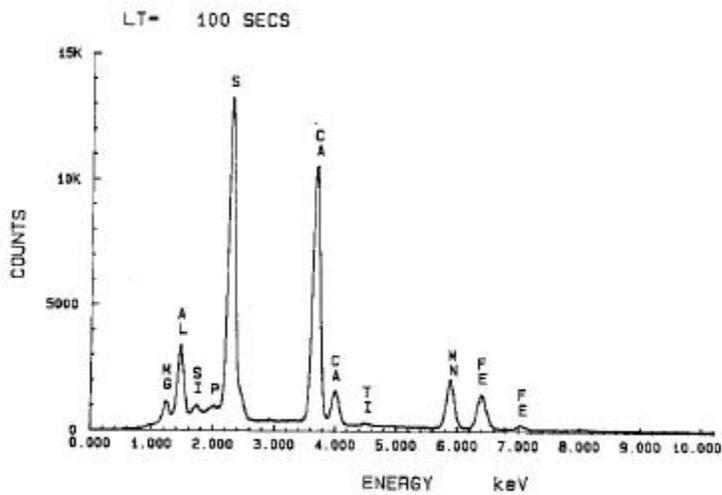
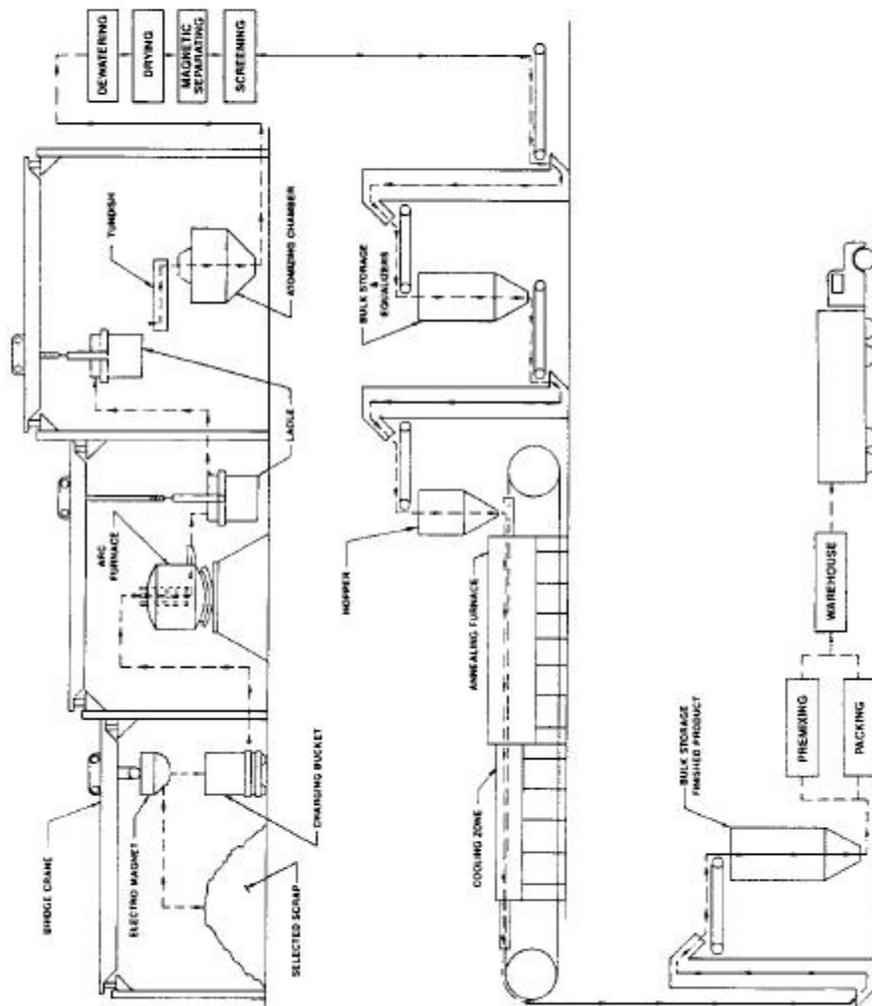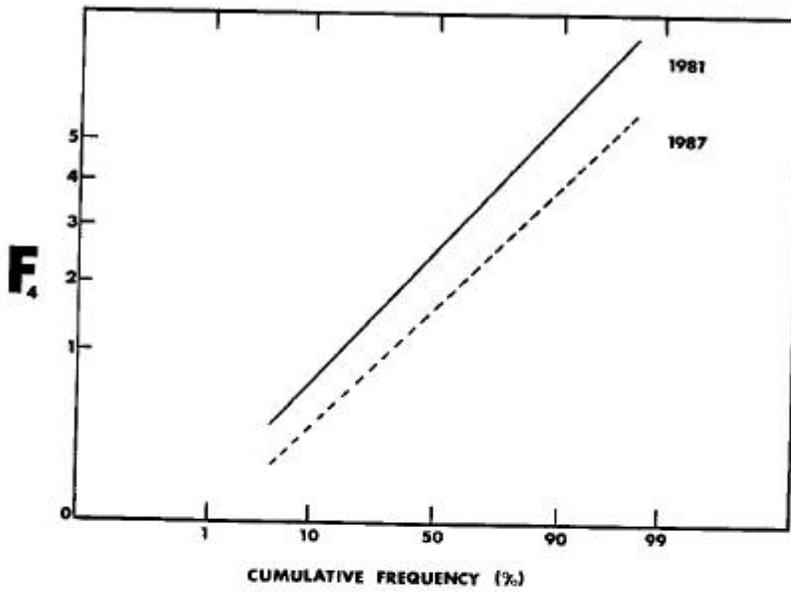11 d) Energy dispersive X-ray spectra for region 3 of the inclusion in 11 a).

