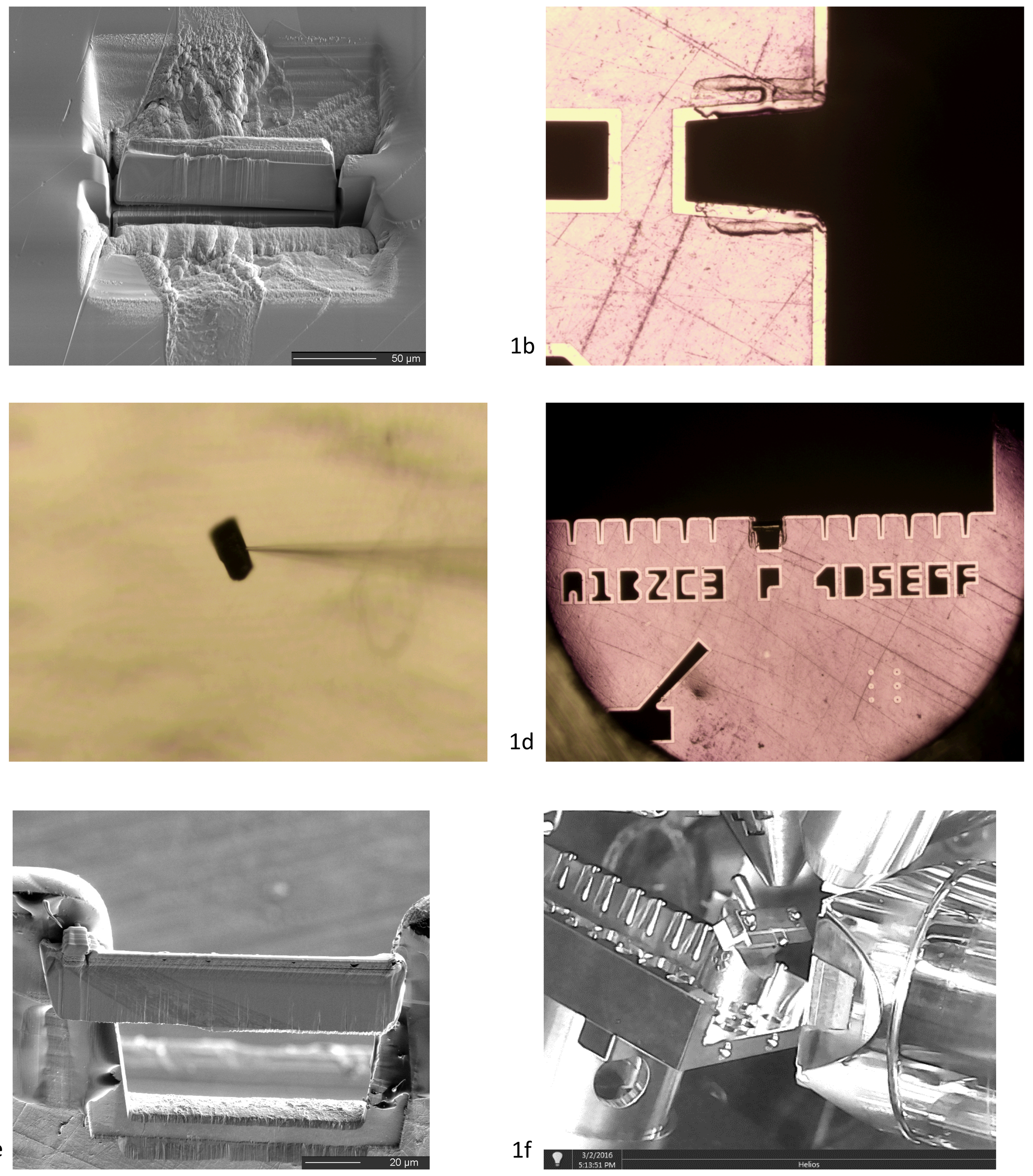
SAND2016-4485C

Site Specific EBSD, SEM, STEM, and XEDS, of a PFIB ex situ Lift Out Meteorite Sample

