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TECHNICAL REPORT ARLCB-TR-83034

SAMPLE PREPARATION AND EVALUATION OF STEEL SPECIMENS FOR INCLUSION RETENTION AND SUBSEQUENT AUTOMATED ASSESSMENT

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OCTOBER 1983

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1. REPORT NUMBER ARLCB-TR-83034	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE *(and Subtitle)* SAMPLE PREPARATION AND EVALUATION OF STEEL SPECIMENS FOR INCLUSION RETENTION AND SUBSEQUENT AUTOMATED ASSESSMENT		5. TYPE OF REPORT & PERIOD COVERED Final 6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR*(s)* Theresa V. Brassard		8. CONTRACT OR GRANT NUMBER*(s)*
9. PERFORMING ORGANIZATION NAME AND ADDRESS US Army Armament Research & Development Center Benet Weapons Laboratory, DRSMC-LCB-TL Watervliet, NY 12189		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
11. CONTROLLING OFFICE NAME AND ADDRESS US Army Armament Research & Development Center Large Caliber Weapon Systems Laboratory Dover, NJ 07801		12. REPORT DATE October 1983 13. NUMBER OF PAGES 9
14. MONITORING AGENCY NAME & ADDRESS*(if different from Controlling Office)*		15. SECURITY CLASS. *(of this report)* UNCLASSIFIED 15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

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18. SUPPLEMENTARY NOTES

This report has been approved and accepted by ASTM as a standard practice for preparing and evaluating specimens for automatic inclusion assessment of steel. It is published in the Annual Book of ASTM Standards, Part 11.

19. KEY WORDS *(Continue on reverse side if necessary and identify by block number)*

Standard Practice
Metallographic Preparation
Steel
Inclusions
Interference Microscopy

20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

A recommended practice for preparation and evaluation of steel specimens for automatic inclusion assessment was developed. The polishing procedure involved the use of diamond abrasives on rotating paper laps. This technique preserved the true morphology of the non-metallic inclusions. Evaluation of a properly prepared sample was accomplished using the sensitive tint condition of Differential Interference Contrast Microscopy.

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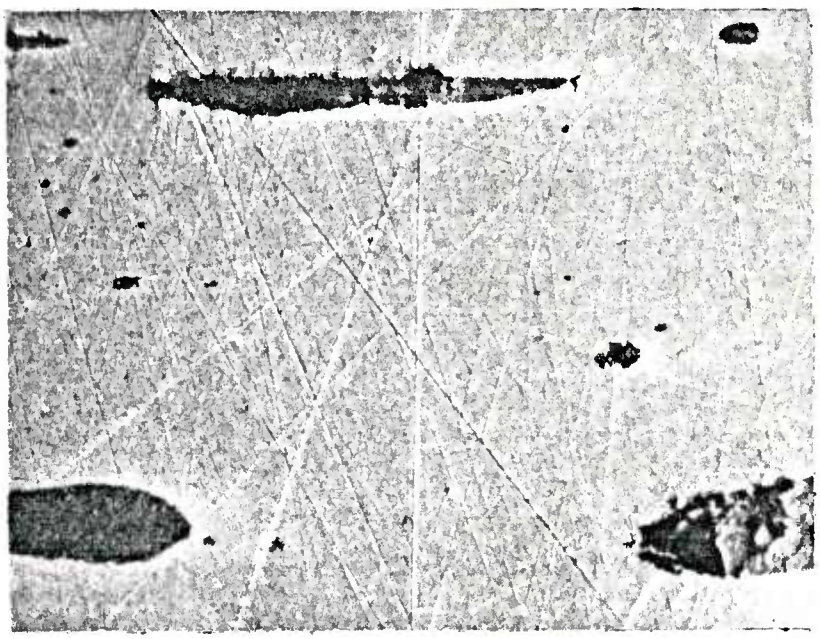
INTRODUCTION

Inclusion amount, size, morphology, identification, and distribution are very important factors in the quality control of steel fabrication. In most large steel manufacturing industries, inclusion rating is performed quantitatively on automated image analyzing computer systems rather than by the former conventional hand count using an eyepiece reticle on an optical microscope. However, in order to accurately and reliably evaluate the amount, size, and distribution of these potentially detrimental inclusions and subsequently establish a standard for acceptance or rejection of a steel component, a very high standard of reproducible specimen preparation is required. The specimen must be flat and free from scratches that are detectable by the automatic image analyzing equipment. Parallel surfaces are necessary when an "upright" form of microscope is used on the measuring systems, since usual devices to level uneven specimens may damage the finished surface and normally are inadequate to preserve focus over the entire specimen area. The specimens should be prepared in such a way that the inclusions are not pitted, left in relief, dragged, removed, or obscured (see Figures 1 and 2).

