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NIST IR 8435

Acceptance of new batches of 9310 steel for the Charpy Machine Verification Program

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Acceptance of new batches of 9310 steel for the Charpy Machine Verification Program

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Abstract

During FY2021, one of the NIST Charpy Contractors returned 43 blanks (rough-machined, oversized, unnotched specimens) of 9310 steel that presented visible black surface defects (similar to gouges, some with raised strips of steel). On 2 of these blanks, optical microscopy and chemical analysis by electron dispersive spectroscopy were conducted in order to establish the nature of such defects. It was concluded that they correspond to segregation zones formed during steel-making, which the final processing failed to remove. These surface layers (< 1 mm) are brittle in nature and are likely to be removed during sample machining.

Keywords

9310 steel; Electron Dispersive Spectroscopy; segregation zones; surface defects.

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1. Introduction

In the month of October 2021, one of the Contractors of the NIST Charpy Machine Verification Program informed us that some Charpy specimen blanks they had obtained from a specific heat[1] of AISI 9310 steel (#J0861), used to produce super-high energy reference specimens, exhibited large surface defects, which they called “some sort of pockets” and “unmachined surfaces”. Figures 1-3 show the photographs sent to NIST by the Contactor to document their observations.

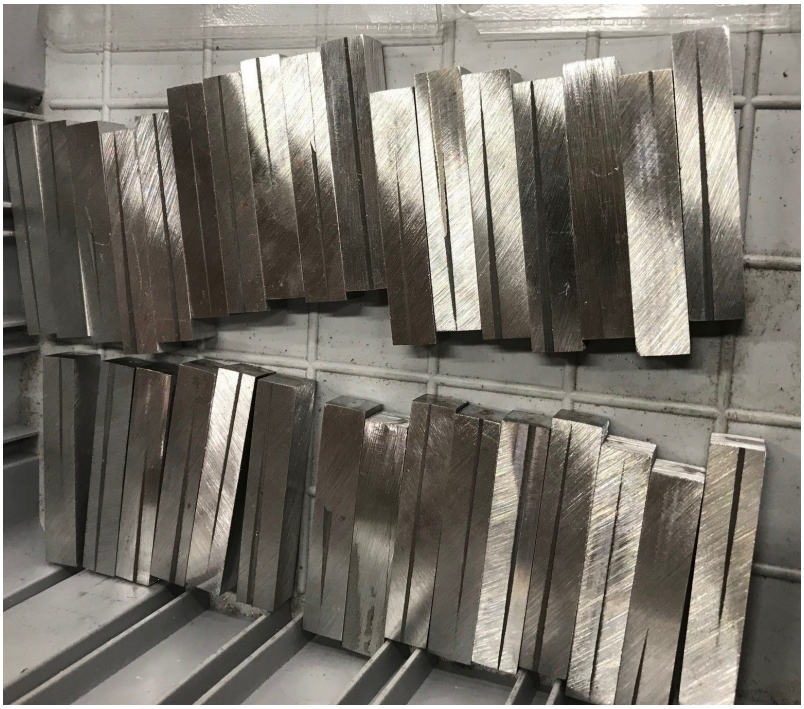

Figure 1 - Blanks obtained from Lot 11 of Heat #J0861 of 9310 steel.

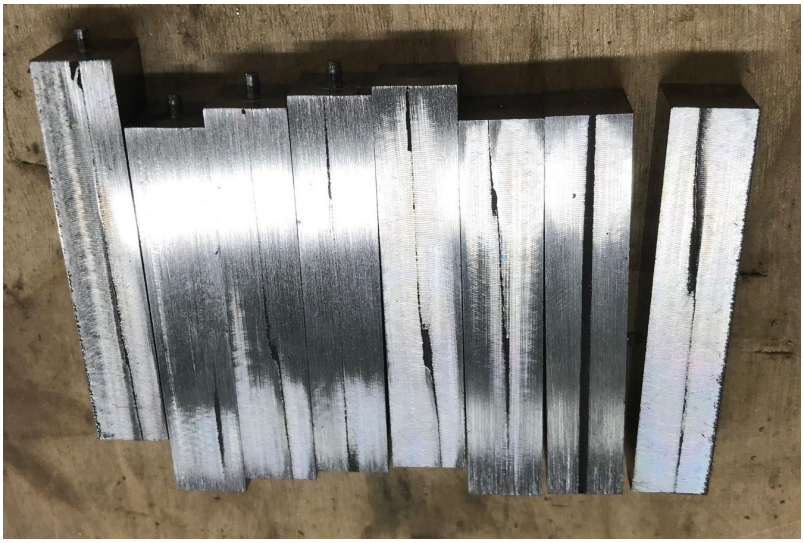

Figure 2 - Blanks obtained from Lot 35 of Heat #J0861 of 9310 steel.