Lucille A. Giannuzzi[1*], Patricia O. Dickerson[2], Michael J. Rye[2], Paul G. Kotula[2] and Joseph R. Michael[2]
[1]EXpressLO LLC, Lehigh Acres, FL USA [2]Sandia National Laboratories, Albuquerque, NM USA

PFIB and EXLO Specimen Preparation



1a 1b 1c 1d 1e 1f

Introduction

The use of *ex situ* lift out (EXLO) for site specific specimens prepared by Ga^+ focused ion beam (FIB) milling and subsequent analysis by electron backscatter diffraction (EBSD) has been reported earlier [1,2]. Further, low energy FIB milling produces less damage and yields better EBSD results [3,4]. It was also shown that a Xe^+ plasma FIB using could be used to mill transmission electron microscopy specimens [5]. In this work, EXLO was used to manipulate a large plasma FIB milling specimen using special grids and methods [6]. The large lift out specimen was then analyzed by EBSD, scanning transmission electron microscope (STEM) and X-ray energy dispersive spectrometry (XEDS).

FIB Specimen Preparation and *ex situ* lift out

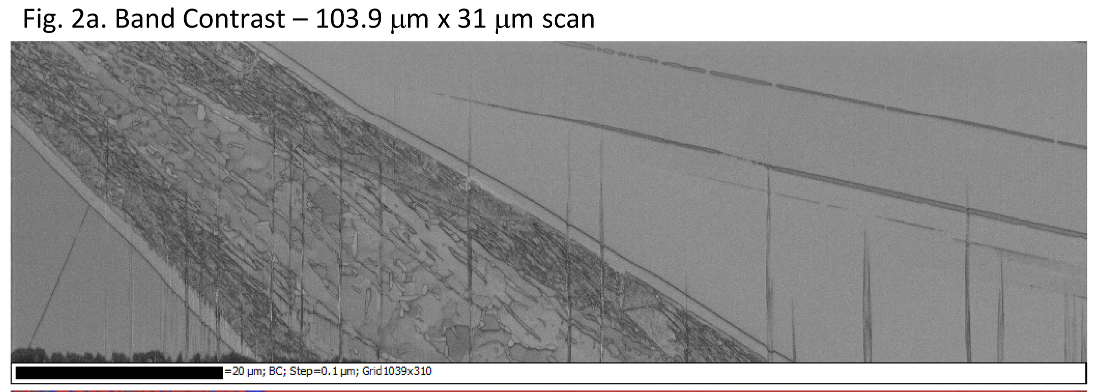
A plasma FIB column installed on an FEI DB 235 FIB/SEM was used to mill a site specific ex situ lift out (EXLO) specimen from a Gibeon meteorite (Fig. 1a). The EXLO specimen was milled using 30 keV Xe^+ to a dimension of ~ 150 µm x 40 µm and manipulated via EXLO to an EXpressLO lift out grid secured with M-Bond glue (Fig. 1a-d). The specimen was further FIB polished for EBSD analysis using 30 keV and finished with 20 keV Xe^+ (Fig. 1e). The specimen/grid mounted in a dual pin holder was moved to an FEI Helios 3G equipped with an Oxford EBSD detector and oriented to a 70° surface tilt (Fig. 1f). EBSD scans were obtained using an electron energy of 20 keV and 100 nm step size over an area of 103.9 x 31 µm.

EBSD Results

The EBSD results are shown in Figs. 2-5. Fig 2a and 2b show the band contrast and phase identification respectively. Note that the surface quality and EBSD results are quite good, except for the occasional curtaining artifact. Fig. 3 shows the orientation results for the FCC austenite phase, Fig. 4 shows the orientation results for the BCC ferrite phase, and Fig. 5 shows the orientation results for both phases. Note that there are specific orientation relationships between the austenite and the ferrite phases. Lift out allows for site specific EBSD without having to mechanically polish a region of interest to the edge of the sample.

