LECO MET-TIPS Ideas for Metallographic Procedures

Revealing Delta Ferrite in Martensitic Stainless Steels

When working with martensitic stainless steel alloys or stainless steel welds, it is often beneficial to know the volume fraction (or percentage) of the delta ferrite phase that may be present. It can be difficult to clearly detect the delta ferrite phase when martensitic stainless steels are etched with the most commonly used etching reagents because the entire microstructure is revealed. Vilella's reagent (1g picric acid, 100 ml ethyl alcohol, 5 ml hydrochloric acid) is one of the typical etchants used for the martensitic stainless steels (e.g. 400 series, 17-4 PH and 17-7 PH alloys). Although delta ferrite is not attacked by this etchant, it still can be difficult to detect in the martensitic matrix.

Here, we showcase a method where only the delta ferrite phase is revealed, leaving the martensitic phase unaffected.

Perhaps the most commonly used method to accomplish this is electrolytically etching a conventionally prepared metallographic sample in a 20% aqueous solution of Sodium Hydroxide (NaOH). The solution can be made in bulk volume and used as needed since it has an indefinite shelf life. Etching is done at room temperature for approximately five seconds using an open circuit voltage of 20V. Austenitic stainless steel is used for both the cathode and the anode probes.

Results from each of these etchants are illustrated in the photomicrographs in Figures 1 and 2. Figure 1 shows a 17-4 PH stainless steel etched with Vilella's reagent, showing the martensitic microstructure. The delta ferrite phase is nearly impossible to resolve. Figure 2 shows the same field as Figure 1 but etched electrolytically with the 20% NaOH etchant. Only the delta ferrite phase is revealed, leaving the martensitic matrix unaffected. This etchant has been used on a wide variety of austenitic as well as martensitic stainless steel alloys and has been found to be very reproducible.

The 20% NaOH electrolytic etchant was developed primarily for when quantitative analyses were needed to determine the volume fraction of delta ferrite present. The analysis was initially conducted via a grid superimposed on the image, and the volume fraction was calculated using the grid intersection points. This is referred to as the “point count” method. More recently, volume fraction analysis is commonly available using Image Analysis software. Image Analysis software requires a certain level of contrast between the phase being analyzed and the surrounding matrix. Comparisons between Figures 1 and 2 illustrate the variation in contrast obtained by the two etchants. The contrast between the delta ferrite phase and the martensitic matrix in Figure 1 would not permit quantitative analysis using Image Analysis software; however, the delta ferrite volume fraction revealed in Figure 2 can easily be analyzed by either method.

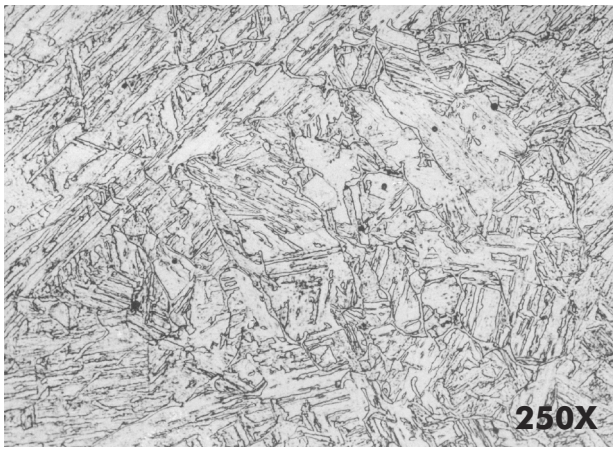

Figure 1. Vilella's reagent—the delta ferrite phase is difficult to detect in the martensitic matrix.

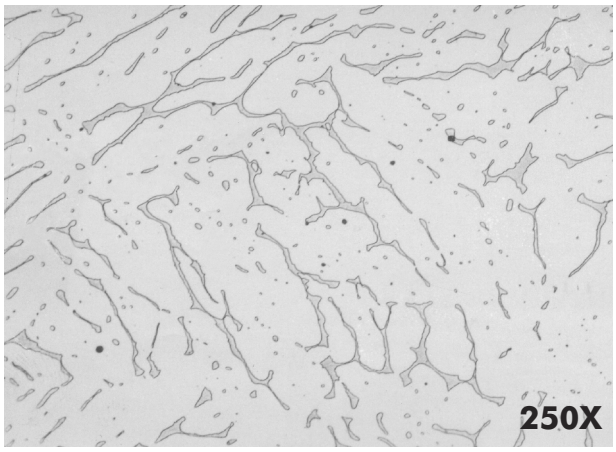

Figure 2. 20% NaOH electrolytic etch—the ferrite is stained dark and is readily distinguished from the martensitic matrix.

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