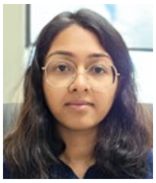
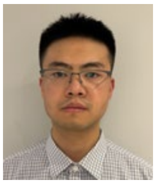
Development of an Improved Inclusion Assessment Approach for Steels





Control of non-metallic inclusion composition, morphology, and distribution is crucial for superior product quality, enhanced mechanical properties, and efficient processing in the era of "clean" steels. This study addresses the limitations of prevailing semi-quantitative inclusion assessment standards, specifically ASTM E45 and ASTM E2142, by benchmarking their results with the quantitative statistical technique, ASTM E2283. A refined approach is proposed that employs relevant statistical data sets for specific grades of oil country tubular goods and line pipe steel tubulars to predict composition-based critical inclusion sizes that may be linked to specific performance properties, such as fracture toughness in hydrogen and sour environments.

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Non-metallic inclusions (NMIs) are non-metallic phases, generally oxides, sulfides or oxy-sulfides, present in all steels regardless of processing route that dictate the "cleanliness" of steels. Presently, coarse (>1 µm) precipitates like Ti and Nb-rich nitrides are also considered inclusions, owing to their detrimental effects on mechanical properties. The type, size, shape, morphology, count per unit area/number density and distribution of NMIs have been extensively linked to the degradation of mechanical property performance and surface quality of steels.[1] The opportunity for better product quality has driven the need for effective control and optimization of NMIs through "inclusion engineering" by tailoring the above-mentioned inclusion characteristics. Monitoring the effectiveness of such control and optimization is crucial and is governed by the inclusion assessment methods employed. Basic inclusion assessment starts with classifying the different types of inclusions, most commonly based on their origin sources, as:

- "Endogenous/indigenous," intrinsic to the steelmaking process and a result of a reaction with elements added during steelmaking such as deoxidizers (Al, Si, Mn), desulfurizers/ inclusion shape modifiers (Ca, Mg), or
- "Exogenous," linked to external sources such as refractory fragments, slag entrapments, etc., or unwanted reoxidation of deoxidized steel from contact with air during steel transfer in the ladle/tundish/ mold.

Alternatively, the NMIs can also be classified by:

- Their chemical compositions as "oxides," "sulfides," etc.
- The stage when they are formed with reference to the start of solidification as "primary," being those formed prior to solidification starting, or "secondary," as those formed following the start of solidification.
- Sizes as "macro," if an inclusion is large enough to cause immediate product failure during processing or use, or "micro," for all other inclusions.

Classification based on inclusion origin sources is most prevalent and forms the basis of most inclusion rating approaches. Inclusion rating refers to a method of qualifying a steel product for technical use through an assessment of inclusion size and distribution based on severity levels defined by established standards such as ASTM E45,[2] ASTM E2142,[3] ISO 4967,[4] JIS G 0555,[5] EN 10247,[6] etc. Indigenous inclusions have been shown to exhibit smaller sizes and can be controlled better through controlled changes in processing like optimizing deoxidization/desulfurization, etc., compared to exogeneous inclusions which are rarer, more difficult to control and can grow to large sizes. Hence, most inclusion rating standards focus on rating indigenous inclusions based on microscopic observation of steel samples.

With the advent of "clean" steels, inclusion sizes and counts have significantly diminished, making it increasingly difficult to track rarer, larger inclusions in the steel through the current standard rating methods that rely on the observation of microscopic samples with field evaluation areas of 100–200 mm^2. Another approach by Murakami[7] adopted into the ASTM E2283[8] standard looks at a statistical method for predicting the largest inclusion within a given area/volume[9] of steel with the potential of initiating a fracture. With Murakami's[7] work on bearing steels and several other carbon steels, they were able to demonstrate a direct correlation between the fatigue strength and the predicted largest inclusion size. Such an approach could be beneficial when trying to draw correlations between the inclusion characteristics observed on metallographic samples and mechanical property performance such as results from a Charpy V-Notch (CVN), drop weight tear test (DWTT), fatigue, J integral/crack tip opening displacement (CTOD), hydrogen-induced cracking (HIC) tests, etc.

The escalating demand for advanced high-strength steels, characterized by superior toughness properties and tailored for applications in challenging environments like sour service and alternative energy, underscores the critical necessity to establish a correlation between inclusion characteristics and mechanical properties. This correlation is pivotal for identifying favorable inclusion characteristics that contribute to achieving desired mechanical properties. Consequently, it drives the adoption of steelmaking and processing strategies geared toward optimal control and modification of inclusions.

The current work aims to establish a novel approach of inclusion assessment in steel that can be easily used to correlate inclusion characteristics (size/shape/frequency/distribution) to mechanical properties (tensile, toughness, fatigue, corrosion, etc.) by combining elements from the existing established standards of ASTM E45, ASTM E2142 and ASTM E2283. Major limitations of these standard approaches are briefly discussed in the following section.

ASTM E45 (Microscopic Method) Limitations

Several gaps in the ASTM E45 standard have been identified. Firstly, there is a lack of information on inclusion type or chemical composition which hinders a comprehensive understanding of their impact on steel properties and limits any knowledge to modify steelmaking practices to produce cleaner steel. Secondly, the standard ignores inclusions with width/thickness <2 μm, regardless of shape. This omission disregards potentially influential small heterogeneities. Thirdly, the area of examination, particularly for clean steels, is limited. The minimum required area of 160 mm^2 constitutes a very small region of the critically stressed section of a specimen extracted from the steel pipe and has a low probability of containing larger, rarer inclusions that lead to test failures. Finally, there is a clear absence of a quantifiable metric for correlation with mechanical properties, which hinders adoption of application-based strategies for inclusion control and/or modification.

While the existing standard methods may suffice for steel products operating under normal service conditions, the heightened demands imposed by environmentally severe conditions such as arctic environments, sour service and gaseous hydrogen transmission necessitate a more discerning approach. In these conditions, the steels become more sensitive to the presence of small heterogeneities that might be deemed acceptable in less demanding circumstances. Therefore, an advanced inclusion analysis approach is imperative, as the current semi-quantitative methods based on ASTM E45 are inadequate for achieving the required level of precision and reliability.

ASTM E2283 Limitations

The ASTM E2283 standard plays a crucial role in statistically predicting the maximum inclusion size within a specified area or volume of steel, typically scaled up to 1,000 times the input area/volume.[9] This predictive methodology has demonstrated remarkable accuracy in establishing correlations with mechanical properties, including fatigue strength and fracture toughness.[7,10] The statistical analysis in ASTM E2283 leverages extreme value (EV) statistics, a tool commonly used to scrutinize extreme events or rare occurrences. In the context of steel production, the focus is on identifying EVs associated with inclusion characteristics such as morphology and distribution.

