2.1 The Structure of Nanoscale Precipitates and Precipitate Interfaces in an Oxide Dispersion Strengthened Steel, — J. Ciston (National Center for Electron Microscopy, Lawrence Berkeley National Laboratory), Y. Wu and G. R. Odette (Materials Department, University of California, Santa Barbara), P. Hosemann (Department of Nuclear Engineering University of California, Berkeley)

OBJECTIVE

The objective of this work is to characterize the detailed structure of nano-scale oxides in and referece ODS alloy MA957 including the interfaces with the ferritic matrix.

SUMMARY

Fusion energy will require developing new, high performance irradiation tolerant structural materials, like oxide dispersion strengthened nanostructured ferritic (ODS) alloys [1]. Fe-14Cr based NFA contain an ultrahigh density of Y-Ti-O enriched nanofeatures (NF) that provide both outstanding high temperature properties and remarkable tolerance to irradiation damage, including the transmutation product He. Understanding the detailed characteristics of the NF is critical to optimizing ODS alloys, but this remains an unresolved issue and a subject of continuing controversy.

PROGRESS AND STATUS

Experimental Methods

We present the initial results of a study on a reference NFA, MA957, using the TEAM 0.5 aberration corrected instrument at the Lawrence Berkeley National Laboratory National Center for Electron Microscopy. The MA957 examined is in a friction stir welded and annealed (FSW-A) condition that has been previously self-consistently characterized by a number of different techniques. Previous TEM measurements showed that this alloy contains ≈ $2.3\text{x}10^{23}$ $Y_2Ti_2O_7$ pyrochlore polyhedral oxides per m^3 with an average diameter of ≈ 2.4 nm and a near stoichiometric composition [2]. However, previous structure and composition measurements, which were made on extracted oxides, proved to be difficult to impossible when the oxides were embedded in the Fe-Cr matrix.

The FSW-A foil was FIB thinned further prior to examination using the TEAM 0.5. The characterization included STEM EELS profiles and HAADF imaging as well as HREM focal series imaging. The resulting rich and detailed database is under continuing analysis, but there are already several important preliminary results.

Preliminary Results

The $Y_2Ti_2O_7$ pyrochlore structure of he nanooxides found in previous studies was confirmed. and $[001]_{fe}//[1\text{-}10]_{ox}$-(100)Fe//$(100)_{ox}$ orientation relationship for the several oxide inclusions were confirmed. The HRTEM image and indexed FFT shown in Figure 1 are for a ≈ 1.8x3.8 nm feature. The FFT is consistent with a fluorite structure for the oxide phase with measured d-spacings of the (400) and (222) planes that are slightly larger than for the bulk oxide by ≈ 6.7 and 3.3%, respectively. This small oxide appears to be fully coherent with the ferrite matrix. The exit wave phase from a focal series restoration of a larger 6.2x10.1 nm particle is shown in Figure 2. In this larger particle, the $Y_2Ti_2O_7$ exhibits smaller d-spacing differences from the bulk

of 1.4 and 1.2%, respectively. The larger particle is coherent with the bcc matrix along the vertical edges, but is incoherent at the top and bottom horizontal edges. In addition, it can be clearly observed from the exit wave that the Fe-Cr matix is strained in tension along the top and bottom edge of the particle. The EELS Ti profile in Figure 3 as well as HAADF count (not shown) profiles suggest that the feature has beveled edges in the beam direction, and is not a true rectangular prism. While the oxide is enriched with Ti relatice to the matrix, there does not appear to be significant Ti segregation at the interface [3].

