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Characterization of Ni-base Superalloys on the Atomic Scale by Atom Probe Tomography and Spherical-Aberration Corrected Analytical Electron Microscopy Techniques

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Materials characterization on the atomic scale is essential in order to improve our understanding of conventional materials, or to design more advanced materials. In terms of the sensitivity and spatial resolution of analysis, atom-probe tomography (APT) is one of the best approaches, since individual atoms can be collected. Therefore, 3 dimensional images of the solute distribution at atomic resolution can be reconstructed. Various statistical analyses can then be performed at any location by extracting the specific information from 3D datasets. The University of Sydney has installed a recently developed IMAGO Local Electrode Atom Probe, which has 10-50x larger field of view and 100-1000x faster acquisition rate compared with conventional atom probe [1]. The limitations of the APT technique mainly lie in the difficulty of obtaining crystallographic information.

To obtain local crystallographic information, complementary techniques such as analytical electron microscopy (AEM) are still required. The analytical capabilities in AEM are weaker in comparison with APT. However, the spatial resolution of analysis via electron energy-loss spectrometry (EELS) and/or X-ray energy dispersive spectrometry (XEDS) has been significantly improved in spherical-aberration (Cs) corrected instruments. It is now possible to analyze materials with atomic-level spatial resolution routinely by EELS [2]. At Lehigh University, two types of Cs-corrected instruments are available: a 300 keV VG HB 603 AEM with a Nion corrector and an Oxford Si(Li) X-ray spectrometer and a JEM-2200FS AEM with a CEOS corrector. Furthermore, in combination with spectrum imaging and multivariate statistical analysis (MSA) techniques, analytical performance in the Cs-corrected instruments is almost comparable with that from APT [3]. Therefore, most information can systematically be gathered on the atomic scale using both advanced APT and Cs-corrected AEM techniques.

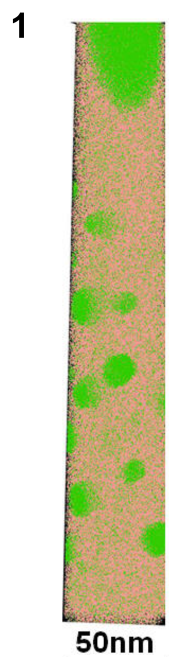
As an initial demonstration of the complementary materials characterization, a Ni-base superalloy X-750 was analyzed using both the APT at Sydney and the Cs-corrected AEMs at Lehigh. The superalloy X-750 is designed for a high-temperature use through precipitation hardening [4] and this particular sample was sequentially aged at two different temperatures to generate a bimodal distribution of $L1_2$-ordered γ' precipitates in a γ matrix. A precipitate-distribution map obtained by APT is shown in Fig. 1. There are two different sizes of the precipitates and a precipitate-free zone (FPZ) can also be seen around the coarse precipitate. Figure 2 shows two sets of quantitative composition maps of Fe and Ti: (a) around a coarse precipitate and (b) far from the coarse precipitates obtained by XEDS in the HB 603. From the maps, it can be seen that the coarse precipitate is >100 nm and smaller are ~10-20 nm in size, and there is a clear PFZ around the coarse γ'. The precipitate compositions determined by XEDS in the HB

603 compare closely with those from APT. However, the APT results show only trace amounts of Mn mainly distributed in the matrix, which is beyond the sensitivity of X-ray mapping with the MSA method in the HB 603.

In addition to the composition analysis, it has also been confirmed that the γ/γ' interface has a coherent cube-on-cube orientation relationship by atomic-resolution phase contrast imaging and high-angle annular dark-field imaging in the JEM-2200FS [5]. This information is challenging to obtain using the APT alone. Therefore, complementary characterization of materials on the atomic scale is best achieved by a combination of the APT and Cs-corrected AEM. More importantly, the APT system at Sydney and the JEM-2200FS AEM at Lehigh have remote-access capabilities and hence these atomic-scale characterizations can be carried out around the world through the internet, opening new possibilities for international scientific collaboration in the 21st century.

REFERENCES

[1] T.F. Kelly, et al., Microsc. Microanal. 10 (2004), 373.
[2] M. Valera, et al., Annu. Rev. Mat. Res. 35 (2005), 539.
[3] M. Watanabe, et al., J. Mater. Sci. (accepted), Microsc. Microanal. (submitted).
[4] M.K. Miller & M.G. Burke, Appl. Surf. Sci. 67 (1993), 292.
[5] D.B. Williams & M. Watanabe, Microsc. Microanal. (in this volume).
[6] The authors wish to acknowledge the support of the NSF through grants (DMR-0320906 and DMR-0304738) and of Bechtel Bettis, Inc.



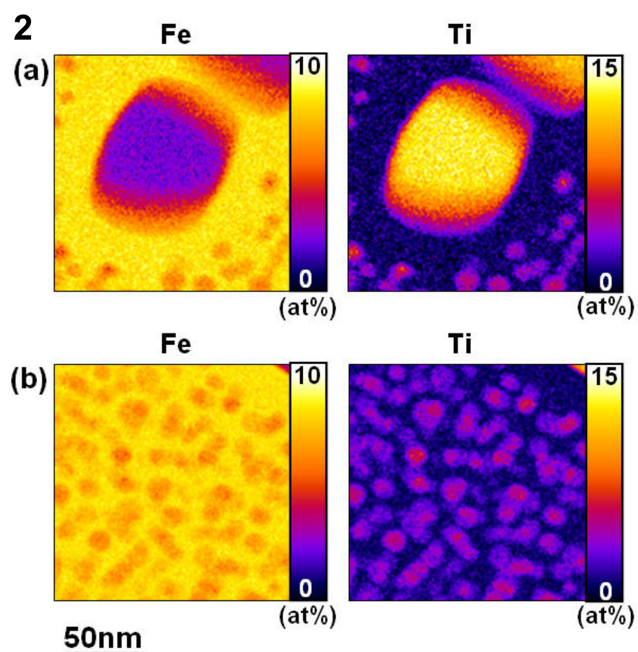


Fig. 1. A precipitate-distribution map obtained from the X-750 Ni-base superalloy using the APT system at Sydney. The analyzed volume is ~50x50x300 nm^3.
Fig. 2. Two sets of composition maps of Fe and Ti (a: around a coarse precipitate and b: far from coarse precipitates) obtained by the XEDS approach in the HB 603 at Lehigh.