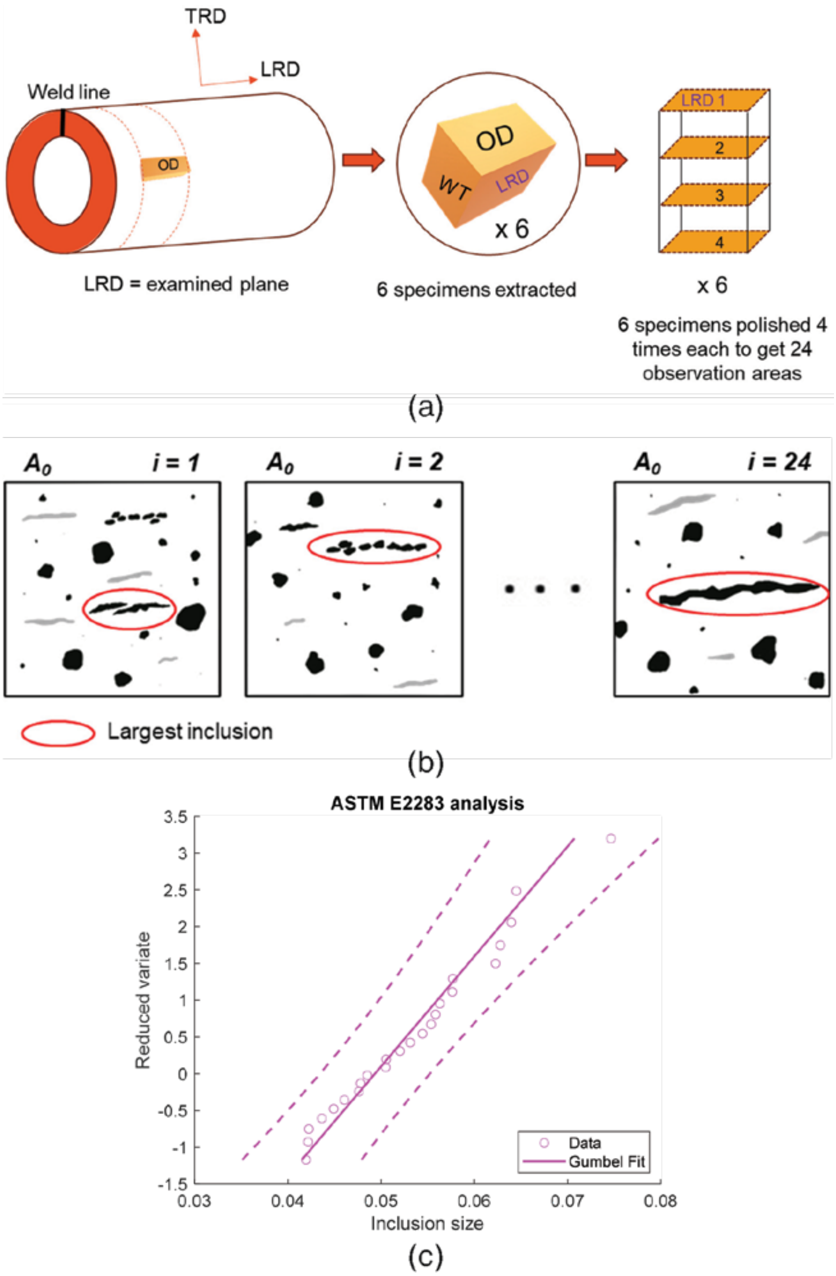
The standard adopts the EV or Gumbel distribution, a proven method in extreme value analysis especially suited for predicting maximum values within a data set. The Gumbel distribution models the tail of the distribution, where extreme values reside, and is defined by two parameters—location and scale, determining position and spread, respectively, akin to mean and standard deviation.

In ASTM E2283, the input data set comprises 24 values, each representing the maximum inclusion size from observed areas of at least 150 mm^2 on six polished metallographic specimens across four planes through thickness (see Figs. 1a and 1b). The observation is usually done through optical microscopy, which facilitates the use of raw data from the ASTM E45 analysis for extracting the 24 values. This data set is then fitted using Gumbel distribution following the method described in ASTM E2283 (Fig. 1c) and the largest inclusion size predicted to occur in an area/volume 1,000 times the input area/volume is calculated.

While ASTM E2283 expands the examination area compared to ASTM E45, examining approximately 24 times the area, it does not fully address all the gaps outlined for ASTM E45.

Figure 1

Example of specimen extraction for E2283 analysis: six specimens extracted and each polished four times to get 24 observation areas (LRD = Longitudinal to rolling direction, TRD = Transverse to rolling direction, OD = pipe outer diameter, WT = pipe wall thickness) (a); Methodology for input data set collected based on maximum inclusion size from each of the 24 examined areas (b); Gumbel distribution fit for the data (with 95% confidence intervals-dashed lines) (c).



Modified ASTM E2283 Analysis

Key modifications to address the gaps in ASTM E45 and ASTM E2283 standard-based inclusion analysis will include two main components: creation of input data sets incorporating composition-based relevant inclusion characteristics, and identification of suitable statistical distributions to characterize each of these data sets.

The first component will be achieved through the use of the ASTM E2142 standard to classify inclusions based on their true chemical compositions and shape (aspect ratios, or ARs) using scanning electron microscope (SEM) equipped with energy-dispersive spectroscopy (EDS); for example, the data set for deformable sulfide inclusions will include size/total interparticle spacing (TIS),[11] etc. The overall idea is to develop custom classification rules as per ASTM E2142 Method 3 for steels with specific compositions and/or targeted applications. Critical inclusion characteristics and their respective measurement methods will be defined in terms of size, shape, count and orientation with respect to the rolling direction (RD). For example, inclusion size usually represented by "length" can be defined in different ways; ASTM E45/E2142 has no strict definition but alludes to maximum dimension in RD, while ASTM E2283 mentions maximum feret diameter (feret diameter is the distance between two parallel tangents on opposite sides of a randomly oriented particle, usually an average or maximum value over several orientations is used). Different interpretations of length and width could lead to different results and affect E45/E2142

Table 1

Comparison of Existing Standard Inclusion Assessment Methods and Proposed Modified E2283-Based Statistical Method