[1] By “heat” of steel, we mean “The product of a single melting operation in a furnace, starting with the charging of raw materials and ending with the tapping of molten metal and consequently identical in its characteristics.” (see www.engnet.us, Engineering Network, accessed 9/8/22.)

Figure 3 – Three 9310 steel blanks obtained from Lot 35 of Heat #J0861 of 9310 steel.

The Contactor was instructed to send the defective blanks back to NIST for evaluation. The results of the investigations performed are provided in this report.

2. Investigations performed and results obtained

The rough-machined blanks received from the Contactor showed regions of long black defects that appeared like gouges on the surface. Some of these gouges were associated with strips of steel that could be pulled away from the surface, leaving a black groove behind (Figure 3).

Optical micrographs of these defects are presented in Figure 4.

One sample from each lot that had been found to exhibit these defects (lots 11 and 35) was sectioned such that the defect was viewed in cross-section. This section was then mounted in bakelite (standard for metallography), polished to a mirror finish, and etched with a 2 % nitric acid in methanol solution (nital). Optical microscopy was conducted on these samples (Figure 5). They show a definite divot in the surface due to these gouges, which measures approximately 150 μm. This depth is comparable to that measured by the differences in focal plane for the top and bottom of the feature, 200 μm to 300 μm. The black layer of the defect is visible in cross-section, Figure 6, but appears to be quite thin (10 μm to 20 μm). This layer is not uniform in thickness nor is it flat, but it infiltrates into the steel surface, much like the behavior of corrosion product in corrosion pits. The section also shows no large defects running through the bulk of the material, just the surface defects. (Note that no large bulk defects were observed prior to or after etching, suggesting the defects are primarily located at or near the surface.)

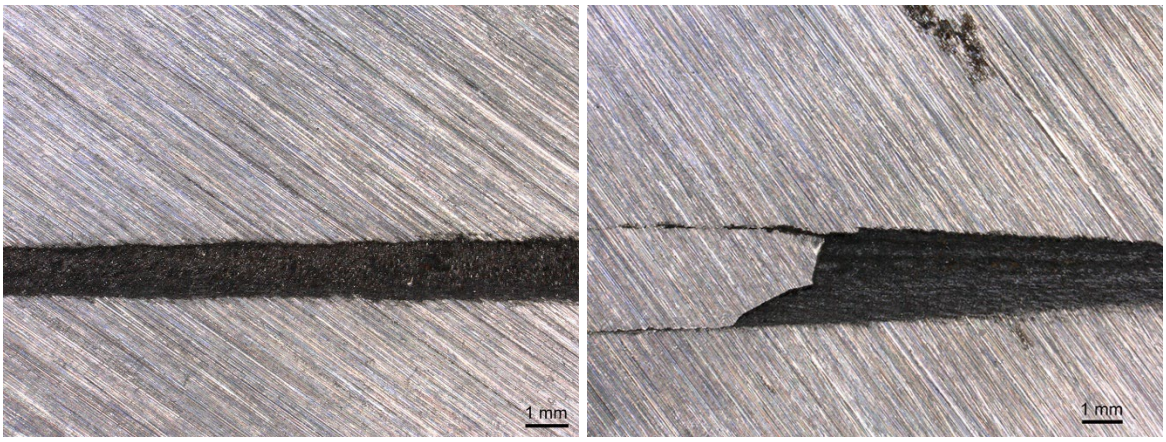

Figure 4 - Optical Micrographs showing the investigated defects. Both blanks were from lot 11, but the appearance is representative of the defects. The left picture shows section of defect extending along entire blank. The right picture shows a defect with a steel sliver remaining over a portion of it.