In the early era of quantitative image analysis projects, reasonable inclusion counts on metallographically prepared specimens (when compared to hand counts using an eyepiece grid on a microscope) were difficult to acquire according to ASTM task group committee members. The author suggested that the inclusions be altered in size and shape by being "pulled out" or "chipped" during the various polishing steps of metallographic preparation. As a result, the image analyzer "saw" these inclusion particles longer and

Mag. 100X Differential Interference Contrast

Figure 1. An improperly prepared sample showing the effects of a severe relief-polish resulting in undesirable "noise" level in the automatic evaluation system.



Mag. 500X Differential Interference Contrast

Figure 2. The effect of an improper polishing procedure on the inclusion retention. Scratches are still evident. Inclusions are pitted, partially removed leaving large pits and ditches which obscure the true inclusion morphology on the image analyzer.

larger than their actual size. An operation using conventional manual methods such as the grid and a microscope could distinguish these artifacts. The instrument could not distinguish them, and interpreted these "pull-outs" or holes as it "saw" them. Considering the above suggestion as a possible cause for this discrepancy, a partial series of tests was run by ASTM Committee E4.14 on an image analyzing system using an area threshold forced as high as possible to read the suspected "pull-out" areas as if the inclusions were still intact. The reported average area was raised from 25 percent to 33-1/3 percent, and thus verified the assumption that the inclusions had been broken and extracted during polishing, leaving ditches which grossly exaggerated the true size of the inclusions. Obviously, this error is not tolerable when establishing a standard which will be utilized as a criterion for acceptance or rejection of a fabricated component.

In order to alleviate this problem, a program was undertaken to develop a new sample preparation technique which would not only retain all of the inclusions, but also maintain a flat, scratch-free surface. This report describes the results of this program.

METHODS

Two metallographic sample preparation procedures were developed at Benet Weapons Laboratory (BWL) and submitted to ASTM Committee E4 on Metallography for acceptance. The two methods described are designated as Method A and Method B. The mounting method was identical for both procedures.

Mounting

Longitudinal samples of resulfurized steel were cut (1 in. x 0.5 in.) using a conventional metallographic cut-off wheel and set in one-inch round flexible silicone rubber molds as embedding containers. In order to obtain a "hard-mount" which is vital for sample flatness, a two-part casting resin containing a hardener was used to encapsulate the entire samples. Approximately two to three hours after casting, while the epoxy was still in the "plastic" stage, hot-pressing with transoptic powder was employed using a mounting press forming 1.5 inch mounts.

Method A

Grinding (Manually)

Five grinding steps using silicon carbide abrasive papers (120, 240, 320, 400, and 600 grits, respectively) on fixed rotating discs were utilized. The paper laps were operated under a reasonable rapid stream of water, in order that loosened abrasive and grinding debris were immediately washed away, and that the specimen was kept cool during the grinding operation. The specimen was cleaned thoroughly and dried before proceeding to the polishing steps. (Automated grinding systems were tried also and yielded the same results.)

Polishing (Manually)

Proper finishing for quantitative measurement requires that there be an absolute minimum of the polishing action. Diamond abrasives were used during the entire polishing procedure in order to insure maximum cutting of particles and to maintain surface flatness. The samples were cleaned thoroughly between each step. The use of a lubricant was required during the polishing steps.

The following polishing steps were used: 15μ-9μ-3μ diamond paste abrasives for two minutes in succession, then .5μ diamond paste abrasive for three to four minutes. For this purpose, high quality tablet paper was utilized on a fast speed rotating disc. A few drops of oil served as a lubricant. The sample was rotated the entire polishing time using medium to light pressure in a clockwise direction. The specimen was then washed with alcohol and dried.

Method B

Grinding (Manually)