Element	E45 (microscopic method)	E2142 (composition based)	E2283-current (statistical)	E2283-modified (statistical based on composition)
Rating	Number of fields of each inclusion type and thickness category are reported for each severity from 0 to 5 in whole or half-severity level increments	Same as E45 for Methods 1 and 2 Method 3 supports custom rating	Predicts largest inclusion size based on extreme value (EV) statistics (Gumbel distribution)	Predicts largest inclusion characteristics (shape/size/count) based on EV statistics (Gumbel/generalized extreme value (GEV), generalized Pareto distribution (GPD), exponential-GPD (EXP-GPD))
Specimen preparation/sampling	Minimum 160 mm^2 polished surface (normal to the rolling plane, parallel to rolling direction for pipes) Recommended at least six specimens per heat but not a requirement	Same as E45	Required six specimens of minimum 150 mm^2 area each, polished four times to get 24 areas of observation	Same as E2283
Parameters measured	Inclusion size (length), width or diameter, count (only for globular oxide inclusions)	Same as E45	Inclusion size (length)	Inclusion size (length), shape (aspect ratio), count per unit area, total interparticle spacing (for all inclusion types mixed together or individual inclusion types)
Inclusion classification	Main categories based on morphology, imaging contrast/gray level: Type A (Sulfide) Type B (Alumina) Type C (Silicate) Type D (Globular Oxide) Subcategories based on width/diameter: Thin, Heavy	Methods 1 and 2: Same as E45 but based on actual chemical composition, with slightly different rules based on AR and added Globular Sulfides as a separate category Method 3 supports custom rules based on chemical composition	No classification; results are displayed based on the data set Data sets can include all inclusion types or individual types	Same as E2283
Applicability	As per E45, para.6.7.: "In determining the inclusion content, it is important to realize that, whatever method is used, the result actually applies only to the areas of the specimens that were examined." Large sampling required for statistical confidence	Same as E45	Predictions apply to an area/volume 1,000 times the input area/volume	Same as E2283

classification that rely heavily on ARs (length/width ratios).

The second component will explore the use of other EV distributions[12] such as generalized extreme value (GEV), generalized Pareto distribution (GPD), exponential-GPD (EXP-GPD) distributions by comparing the best fit to the input data.

A comparison between the modified approach and the existing standard-based approaches is summarized in Table 2. This modified approach is an ongoing development that aims to provide a more comprehensive understanding of the relationship between inclusion characteristics and mechanical properties, refining the predictive capabilities of ASTM E2283. The overall benefit of the proposed inclusion analysis method lies in the ability to capture inclusion evolution at various stages of the manufacturing process; for example in pipe manufacturing, inclusion characteristics can be tracked by sampling at various processing stages starting from steelmaking (ladle, degasser, caster) to rolling (skelp), through pipe forming (final product). This strategic approach acknowledges the dynamic nature of inclusions, recognizing that their characteristics may vary at different stages of production, thereby creating opportunities for effectively analyzing the impact of various process modifications.

The following elements are considered within the scope of this article:

- ASTM E2142 Method 1, which is an SEM analog of the ASTM E45 classification method, will be used to classify inclusion types based on chemical composition and ARs.
- Conventional Gumbel distribution will be considered for the E2283 analysis.

Fig. 2 shows a flowchart of the inclusion characterization methodology that defines the parameters used for the conventional vs. modified analysis approaches. Overall inclusion characterization is based on shape, size, count (per unit area)/number density and orientation to the rolling direction (RD). For the sake of brevity, the results in this article will focus only on the size parameter for the ASTM E2283 analysis. Different parameters to represent size will be used to study their impact on the predicted inclusion sizes. The basis for the choice of these parameters will be discussed in the following section.

To summarize, the current article will focus on comparison of results from:

- Conventional ASTM E2283 analysis to predict the largest inclusion size in a given steel area based on the ASTM E45 (contrast/morphology based)-produced input data set for mixed and individual inclusion types (hereafter referred to as Case 1).
- Modified ASTM E2283 analysis to predict the largest inclusion size in a given steel area based on the ASTM E2142 Method 1 (composition/morphology based)-produced input data set for mixed and individual inclusion types (hereafter referred to as Case 2).

Materials and Methodology

Material

A low-alloy proprietary 110 ksi oil country tubular goods (OCTG) steel with the chemical composition shown in Table 2 was used for the analysis. The steel was manufactured in an electric arc furnace and underwent thermomechanical-controlled processing followed by pipe forming through electric resistance welding. After forming, the pipe was subjected to austenitization, quenching and tempering treatment to achieve its final condition. This steel is designed for use in environments exposed to aqueous hydrogen sulfide (sour service conditions). Stringent inclusion control is therefore extremely important for enhanced performance of such steels.

Methodology

The specimens were extracted from seven different circumferential positions of a finished pipe as shown in Fig. 1a. These positions were located away from the seam weld, therefore inclusions associated to or affected by the welding process were not analyzed. The extracted specimens were cut in half such that one half was used for ASTM E45 analysis using optical microscopy (OM) to extract data for ASTM E2283 analysis and the other half (same plane) was used for both ASTM E45 analysis and ASTM E2142 Method 1 analysis using SEM-EDS.

The analysis was done on the LRD face as shown in Fig. 1a. For ASTM E45 analysis, automated inclusion analysis using the Clemex Inclusion Rating (CIR) Version 9.7 OM-based software as per ASTM E1245[13] was performed for an observation area of ~163 mm^2 per specimen, while for ASTM E2142 Method 1, automated inclusion analysis software, ASPEX automated feature analysis (AFA) Version 1 was utilized for observation areas between ~150–160 mm^2 for all specimens except one that had an observation area of ~136 mm^2.

For ASTM E2283 analysis, each of the seven specimens were analyzed using automated ASTM E1245-based optical measurements across three additional planes by polishing down in the through-thickness direction as illustrated in Fig. 1a. As a result, 28 areas of observation were obtained (~163 mm^2 each) from which sizes for 28 largest inclusions were extracted.

Table 2

Chemistry of Studied Steel, wt. %

C	Mn	Ti	B+N+S+P	Ca+Si+Cu+Ni
0.25	0.43	0.02	0.02	0.71

The largest 24 inclusions, irrespective of type (all types mixed), defined by ASTM E45 were selected from the 28 for the input data set for a conventional E2283 analysis. Similarly, a modified inclusion analysis approach included 24 largest inclusion sizes for mixed types as well as individual types defined by composition-based classification of ASTM E2142 Method 1 as input data sets for the analysis. For comparison with E2142 defined inclusion types, data sets of the 24 largest inclusion sizes for individual inclusion types were also obtained from ASTM E45 data.

The largest predicted inclusion size was then calculated according to ASTM E2283 for each of the above-mentioned input data sets.

Apart from the effect of true composition-based classification on inclusion analysis results, the response of different parameters for representing inclusion size and shape in terms of length and AR on the classification results was studied. As shown in Fig. 2, inclusion size (or length) and AR can be represented by following parameters as per the standards and/or automated OM-based inclusion analysis software:

- F_{maxRD}: Maximum feret diameter in RD as per ASTM E45/E2142/E1245 (feret diameter definition explained in "Modified ASTM E2283 analysis" section).
- F_{max}: Maximum feret diameter (longest of 8, 16, 32 or 64 feret diameters — irrespective of orientation to RD) as per CIR OM-based inclusion analysis software.
- AR1: Ratio of maximum (F_{max}) to minimum (F_{min}) feret diameter as per CIR software (F_{min} = minimum Feret diameter or the shortest of 8, 16, 32 or 64 feret diameters — irrespective of orientation to RD).

