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Chromium Segregation at the Grain Boundaries in Ni-Fe-Cr Alloys.

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Mechanical strength of ternary metal alloys usually depends upon the grain boundary characteristics. Stress corrosion cracking, brittleness, oxidation behavior, mostly originating at the grain boundaries can cause long term durability and stability problems in virtually every manmade metallic structure from spacecraft to naval vessels to nuclear reactors. Chromium based diffusion in stainless steel or other Ni-Fe-Cr based ternary alloys are one of the open scientific problems that can compromise the stability and corrosion resistance of metals [1]. Ni-Fe-Cr alloys in the austenite form are particularly prevalent in the space and jet engine industry for their improved resistance to the stress corrosion cracking [2]. Ni-alloy based components are also used extensively as interconnects and seals in solid oxide fuel cell [3]. Modeling shows that chromium precipitation mainly occurs at high angle grain boundaries. The angular misorinetation between the two grains was believed to be the main reason for Cr segregation [4].

In this paper, we analyze Cr grain boundary segregation in Ni-Fe-Cr alloys by simultaneous EBSD and EDS mapping. A polished Ni-Fe-Cr alloy with an approximate composition of 75-10-15 was chosen for the analysis. Simultaneous EBSD and EDS mapping of Ni-Fe-Cr alloy was performed using a Hikari high speed EBSD detector from TSL/EDAX and Genesis EDS system from EDAX mounted on a FEI Quanta FEG dual-beam microscope. EBSD mapping data was collected at various speeds ranging from 20-250 fps with planar indexing accuracy over 95%. The results in Figure 1 are EDS maps collected from the Ni-Fe-Cr sample. The last image in Figure 1 is an EBSD image quality map showing the grain boundary structure in the alloy. A uniform spread of Ni and Fe was observed in the alloy. In the case of Cr, it was noticed that apart from being distributed in the alloy, preferential segregation of it at the grain boundaries was quite visible. Because the scales of EBSD image quality map and EDS maps are same, the grain boundary structure seen in the EBSD band contrast image can be compared to accumulated Cr sites to conclude Cr segregation at the grain boundaries.

The inverse pole figure (IPF) grain orientation map from the same region is compared to the band contrast image in the Figure 2 (a, b). The inset of Figure 2(a) is SEM image of Ni-Fe-Cr alloy. The difference in the contrast imaging from the backscattered secondary electrons indicates well defined grain boundary structure. A detailed grain boundary analysis was performed from the IPF image in Figure 2(b). The crystal orientation of each grain and the colors associated with them are shown in the schematics and ternary IPF orientation plots. The dark lines crossing each grain boundary corresponds to the significant Cr segregation boundaries seen in EDS map of Figure 1. The white lines crossing the grain boundaries indicate that no substantial Cr segregation was noticed across those boundaries. We have calculated the grain boundary misorientation angles between each grains that are numerically shown in the Figure 2(b). No substantial grain boundary angle Cr-segregation dependence was noticed. Due to purely geometric nature of the high angle grain boundary phase segregation model, more realistic Cr segregation conditions may need to be considered. In the detailed version of this study, we plan to utilize additional analytical capabilities and consider additional models such as structural units model, which emphasizes favored and non-favored grain boundaries for chromium segregation [1, 5].

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[5] The research was performed using EMSL, a national scientific user facility sponsored by the DOE's Office of Biological and Environmental Research located at PNNL. PNNL is operated by Battelle for the US DOE. The work support for is provided by BER under capability development funds at EMSL through the grant contract DE-AC06-76RL01830.