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Figure 12: Steel atomizing process.

DEWATERING
DRYING
MAGNETIC SEPARATING
SCREENING

TUNDISH
ATOMIZING CHAMBER
LADLE
ARC FURNACE
HOPPER
BULK STORAGE EQUALIZERS

BRIDGE CRANE
ELECTRO MAGNET
SELECTED SCRAP
CHARGING BUCKET

COOLING ZONE
ANNEALING FURNACE

BULK STORAGE FINISHED PRODUCT
PREMIXING
PACKING
WAREHOUSE

15

Figure 13: Ancorsteel 4600V - Comparison of F_4 inclusion levels for 1981 and 1987.
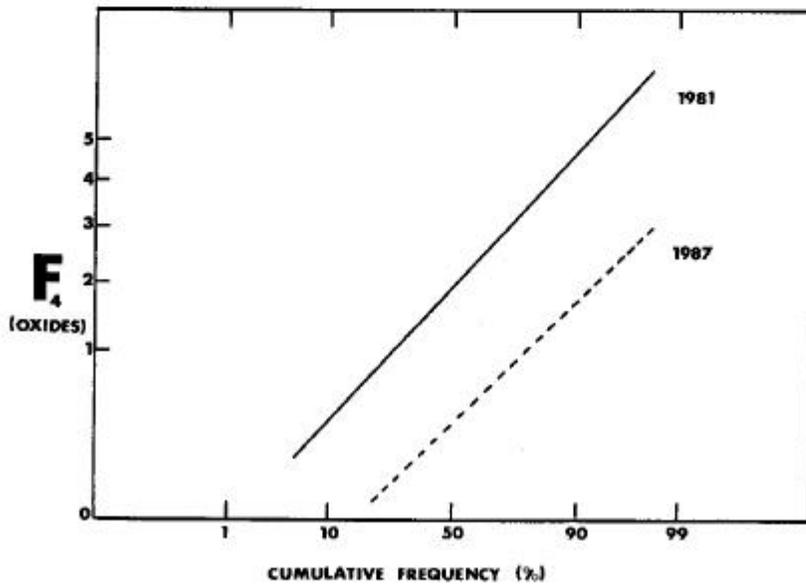
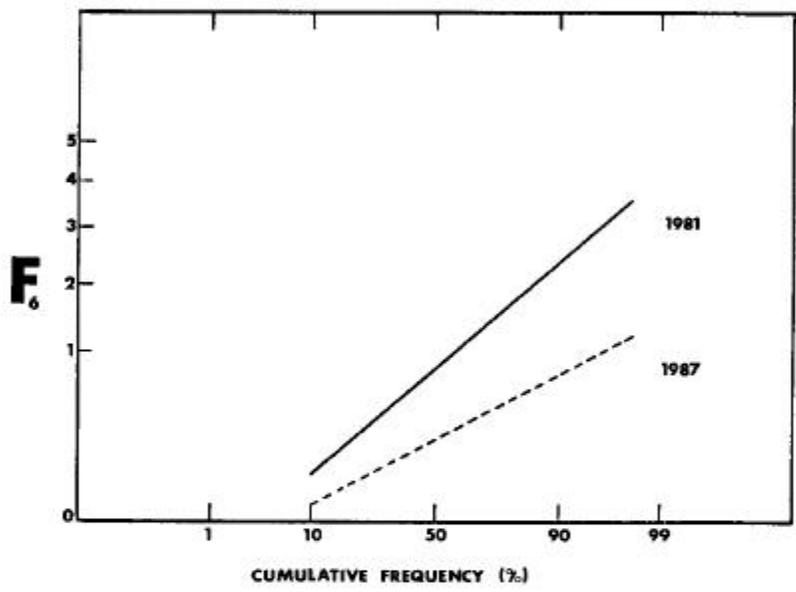Figure 14: Ancorsteel 4600V - Comparison of F_4 oxide inclusion levels for 1981 and 1987.

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Figure 15: Ancorsteel 4600V - Comparison of F_6 inclusion levels for 1981 and 1987.
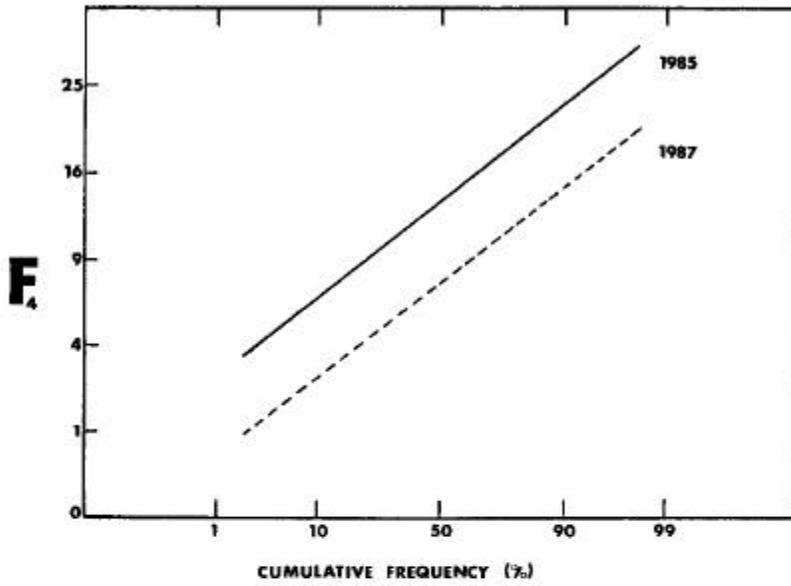
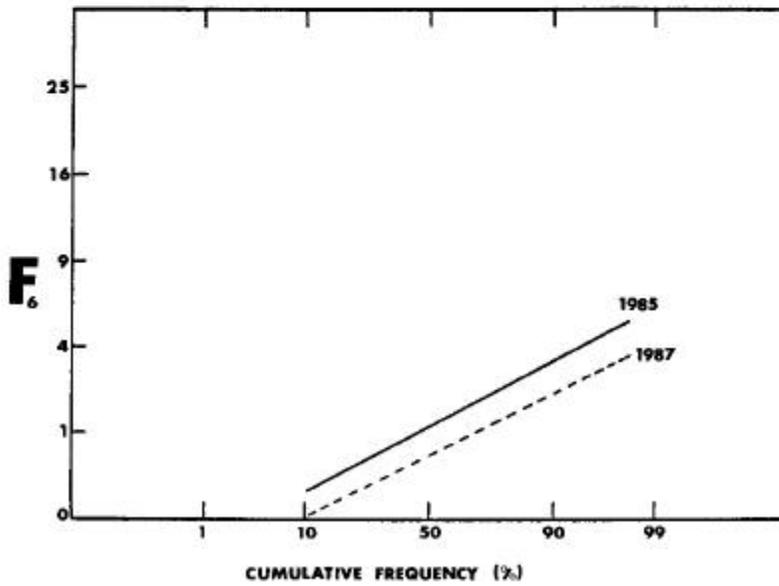Figure 16: Ancorsteel 2000 - Comparison of F_4 inclusion levels for 1985 and 1987.