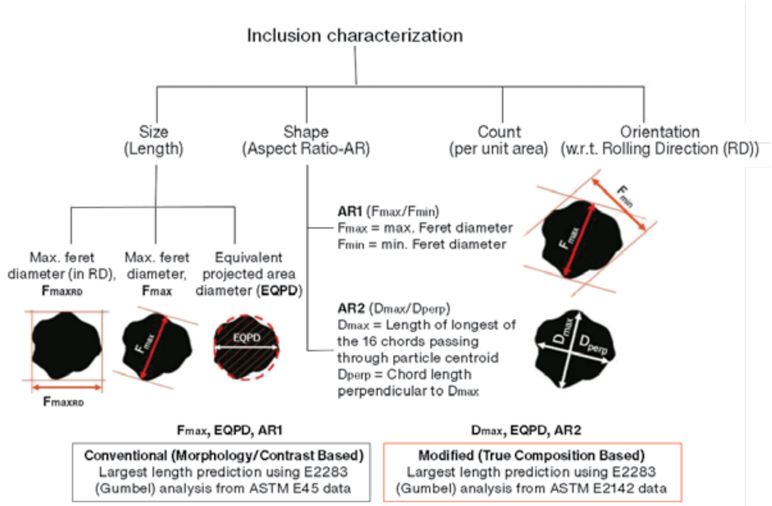
Other parameters of interest include (Fig. 2):

- D_{max}: Length of longest of 16 chords passing through the particle centroid used by ASPEX AFA software.
- AR2: Ratio of D_{max} to D_{perp} (longest chord perpendicular to D_{max}) used by ASPEX AFA software.
- EQPD: Equivalent projected area diameter, or the diameter of a circle with the same area as the particle's projection, described as $\sqrt{(4\times Area/\pi)}$ as a measure of inclusion size; Area is considered as the sum of the pixels constituting the particle as measured by the Clemex or ASPEX AFA software for globular particles (AR <2), or the product of the longest length (F_{max} or D_{max}) and maximum width (F_{min} or D_{perp}) for elongated particles (AR ≥2).

Of these, F_{max}, AR1 and D_{max}, AR2 were chosen to represent the conventional and modified approaches, respectively. This was based on the observation that most users of the automated OM or SEM-based software tend to follow the pre-set measurement dimensions that the software utilizes for rating or classification. EQPD (based on actual inclusion area) was included in both approaches as an additional parameter for comparison. EQPD is based on Murakami's parameter which showed good correlation with fatigue limit for various steels. While using the effective "area" of the inclusion does account for the size as well as shape, the basis of just taking square root of the area is unclear, hence, a more reasonable particle size estimation parameter, EQPD, that is a true length equivalent of the projected area was chosen.

Figure 2

General inclusion characterization methodology and parameters for conventional and modified approaches presented in this work.



Results and Discussion

Comparison Between Inclusion Characteristics Between ASTM E45 and ASTM E2142 Method 1

Table 3 presents the ASTM E2142 Method 1 inclusion classification rules based on chemical composition and AR. A critical AR of 2 was used to distinguish between globular and elongated inclusions, which is analogous to ASTM E45, and contingent with ASTM E2142 Method 1 description in the text (Para 4.3.1) but a ratio of 5 is reflected in the flowchart — Fig. 1 of the standard (which the authors believe is a typographical error and is sourced from now discontinued ASTM E1122 standard). Note that ASTM E45 classifies the inclusions into the broad Types A, B, C and D while

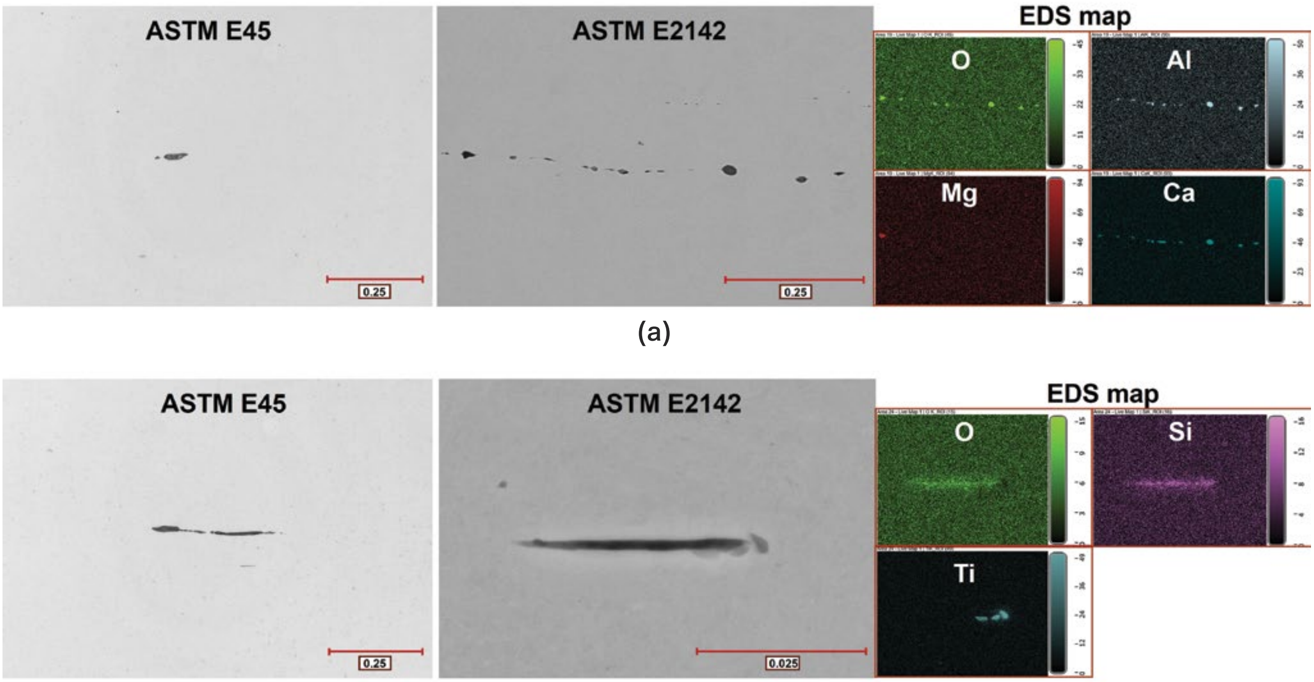
Table 3

Classification Rules for Defining Inclusion Types Using ASTM E2142 Method 1 (“No Type” are inclusions that did not fit any category defined by the standard)

Inclusion type		Classification rules (based on element wt.% and aspect ratios, AR1 or AR2)
Category	Subcategory	
A-Sulfide	MnS	Mn + S >50, AR = any
	Other sulfides: (Mn,Ca)S	Mn + Ca + S >50, AR ≥2 (elongated)
B-Alumina	(Al, Ca, Mg) oxides	Al + Ca + Mg >70, AR <2 (globular)
C-Silicate	Si oxides	Si >10, Ti <50, AR ≥2 (elongated)
D-Globular	Sulfides	S >10, AR <2 (globular)
	Oxides	Remaining AR <2 (globular)
No type	Ti (C, N)	Ti >50, AR = any
	Other	Remaining (mostly a combination of oxides/nitrides described above or foreign contaminants/non-inclusions)

Figure 3

Representative OM, SEM images with EDS elemental maps for various inclusion categories defined as per ASTM E45 and ASTM E2142 Method 1 (Scale bars are normalized by length of largest inclusion observed for the given steel).