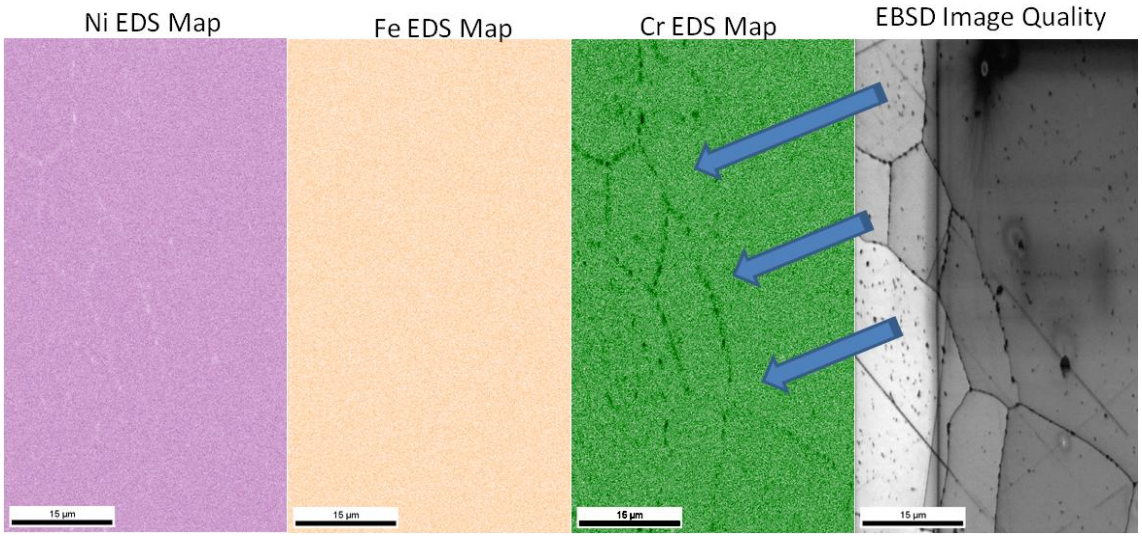

Figure 1. EDS elemental maps of Ni, Fe and Cr at the location indicated in EBSD band contrast map

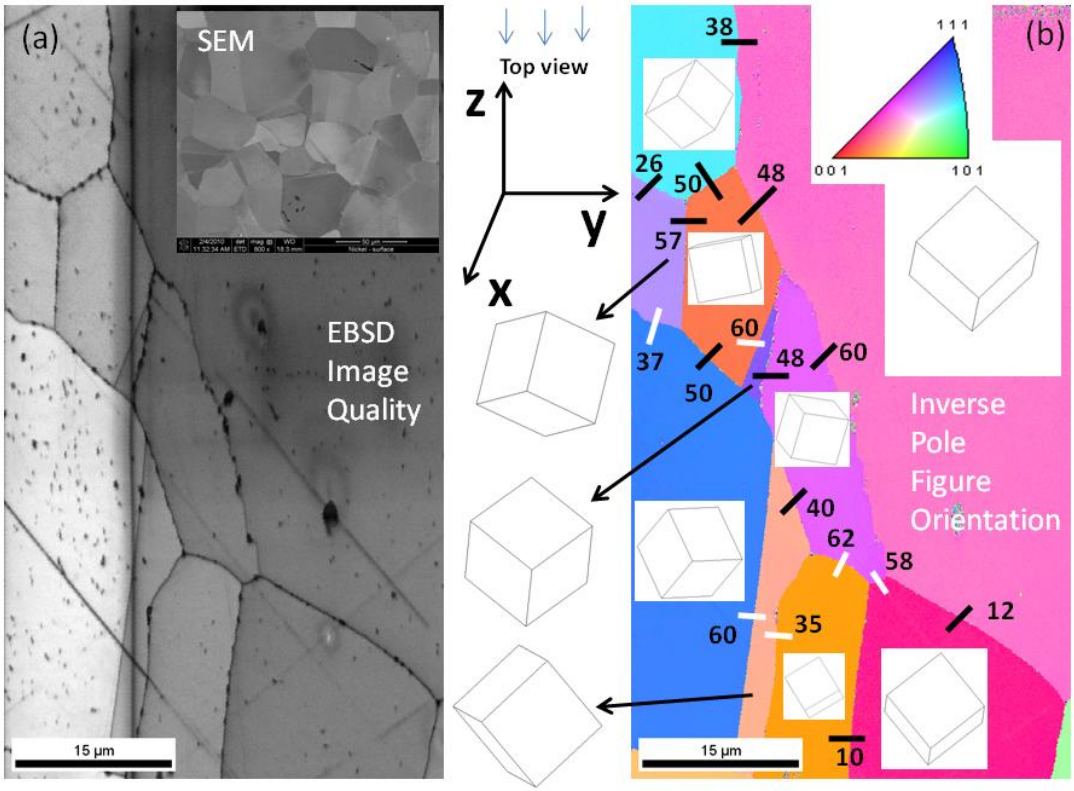

Figure 2- (a) EBSD band contrast image of Ni-Fe-Cr alloy. Inset: SEM image of the alloy. (b) IPF grain orientations and grain misorientation angles. The dark and white bars are possible preferred and non-preferred Cr segregation grain boundaries.

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