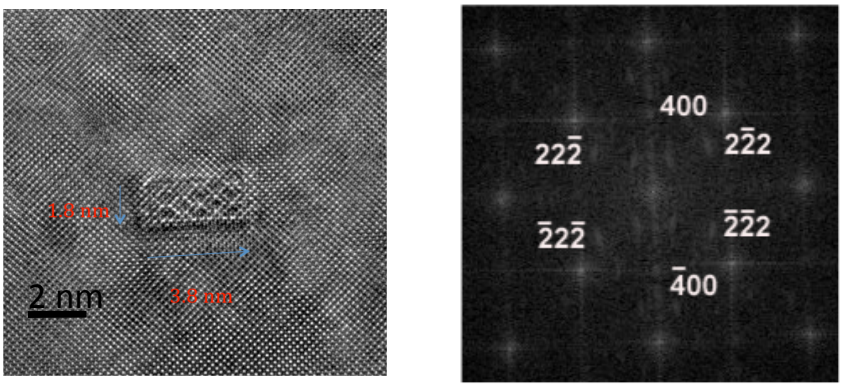

Fig 1. HRTEM image of a NF in FSW-A MA957 (left) and the corresponding FFT indexed to the $Y_2Ti_2O_7$ phase (right).

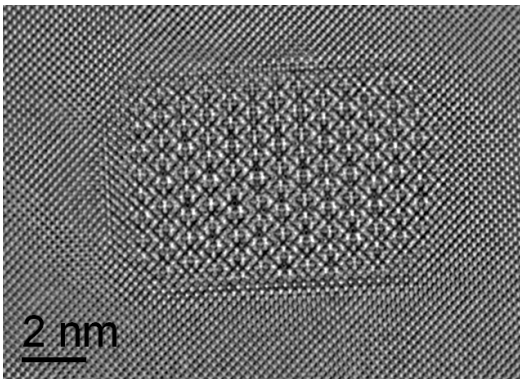

Fig 2. Phase of restored exit wave of FSW-A MA957 particle indicating direction-dependant interfacial coherence.

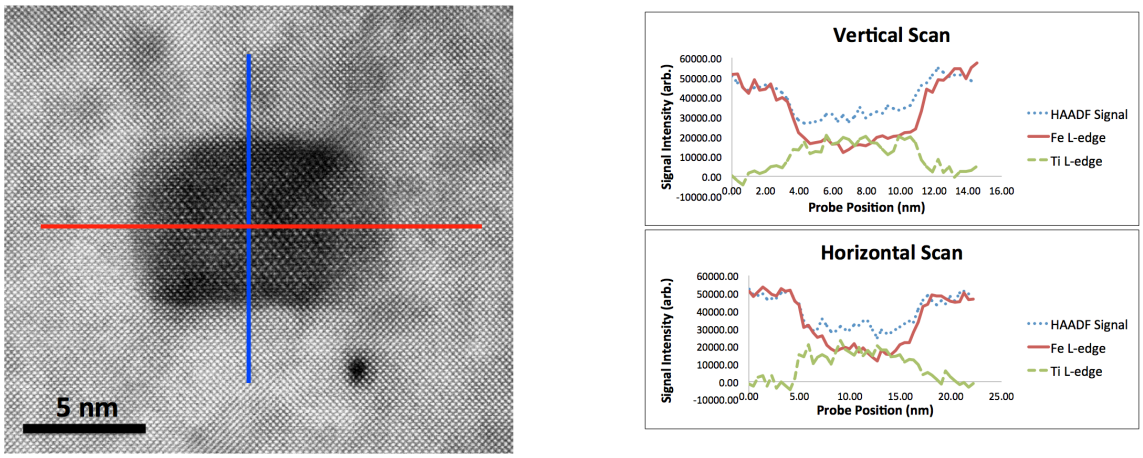

Fig 3. HAADF-STEM image of particle in Figure 2 with corresponding EELS line scans in horizontal and vertical directions for 0.5nm probe.

FUTURE WORK

The results summarized here are just a small part of the research plan for using the TEAM 0.5 microscope. We have alrady colleted extenive data using a variety of TEM methods on a variety of MA957 conditions that remains to be analyzed. Future studies will include ofhter ODS alloys and variants, dual ion irradiated alloys and meso-macroscale interfaces between single crystal $Y_2Ti_2O_7$ and electron beam physical vapor depsited Fe films.

ACKNOWLEDGMENTS

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