(a)

(b)

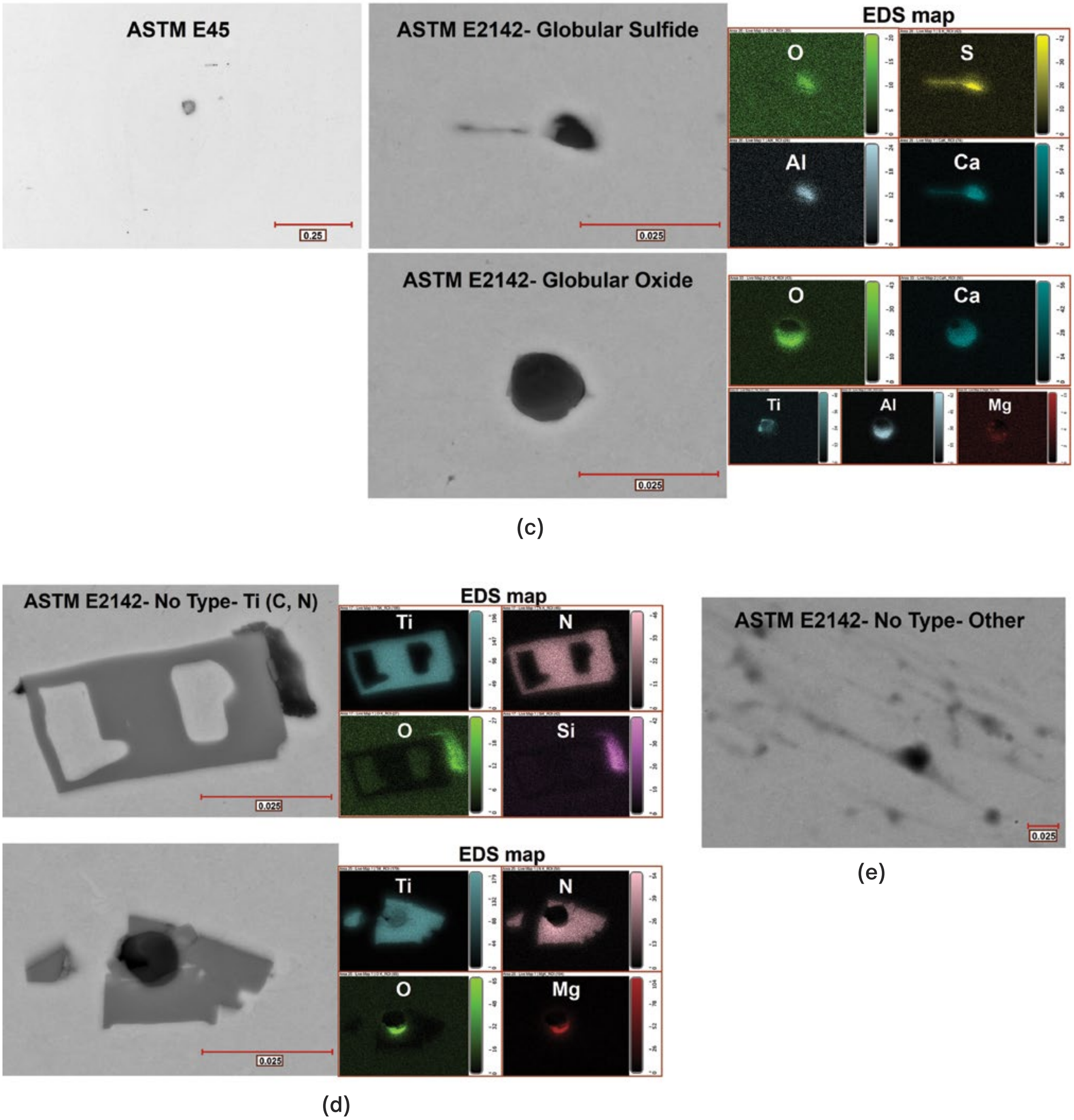
ASTM E2142 Method 1 groups them further into subcategories as shown in Table 3.

Inclusion Types: Figs. 3a–3e represent the inclusion types observed in the OM as per ASTM E45 and in SEM-EDS as per ASTM E2142 Method 1. Fig. 3e represents the SEM image for a non-inclusion feature placed in the "No Type" uncategorized inclusions that was picked up by the ASPEX AFA analysis. Note that no proper representative images of Type A Sulfide inclusions could be captured.

Count (per unit area) : Table 4 presents the results in terms of % of the total inclusion count (per unit area). A

Figure 3 (cont'd)

Representative OM, SEM images with EDS elemental maps for various inclusion categories defined as per ASTM E45 and ASTM E2142 Method 1 (Scale bars are normalized by length of largest inclusion observed for the given steel).



(c)

(d)

(e)

Table 4

Comparison of Inclusion Count Between ASTM E45 vs. ASTM E2142 Method 1 Analyses

Inclusion type		ASTM E45	ASTM E2142 Method 1
Category	Subcategory		
A-Sulfide	MnS	22.2	0.9
	Other sulfides: (Mn,Ca)S		2.5
B-Alumina	(Al, Ca, Mg) oxides	1.6	0.4
C-Silicate	Si oxides	0.2	0.5
D-Globular	Sulfides	76.0	0.7
	Oxides		9.0
No type	Ti (C, N)	Not applicable	78.8
	Other		7.2

key thing to note is the total count of inclusions identified through SEM was 3 times that observed through OM for the same observation area.