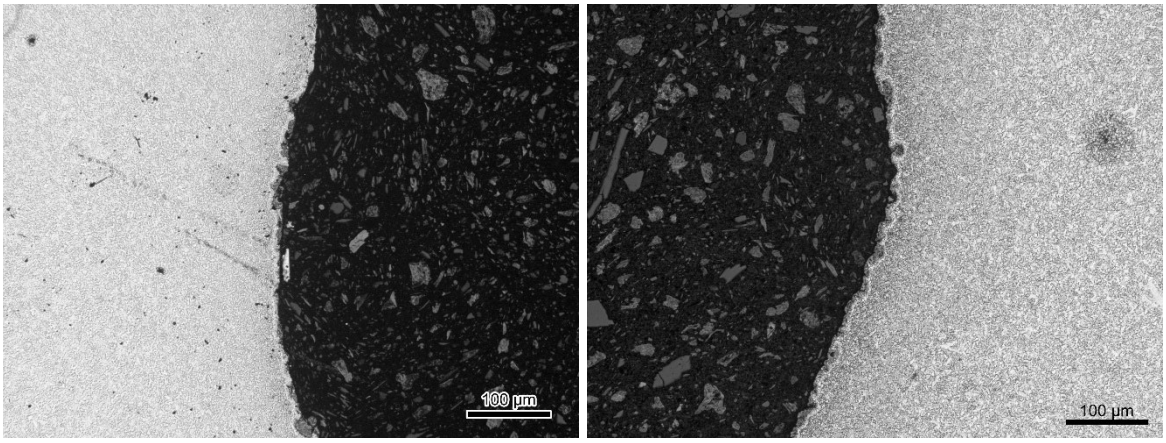

Figure 5 - Optical micrographs of mounted, sectioned, and etched samples showing the cross-section of the divots. The left picture shows a sample from Lot 35, while the right picture shows a sample from Lot 11. The black material is the bakelite mount.

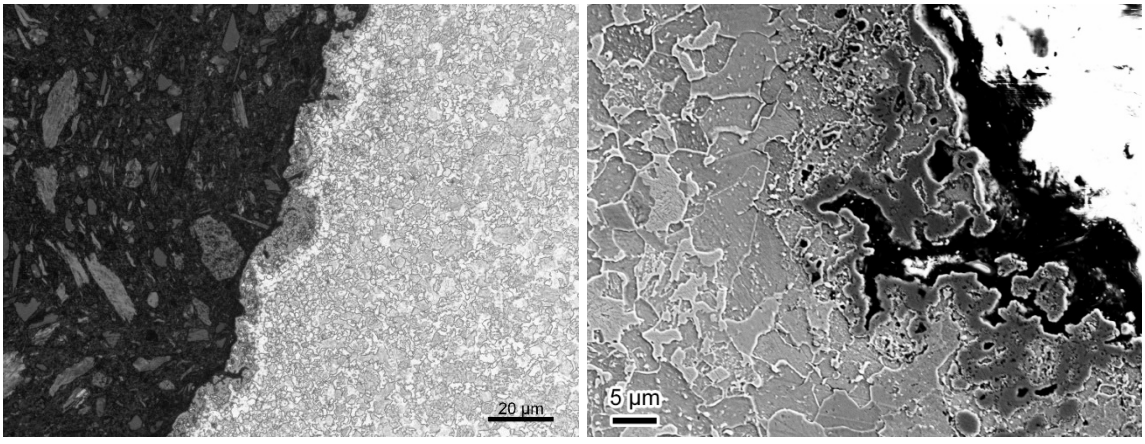

Figure 6 – Left picture: high magnification optical micrograph of base of sectioned divot, sample from Lot 11 (Figure 5 right). Note the thick (10 µm to 20 µm) dark grey layer at the surface of the steel. Right picture: SEM micrograph of the cross-section showing infiltration of the (darker) surface layer into the steel microstructure.

Chemical analysis by electron dispersive spectroscopy (EDS) was conducted on a scanning electron microscope (SEM) operated at either 10 kV or 30 kV. EDS uses characteristic x-rays generated by the sample interacting with the electron beam to determine the elemental composition of the material. Spectra taken from the surface of these areas reveal the presence of potassium, silicon, and oxygen (Figure 7), with the exact components and the relative contributions to the spectra varying as a function of position. The spot scans still reveal iron, chromium, and nickel signal, but the possibility remains that these could be due to background contributions (underneath the thin surface layer) rather than due to the metallic contributions within the black layer.

