

Considerations to be taken when performing point counting of duplex stainless steels

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ABSTRACT

Ferrite content is considered important for many reasons and the degree of relevance and optimal level is discussed in different forums. In order to even discuss different ferrite levels it is important to have accurate measurement methods. Recently some considerations that should be taken into account using a ferrite scope have been presented¹. In the present paper some considerations for measuring the ferrite content according to ASTM E562² are explored. The different areas that should be considered relevant when performing point counting are:

- sample preparation, including both polishing and etching
 - o Every step of a polishing procedure must remove the deformed volume from the previous step
 - o A clear definition of location of phase boundary, decided and verified. Normally one should not choose the dark outline caused by an etchant.
- Adequate sample types for measurements and relevance to final result.
 - o Measure cross-sections
 - o Consider only mean values as result and not distributions
- Measurement setup and helpful strategies
 - o Use as low magnifications as possible still fulfilling recommendations in ASTM E562² for size of grid or other standards setting specific magnification.
 - o Actively decide location of grid point, especially close to phase boundaries. Use zoom as a tool. Introduce rules for decisions for consistent results.
 - o Verify procedure and strategy by reference measurements

Key words: Duplex stainless steels polishing point counting ASTM E562² ASTM E1245⁵ etching Beraha KOH

INTRODUCTION

The ferrite content has over the years more or less always been in focus as a feature for duplex stainless steels that needs to be controlled, or at least measured, as it has been shown to affect many different properties^{3 4}. Generally, it can be stated that the relevance of keeping certain ferrite levels changes for different properties and, as ferrite measurement methods always have an intrinsic inaccuracy, it has been suggested that relevant requirement on properties should be better than strict requirement on ferrite contents³.

Common methods for measuring the ferrite content are metallographically by image analysis (ASTM E1245⁵) or point counting (ASTM A562²), and by permeability with ferrite scopes. There are also other methods, both metallographic or magnetic^{1 4}. A method now relatively available that could be considered as a reference method is electron backscatter diffraction (EBSD) in the scanning electron microscope (SEM).

Historically, the most used method is point counting and it is considered a good and normally relatively quick and easy method to use, but with some drawbacks. The method is somewhat subjective and can also be somewhat tedious. With this method, a grid of points is distributed over a number of image fields taken from an etched cross-section of the material. Points that fall in ferritic areas are counted and mean value of ferrite content and statistics are estimated.

For different duplex stainless steel materials many types of microstructures may appear. Depending on whether a cast material or a hot- or cold-rolled material is to be examined, the microstructure may differ extensively. It may also be a weldment and sometimes quite local areas are of interest, for example the high temperature heat affected zone (HTHAZ).

Sometimes the examination is very straightforward and normally considered easy but sometimes different factors make examinations harder or at least require particular care. One case could be if the microstructure is small and high magnifications must be used making decisions of point location harder.

Some considerations and suggestions for improving point counting methods are reviewed in this report by presenting and discuss some measurement examples.

EXPERIMENTAL PROCEDURE

Material that has been used for chosen examples has been 2507 super duplex stainless steel, see Table 1 for chemical analyses.

Table 1 Nominal composition of material examined

UNS	EN	ASTM	C	Mn	Cr	Ni	Mo	N
S32750	1.4410	S32750	0.02	0.5	25.0	7.0	4.0	0.27

Sample preparation methods included grinding on stone, diamond polishing at 9µm & 3µm, and oxide polishing at 0.25µm. Relatively normal polishing steps following stone grinding were performed, which consists of ~4.5 minutes of polishing at 9 µm, ~two 3-minute polishing steps at 3 µm and 2 to 3 polishing steps for 3.5 minutes at 0.25 µm.

One example shows a failed polishing due to exclusion of the 9 µm step followed by too short polishing steps afterwards.

The etchants used in the shown examples are KOH, NaOH and a modified Beraha II solution.

- Etching with KOH (45gr in 60ml H₂O), performed electrolytically at 2V for 6 to 15 seconds.
- Etching with NaOH (40gr in 100ml H₂O), performed electrolytically at 2V for 6 to 15 seconds.
- Etching with modified Beraha II (33ml HCl, 66ml H₂O and 1gr of K₂S₂O₅) is performed at RT for 1 to 3 s.

The method that will be discussed for different examples is ASTM E562².

RESULTS AND DISCUSSION

Some decisions that need to be made before starting a point counting examination of a chosen material are listed below.

Area for chosen cross-section:

Normally one should choose a cross-section transverse to the rolling direction if a sample from a rolled material is examined. Generally, the result should represent a mean value of the entire cross-section and no or very little information of distribution will be included in the result. Measuring areas from surface to surface can also be of interest but is usually only done for development purposes.

Importance of preparation of cross-section:

It should be emphasized that a proper polishing procedure needs to be used before etching takes place. One problem that may happen is that polishing steps have been too few or that certain steps have not managed to adequately remove deformed regions of previous polishing step. If this happens a false result may be achieved after etching. As shown in Figure 1, the austenite phase may be smeared over the ferritic phase giving too low ferrite content.

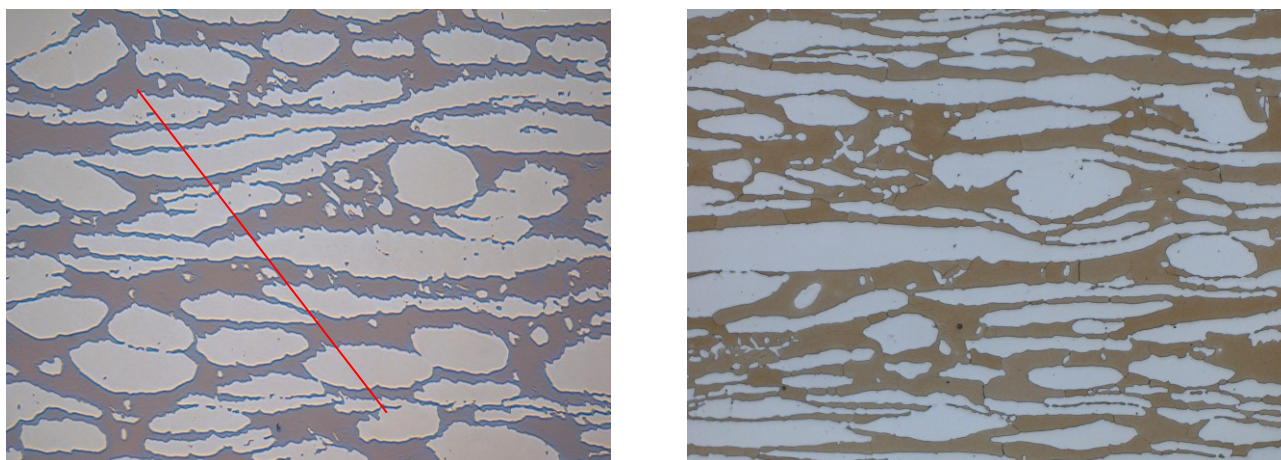


Figure 1 Example of austenite smeared over the ferritic phase to the left compared to a polishing procedure with no smearing to the right. The direction of smearing is shown by inserted line. Etching in KOH to the left and NaOH to the right.

A similar case to that above occurs if deformed material from a cut edge remains after the final polishing. Then similar effects may be seen after etching.

Type of etchant chosen:

Generally, one should choose an etchant giving high enough contrast between austenite and ferrite and giving sharp phase boundaries. For point counting, high contrast is not as critical as for image analyses, except when a semiautomatic method/tool is used in which thresholding is used for first “suggestions of phase”. For the latter the user then accepts or changes the suggestion made by the tool.