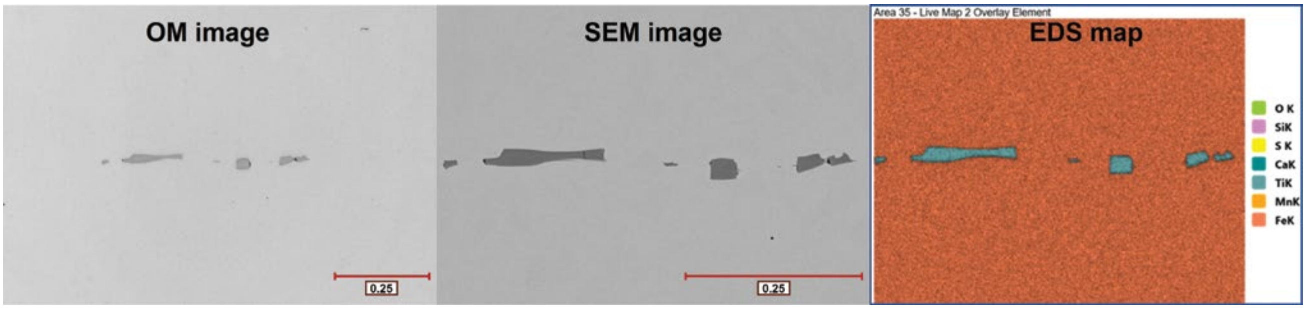
It is evident that ASTM E45 morphology-based analysis predicts a lot more Type A-Sulfides and Type D-Globular Oxides than is actually present in the steel. It was observed that some large Ti (C, N) particles (Fig. 4) were wrongly identified as sulfides through E45-based optical analysis. For Type D-Globular oxides, ~79% of them were identified as Ti (C, N) inclusions that constitute majority of the inclusion population. This shows a major gap in identifying a large population of inclusions through both ASTM E45 and ASTM E2142 Method 1 approaches that can not only lead to erroneous cleanliness data but also affect steel quality due to erroneous mitigation strategies being used to control them based on such results. For example, based on the current analysis, nitrides would get classified as oxides or in some cases sulfides by ASTM E45, while ASTM E2142 Method 1 would completely ignore them and consider sulfides as the most abundant. Based on these results, the user would then ask for strategies to control deoxidation or desulfurization which may not be effective. This highlights the significance of incorporating chemical composition-based inclusion analysis approaches for steel characterization, especially in applications where stringent control of specific inclusion types is essential for optimizing mechanical properties.

Size: While count and composition are important, the inclusion size and shape (AR) can have a major influence on mechanical properties and are often correlated directly to fracture initiation.[1,7,14] Figs. 5a and 5b present the largest inclusion size for each category/subcategory in terms of F_{max}, EQPD for ASTM E45-based analysis, and D_{max}, EQPD for ASTM E2142 Method 1 analysis. The F_{max}/D_{max}, EQPD values are normalized by the largest F_{max}/D_{max}, EQPD obtained for the given steel.

Note that for the ASTM E45 analysis, the largest F_{max} and EQPD correspond to the same inclusion. With this in mind, a noticeable difference between F_{max} and EQPD results is that using EQPD reduces the relative difference in the largest and smallest sizes, showing that it accounts

Figure 4

SEM image and EDS element map of a large Ti (C, N) inclusion wrongly identified as sulfide through ASTM E45 automated analysis (Scale bars are normalized by length of largest inclusion observed for the given steel).



for the differences in shape by including the effective inclusion area in its calculation. Further differences between the overall trends are discussed below.

Fig. 5a shows that if length is measured as F_{max}, the trend follows Type B > Type C > Type A > Type D. If EQPD is used, the trend becomes Type B > Type D > Type A > Type C. Except for the largest inclusion, the trend is completely opposite for the different size parameters even if they represent the same inclusion.

Similarly, Fig. 5b shows that if D_{max} is used, Type A (MnS) > No Type – Other > Type A (Other sulfides) > Type D (Globular Oxides). For EQPD, the trend becomes Type D (Globular Oxides) > No Type – Other > Type D (Globular Sulfides) > Type A (Other sulfides).

According to the results, ASTM E45 would predict alumina inclusions to be the largest, while ASTM E2142 Method 1 would point to MnS-type sulfides or globular oxides as the largest. This shows that depending on the type of parameter chosen to represent size, the results can be completely different even using the same standard. To pick the appropriate parameter, a study to correlate different size parameters to the mechanical properties of interest is required.

Aspect Ratio: To understand the shape or degree of deformability of the inclusion types, AR is used. This provides useful information on how an inclusion will contribute to the fracture process. AR is related to the relative plasticity of the inclusions with the steel matrix. Inclusions with high AR (deformable) are usually stretched with the steel when subjected to external stresses until they fracture, leading to cracks in the inclusion-matrix interface. Inclusions with low AR (non-deformable) cause high stress concentration when subjected to stresses and debond from the matrix, creating voids which act as cracks. However, AR alone is not sufficient to determine if an inclusion is detrimental. Thus, for high-AR inclusions, the size would be an important consideration, while for those with low AR, the count would be a better reflector of the harmful effects of the inclusion. As per ASTM E45 and ASTM E2142 Method 1, the distinction between deformable (high AR) and non-deformable (low AR) is given by the critical AR of 2.

Figure 5

Largest inclusion size for each inclusion type for ASTM E45 analysis (a); ASTM E2142 Method 1 analysis (b). (Note that the F_{max}/D_{max}, EQPD values are normalized by the largest F_{max}/D_{max}, EQPD obtained for the given steel. Also, the three largest inclusions have been indicated for each analysis method.)