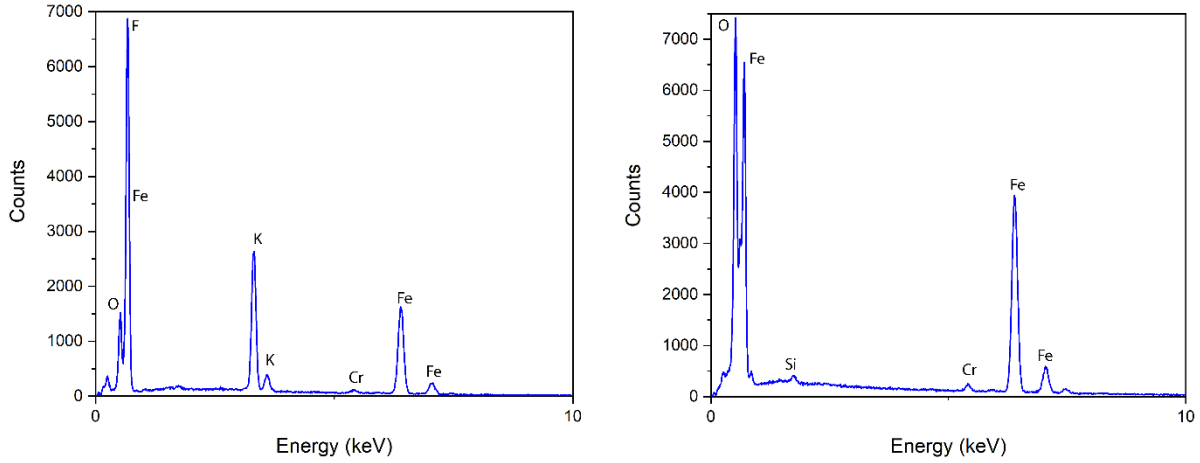

Figure 7 - EDS spectra from two different points on the surface of the defect.

The EDS chemical analysis of the general surface of the cross-section shows an oxygen-rich layer approximately 500 nm thick, consistent with a native oxide surface layer. Within the pit itself, the surface layer becomes larger (Figure 8). The chemical composition again varied by position, but suggested that the layer was generally rich in oxygen, with some areas rich in silicon. It is possible that the phases that were rich in lighter elements like potassium were dissolved on the surface layer by the acid etchant, and that is why they were not detected in the sectioned samples.

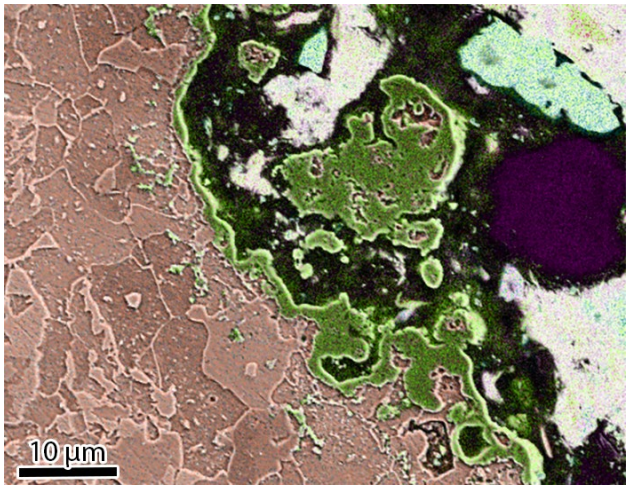

Figure 8 - Element color maps overlaid on SEM micrograph. Green is oxygen-rich area, orange is iron-rich area.

3. Conclusions

The composition and appearance of the material inside these defects suggest that they are segregation zones formed during steel-making, which were not properly removed during the final steps of processing. While the phases were not identified though diffraction analysis, the results presented here are consistent with oxides and other slag components. The flaking off of the covering steel surface layer, and the composition, suggest that this material is brittle. As it appear to be confined to the surface (< 1 mm), it is possible that this layer will (or can) be mostly removed during sample machining. The bluntness of the shape of these defects means that they are unlikely to be strong stress concentrators during mechanical loading and, therefore, are unlikely to affect Charpy testing performance. However, they are an undesirable and visually noticeable feature that should be avoided in the future.