Figure 17: Ancorsteel 2000 - Comparison of F_6 inclusion levels for 1985 and 1987.
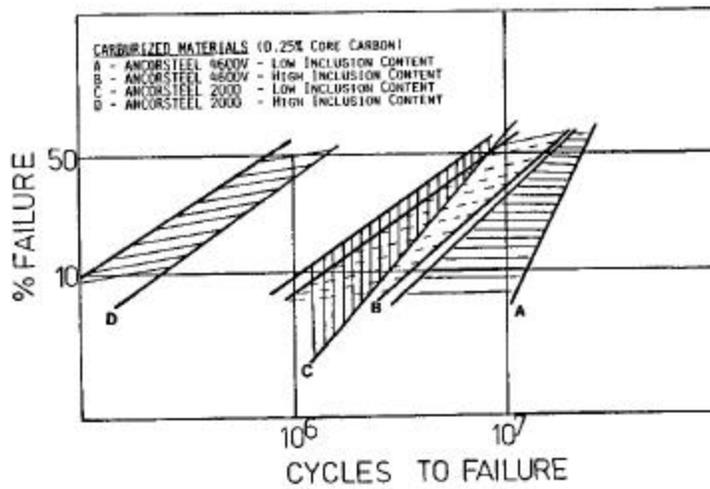
Figure 21: Weibull plots of rolling contact fatigue data for carburized low alloy samples.
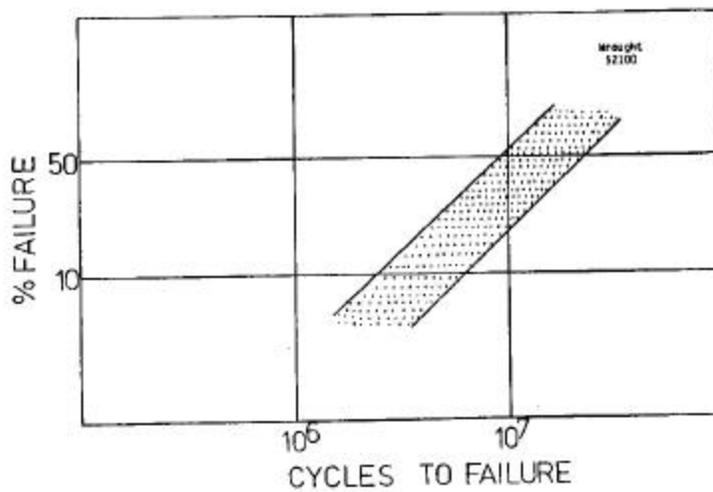

Figure 22: Weibull plots of rolling contact fatigue data for bearing quality AISI 52100.