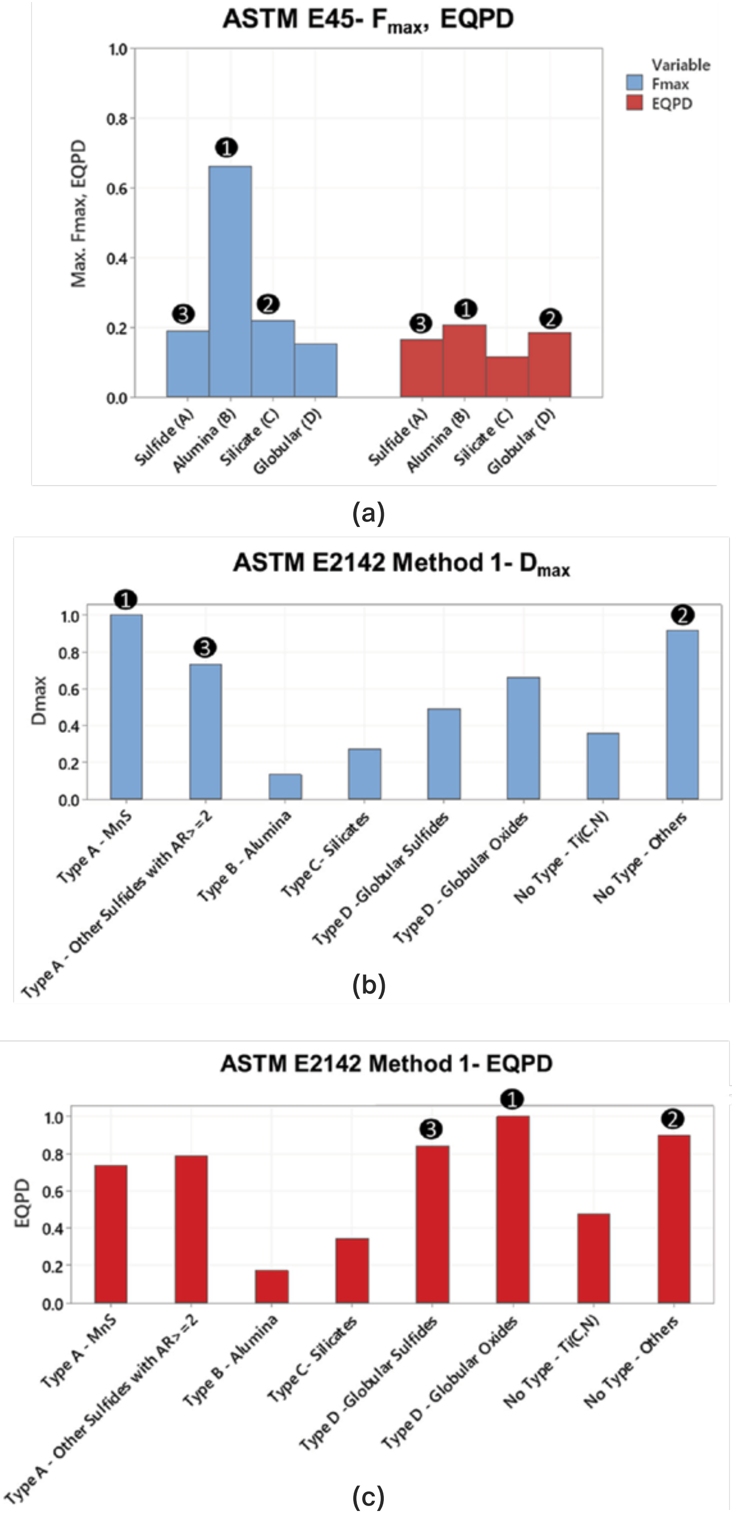


Table 5

AR Ranges, Means and AR for Largest Inclusion Defined by F_{max} /D_{max} or EQPD Parameters for Each Inclusion Type for the ASTM E45 and ASTM E2142 Method 1 Analyses

Inclusion type		ASTM E45		ASTM E2142 Method 1		
Category	Subcategory	AR range (mean)	AR for largest F_{max}, EQPD	AR range (mean)	AR for largest D_{max}	AR for largest EQPD
A-Sulfide	MnS	2–14 (4)	6	1–16 (3)	15	3
	Other sulfides: (Mn,Ca)S			2–59 (5)	5	3
B-Alumina	(Al, Ca, Mg) oxides	2–49 (10)	48	1–2 (1)	2	2
C-Silicate	Si oxides	5–17 (8)	17	2–56 (9)	3	3
D-Globular	Sulfides	1–2 (1)	2	1–2 (1)	1	1
	Oxides			1–2 (2)	2	1
No type	Ti (C, N)	Not applicable		1–90 (3)	4	3
	Other			1–56 (4)	11	3

Table 5 presents the AR ranges and means for each category for the ASTM E45 and ASTM E2142 Method 1 analyses. The AR for the largest inclusion sizes for each category/subcategory are also depicted. For ASTM E45 analysis, the AR for inclusions with the largest F_{max} and EQPD were same. For ASTM E2142 Method 1 analysis, the values for D_{max} and EQPD coincided only for alumina, silicates and globular sulfides and seemed to correspond to a somewhat globular shape (AR 1–3). For the rest of the types, the aspect ratios for inclusions with largest D_{max} were larger than those with the largest EQPD.

Comparing the E45 analysis AR ranges and means with that of ASTM E2142 Method 1, it seems that most of the other sulfides (CaS, CaS-MnS mixed) which have a higher AR are not accounted for in ASTM E45-Type A. Type B-Alumina inclusions show an opposite trend and seem to have a higher AR for ASTM E45. For Type C-Silicates, while the means for both analyses are similar, ASTM E45 fails to capture some of the higher AR inclusions encountered through ASTM E2142 Method 1. For Type D-Globular, the AR ratios are comparable.

Overall, although ASTM E45 approach fails to account for inclusions with higher AR (>49), it seems that the largest inclusion sizes from the composition-based ASTM E2142 Method 1 analysis correspond to lower ARs (1–15) and should be accounted for in ASTM E45 results.

Combining type, count, size and AR information, it can be said that:

- Conventional ASTM E45 analysis (using F_{max}) would indicate that while globular oxides will be the most abundant, they will be relatively smaller in size. This is followed by sulfides which will be slightly larger than the oxides and have a higher AR for the largest sulfide (~6). The other inclusions would be less abundant (<2%) and are less likely to cause failure.
- Conventional ASTM E2142 Method 1 analysis (using D_{max}) would indicate (assuming all "No Type" inclusions are ignored) that globular oxides will be most abundant but smaller in size, followed by other sulfides (CaS, CaS-MnS mixed). These will be slightly larger than the oxides and have a higher AR for the largest sulfide (~5). The other categorized inclusions would be less abundant (<4%) and are unlikely to cause failure.

This shows that the conventional approaches of both ASTM E45 and ASTM E2142 Method 1 are hardly adequate to have a proper assessment of inclusions in a given steel.

A comprehensive understanding of the severity or harmful index of an inclusion needs to account for not only the size but shape, count (per unit area) and orientation to the RD to account for the critically stressed region of the specimen subjected to a mechanical test. This aligns with the intent of the modified inclusion analysis approach proposed by the authors that not only accounts for a true chemical composition based categorization of inclusions based on particular steel grades/microstructures based on ASTM E2142 Method 3 but also using the inclusion characteristics data obtained from the SEM-EDS analysis to predict the largest possible values that could be encountered in the critically stressed area/volume of steel specimens tested for mechanical properties.

Comparison of ASTM E2283 Analysis Based on ASTM E45 (Case 1) and ASTM E2142 Method 1 (Case 2) Inclusion Data

Table 6 summarizes the ASTM E2283 analysis results for the ASTM E45 data-based conventional (Case 1) and ASTM E2142 Method 1 data-based modified (Case 2) approaches. The results for the largest predicted inclusion sizes in 1,000 times the examined area, along with their standard errors (SE) and 95% confidence intervals (CI) were calculated for data sets with all inclusion types mixed together as well as for the individual types defined by ASTM E45 and ASTM E2142 Method 1.

The results show that when all inclusion types are considered, both Cases 1 and 2 show similar values (<10% difference) for length parameters – F_{max} and D_{max}. However, the predicted values are ~93% higher for Case 2 when EQPD is considered. Note that another data set with all inclusion types from ASTM E2142 except “No Type – Other” was also analyzed to exclude any exogenous inclusions that may have been included in that category. As seen from the results, the predicted values for this category do not differ from those with all inclusion types by more than 10%.