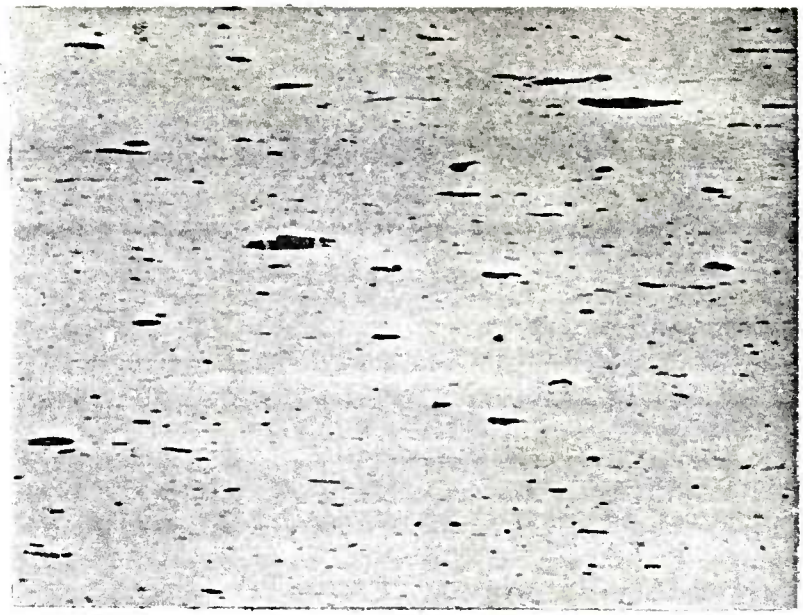
Ultralap papers of 12μ-9μ and 3μ Al_2O_3 were sequentially glued to a piece of glass as backing material. The sample was conventionally hand ground using water as a lubricant.

Polishing (Manually)

Diamond paste abrasives of 3μ and .5μ were applied for three to four minutes each using high quality tablet paper on a fast speed rotating disc. A few drops of oil served as a lubricant. The sample was rotated the entire polishing time using medium to light pressure in a clockwise direction. The sample was then washed with alcohol and dried.

Final polishing either automated or manually using an abrasive slurry on napped cloths was not used in either method as it produced a relief polished surface which was unacceptable for quantitative image analysis equipment.

Methods A and B produced specimen surfaces which met all the previously mentioned requirements for a properly prepared specimen. The following evaluation method is also included in the new standard, and the photomicrographs in Figures 3 and 4 illustrate the effectiveness of this polishing technique.



Mag. 100X Differential Interference Contrast

Figure 3. Properly prepared specimen using BWL technique. All inclusions are intact and no matrix relief is apparent.

Mag. 500X Differential Interference Contrast

Figure 4. Inclusions are flat with the matrix, and not dragged or pitted, thus insuring true evaluation by the automated image analyzing system.

RESULTS

Evaluation Methods

Differential Interference Contrast (DIC)

A microscope equipped with Differential Interference Contrast (DIC) illumination at magnifications of about 100X and 500X should be used in the preparation procedure to verify the true surface topography of properly prepared specimens. These instruments provide adjustments to obtain three classes of image. The "dark field" DIC image is black for a perfect mirror surface, but bright for any departure from flatness. This image is useful in detecting residual scratches and other surface defects, but does not normally permit simultaneous observation of the actual inclusions. The "topographic" DIC image gives an overall impression of the combination of surface roughness and actual microstructure, but must be adjusted with care to insure that the topography is not seen inverted. The "sensitive tint" condition is usually the most useful and should be adjusted so that the flat areas are magenta and the actual inclusions dark. Magnification at 100X will reveal whether residual scratches are present and whether background relief is evident. All scratches detectable at 100X DIC should disappear following the diamond polishing steps. At 500X in the DIC sensitive tint condition, all of the edges of inclusion particles should be sharp and recognizable as fine edges. Narrow bright lines indicating narrow ditches at some inclusion edges may, however, still be seen. A test may be made to determine whether these ditches will be measured by the television system. The polarizer is slowly rotated away from the full DIC position, and the inclusion edges watched for apparent motion. There is a critical trough configuration beyond which the bright

troughs turn black at some point in the transition to bright field illumination and the inclusions appear to become larger. The use of a filar eyepiece may be helpful in determining at this point whether or not the inclusion trough has widened. Narrow troughs can be accepted as harmless when of a sub-critical configuration such that they disappear into the background when the polarizer has opened a small part of its range. Some fine scratches from final polishing will usually be seen under DIC at 500X. These normally also disappear on opening the polarizer, and thus will not affect quantitative measurement at lower magnification. For comparison within a group of specimens of the same type of steel, contrast enhancement treatments such as staining, anodization, or coloring methods may be used. Acid etchants or other reagents which dissolve part of the surface are not allowable. Specimens which are not measured immediately or which may require remeasurement, must be stored in a fully effective desiccator.

CONCLUSIONS

A satisfactory and acceptable method of preparing steel samples containing inclusions for quantitative automatic inclusion assessment has been developed at Benet Weapons Laboratory. The procedure requires the use of diamond abrasives and high quality tablet paper on rotating discs. A method of evaluating a properly prepared sample has also been developed using the sensitive tint condition of Differential Interference Contrast Microscopy.

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