Steel Cleanliness: Inclusions in Steel

Steel cleanliness has become a topic of great interest to the heat treater given the reality that steel is a global-sourced commodity. Often, the type and distribution of inclusions have a significant influence on mechanical properties. It's time to review this subject so that we can continue to better understand the steel we process. Let's learn more.

The composition, size, number and distribution of inclusions determine the cleanliness of steel. Very large nonmetallic inclusions of any sort are undesirable, while very fine dispersions can be either helpful or harmful. Larger inclusions are responsible, to a greater or lesser extent, for directional properties, notch sensitivity, creep and reportedly poor fatigue properties.

There are a number of rating systems and methods to determine inclusion count, and these are commonly presented in terms of severity (Table 1 – online only). Common test methods include ASTM E 45-05e2 (Standard Test Method for Determining the Inclusion Content of Steel) and ASTM E 2142-08 (Standard Test Methods for Rating and Classifying Inclusions in Steel Using the Scanning Electron Microscope).

How are Inclusions Classified?
Nonmetallic inclusions are typically divided into the following groups: oxides, sulfides, silicates, nitrides and phosphides. Inclusions bonded with oxygen are further divided into free oxides, spinels (compound oxides in bi- and tri-configurations) and silicates (pure or admixed).

Types of Inclusions
Inclusions fall into the following general categories:

1. CaO·SiO₂·A₁₂O₃
2. FeₓMₙ₁₋ₓO·SiO₂·A₁₂O₃
3. FeₓMₙ₁₋ₓO·SiO₂·CrᵧA₁₂₋ᵧO₃
4. FeₙOₙ(oxides)
5. FeₙOₙ(oxides) + Transition Metals (lanthanides, Ti, Zr, Hf, V, Nb, Ta)
6. MgO·SiO₂·A₁₂O₃
7. MnO·SiO₂·A₁₂O₃
8. MnS (sulfides)
9. Se + Te

To facilitate identification, inclusions can be grouped based on three predominant optical characteristics: color, reflectivity and reaction to polarized light (Table 2 – online only). Some inclusions fall into multiple categories.

Here’s a brief overview of the more common types of inclusions:

1. A lumina (A₁₂O₃) is found in steel in which aluminum has been added typically to control grain size or as a deoxidant. It can also be carried into steels from refractories in which case the inclusions tend to be large and isolated. Optically, they appear as stringers, often with “comet tails” due to polishing.
2. A lumina sulfide (A₁₂S₂) is found in steels deoxidized with excess amounts of aluminum. These inclusions are less soluble in steel than manganese sulfide and separate out earlier, forming large particles.
3. Chromite (MnO·Cr₂O₃ or FeO·Cr₂O₃) is a spinel found in chromium steels or where ferrochromium has been added. Particles are angular with a tendency to segregate.
4. Chromium sulfide (Cr₂S₃) is generally found in 1% and higher chromium steels. In cast steel they are globular, triangular or rectangular in shape and often mistaken for iron sulfide (FeS).
5. Fayalite (2FeO·SiO₂) is found in steels deoxidized with silicon. Two types are typical – translucent and glassy.
6. Graphite (C) is one of the principal elements in cast irons.
7. Iron aluminate (FeO·Al₂O₃) is a spinel found in steels deoxi-
dized with aluminum. Inclusions are found in cast steels singly and as laths or tabular crystals and are often observed with iron oxide (FeO) inclusions.

8. Iron nitride (Fe$_2$N, Fe$_6$N) is found in steels high in nitrogen (or in nitrided steels), and it’s not normally classified as a nonmetallic inclusion. It occurs as needles or bands that tend to follow the grain boundaries.

9. Iron oxide is generally found with manganese oxide. In cast materials, inclusions are globular, dendritic, or angular in shape. Often mistaken for MnS (silver nitrate whitens MnS but leaves FeO unattacked).

10. Iron sulfide is found in low-manganese and high-sulfur steel (Mn:S ratio <4). It can cause hot-shortness due to its low melting point (~988°C (1810°F)). In cast material, it is often observed at grain boundaries.

11. Manganese-iron-silicates are a complex inclusion type in which the silicates can be either acidic (large amounts of silicon with some manganese going into solution) or basic (average amounts of silicon and average-to-low amounts of manganese).

12. Manganese sulfide is the most common type of inclusion encountered. According to analysis based on the steel ingots containing 0.01-0.15% sulfur, the morphology is typically classified into three types:

   a. Type I are silicon-killed steels (or steels incompletely killed with aluminum, zirconium, or titanium). MnS are globular with a wide range of sizes and often duplex with oxides.
   b. Type II are aluminum-killed steels with no excess of aluminum having a dendritic structure (called grain-boundary sulfide because MnS is forms on grain boundaries).
   c. Type III are found if excessive amounts of aluminum are present or if magnesium is used as a deoxidant. They are angular and always form as monophase inclusions.

Massive spherical inclusions are found in high-sulfur, free-machining steels. In this type of steel, low silicon contents give inclusions with the best length-to-width ratio for optimum machinability. Type I and Type III are preferred since Type II is associated with low ductility.

13. Manganese-aluminum-silicate (2MnO·Al$_2$O$_3$·3SiO$_2$) forms in the presence of manganese oxide, aluminum oxide, and silica. Large inclusions have six sides, and smaller inclusions are globular in appearance.

14. Titanium carbonitride (Ti(CN)) is found in high carbon and alloy tool steels and in plain-carbon steels that have been killed with ferro-titanium. Particles are well-defined and angular and are often mistaken for titanium sulfide.

15. Titanium oxide is found in steels deoxidized with titanium or ferro-titanium. Four oxides of titanium are commonly found (Ti$_2$O$_3$, TiO, FeO·TiO$_2$, Ti$_2$O$_3$). TiO appears as cubic crystals often found near Ti$_2$O$_3$ inclusions that are globular in shape and often contain iron. TiO is often confused with alumino-silicates.

16. Zirconium nitride (ZrN) is found in steel that has been deoxidized with zirconium. In cast materials, inclusions can be square, rectangular, triangular, trapezoidal, or octagonal. Low zirconium content produces softer inclusions. May be found within an oxide inclusion.

17. Zirconium sulfide (ZrS$_2$) is found in steels deoxidized with zirconium. Inclusions with less zirconium tend to deform more easily. In cast material, the inclusions are globular.

Scanning Electron Microscopy (SEM) Methods

Since modern steels have fewer and finer nonmetallic inclusions, the use of scanning electron microscopy (SEM) has become popular for those who have this tool available. Individual inclusions often consist of more than one type, such as sulfides and silicates, so energy dispersive spectroscopy (EDS) analysis is a useful tool. EDS offers both point analysis and X-ray maps to distinguish among types of inclusions directly, not just by inference from shape.

ASTM E 2142 defines three methods of inclusion analysis. Method 1 is analogous to E45 or E 1122 for automatic image analysis. Method 2 uses EDS to sort the inclusions into traditional composition classifications as defined in E 45. Method 3 allows customization into classes as necessary, and it is this method that is particularly useful for duplex or complex inclusions. Although the standard is written for inclusions in steel, the method could be extended to other discrete second phases or particles.

Inclusion Control

An example of manipulation of inclusions is the modification of composition and morphology of oxides and sulfides in calcium-killed steel. Some of the reported benefits include improved castability, avoidance of tearing and reheat cracking in welding applications, improved machinability and increases in both tensile ductility and tensile strength. IH

References (online only)