For Type A inclusions, ASTM E2142 predicts ~60–80% longer inclusion length/EQPD than ASTM E45. ASTM E2142 also predicts similar values for MnS and Other sulfides. This may be due to other large inclusions such as Ti (C, N) being categorized as Type A, as discussed previously.

For Type B inclusions, ASTM E2142 predicts ~75–90% smaller inclusions than ASTM E45.

For Type C inclusions, ASTM E45 predicts similar inclusion lengths when using F_{max}/D_{max} while ASTM E2142 predicts it to be 55% longer when using EQPD. An interesting thing to note is that the F_{max} values for ASTM E45 are similar to the EQPD values for E2142.

For Type D inclusions, ASTM E2142 predicts ~50–60% longer inclusions than ASTM E45 for globular oxides. ASTM E2142 also predicts globular oxides to be up to 45–75% longer than globular sulfides.

For “No Type” inclusions, ASTM E2142 predicts ~40–50% longer Ti (C, N) type inclusions than ‘No Type – Other’ inclusions. This shows that either ASTM E2283 analysis is capable of handling data sets with exogenous inclusions or that the detected inclusions were not exogenous. Further analysis is needed to confirm this hypothesis.

Overall, the ASTM E2142 Method 1 data set with all inclusion types excluding “No Type – Other” predicted the longest inclusion size in terms of EQPD. However, with such a data set it is difficult to find out which type of inclusion will achieve such a size. Moreover, different inclusion types would have different parameters for the Gumbel distribution which may not be as accurately captured in a mixed data set. Thus, individual types need to be separately analyzed through ASTM E2283 to identify the longest inclusions.

In this case, the longest inclusion is predicted to be that of Type B by ASTM E45 with F_{max} as the length parameter and Type D globular oxides or Type C Silicates with EQPD as the length parameter. As per ASTM E2142 Method 1, the longest inclusion belongs to Type A-MnS if D_{max} is considered or Type D globular oxides if EQPD is considered. These differences in results can be attributed to:

- Absence of large Type B stringers identified through ASTM E2142 analysis that were incorrectly classified in ASTM E45 analysis.
- Inability of ASTM E45 analysis to detect larger Type D globular oxides which have a higher EQPD than Type C silicates.

Looking at the processing history of the given steel, it was observed that higher than usual amounts of deoxidizers had to be added to bring down the oxygen content, which increased the amount of deoxidation products (inclusions) and processing time. Both might have contributed to the population and growth of the globular oxide inclusions. This indicates that the latter prediction of Type D globular oxides being the longest inclusions seems reasonable. Apart from the importance of true chemistry-based classification using ASTM E2142, this also points to the fact that EQPD would be a more relevant size parameter to reflect the combined effect of size and shape (AR) for globular inclusions.

The next step would be to correlate these results using suitable mechanical tests using specimens having a critically stressed area less than or equal to the predicted area (~150,000–160,000 mm^2) such as CVN/DWTT/fatigue and confirm these predictions.

As mentioned earlier, the above work is a part of a larger campaign to develop a robust inclusion analysis approach that includes:

- ASTM E2142 Method 3 to customize inclusion classification for specific steel grade/microstructure groups.
- Identify appropriate parameters to define inclusion characteristics (shape, size, count, orientation, etc.); other parameters like TIS,[11] count and orientation will be explored.
- Select suitable inclusion type-parameter combinations as data sets for ASTM E2283-based statistical analysis.

Table 6

ASTM E2283 Analysis Results for the Conventional and Modified Approaches (SE = standard error, CI-95%= 95% confidence interval)

Inclusion type					
ASTM E45	ASTM E2142 Method 1	Parameter	Max. values	SE	CI-95%
All types mixed		F_{max}	1.10	0.12	0.25
		EQPD	0.09	0.01	0.02
	All types mixed	D_{max}	1.18	0.12	0.23
		EQPD	1.26	0.11	0.23
	All types mixed, excluding "No Type – Other"	D_{max}	1.10	0.11	0.22
		EQPD	1.38	0.15	0.29
A-Sulfide		F_{max}	0.30	0.03	0.06
		EQPD	0.17	0.01	0.02
	MnS	D_{max}	0.96	0.12	0.23
		EQPD	0.86	0.09	0.18
	Other sulfides	D_{max}	0.81	0.08	0.16
		EQPD	0.90	0.09	0.17
B-Alumina		F_{max}	1.13	0.13	0.27
		EQPD	0.57	0.07	0.13
	B-Alumina	D_{max}	0.10	0.01	0.02
		EQPD	0.14	0.02	0.03
C-Silicate		F_{max}	0.47	0.06	0.11
		EQPD	0.21	0.02	0.04
	C-Silicate	D_{max}	0.35	0.05	0.09
		EQPD	0.46	0.06	0.12

Table 6 (cont'd)

Inclusion type		Parameter	Max. values	SE	CI-95%
ASTM E45	ASTM E2142 Method 1				
D-Globular		F_{max}	0.32	0.04	0.08
		EQPD	0.48	0.06	0.12
	D-Globular sulfides	D_{max}	0.43	0.06	0.11
		EQPD	0.65	0.09	0.18
	D-Globular oxides	D_{max}	0.76	0.08	0.16
		EQPD	0.93	0.10	0.20
	No Type – Ti (C, N)	D_{max}	0.38	0.02	0.05
		EQPD	0.50	0.03	0.06
	No Type – Other	D_{max}	0.81	0.07	0.14
		EQPD	0.86	0.06	0.13

- Optimize the ASTM E2283 analysis using different applicable EV distributions.
- Correlate predictions using mechanical tests.

Conclusions

A novel methodology to quantify non-metallic inclusions has been provided in this article. This methodology can be correlated with the mechanical/fracture properties of the steel. It was demonstrated that there is a strong need to establish a practical and relevant inclusion classification and rating system that can better represent modern clean steels with varying applications. Prevalent cleanliness analysis standards like ASTM E45 rely on morphology and imaging contrast to classify inclusions and rate steel cleanliness, which was shown to produce completely different results for inclusion characteristics (count, size and AR) than when an SEM-EDS (composition)-based classification method based on another standard ASTM E2142 was used. The proposed novel approach for rating inclusions is based on prediction of the largest inclusion characteristics (D_{max} or EQPD) for individual inclusion types using ASTM E2283 statistical analysis; the inclusion types being defined by ASTM E2142 Method 3-based custom classification for particular steel grades/microstructures. Modifications to the existing ASTM E2283-based method will be to include other distributions to analyze the best fit of observed inclusion data. This not only gives a quantifiable parameter that can be linked to mechanical property performance but also helps identify suitable inclusion characteristics that guide fracture for individual inclusion types. For example, EQPD was identified as a better parameter through this work to represent combined effect of inclusion size (length) and shape (AR) for globular inclusions.

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