EBSD

Fig. 2a. Band Contrast – 103.9 µm x 31 µm scan



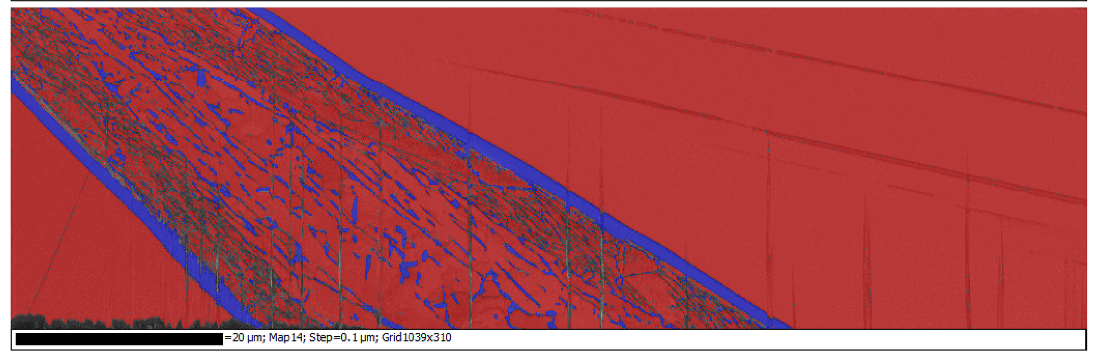


Fig. 2b. Red: BCC ferrite, Blue: FCC austenite

No cleaning applied to all of the following maps:

Fig. 3. FCC - austenite

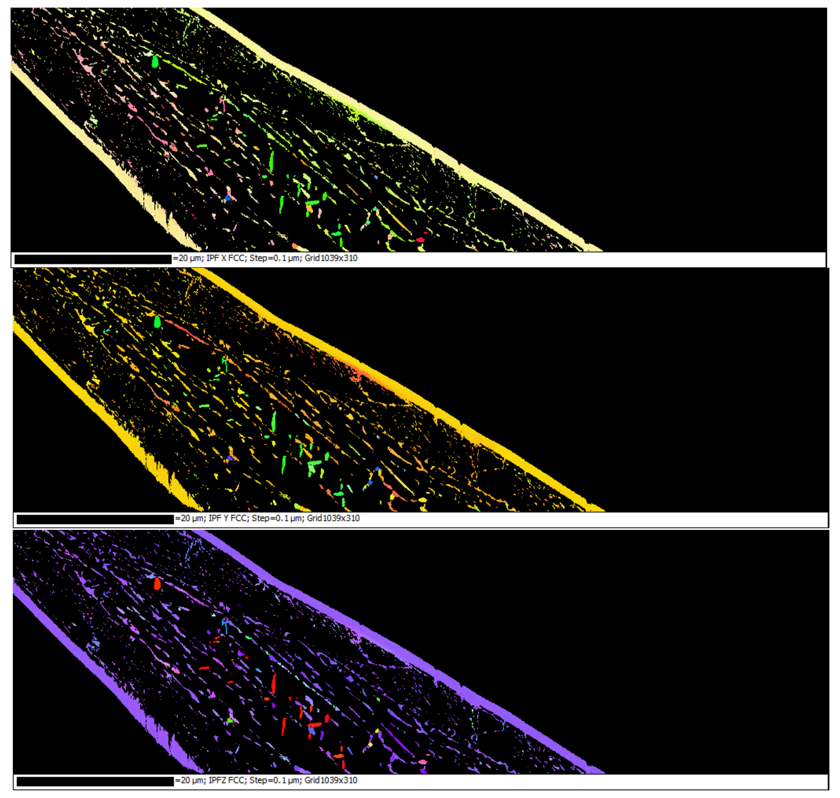


Fig. 4. BCC - ferrite

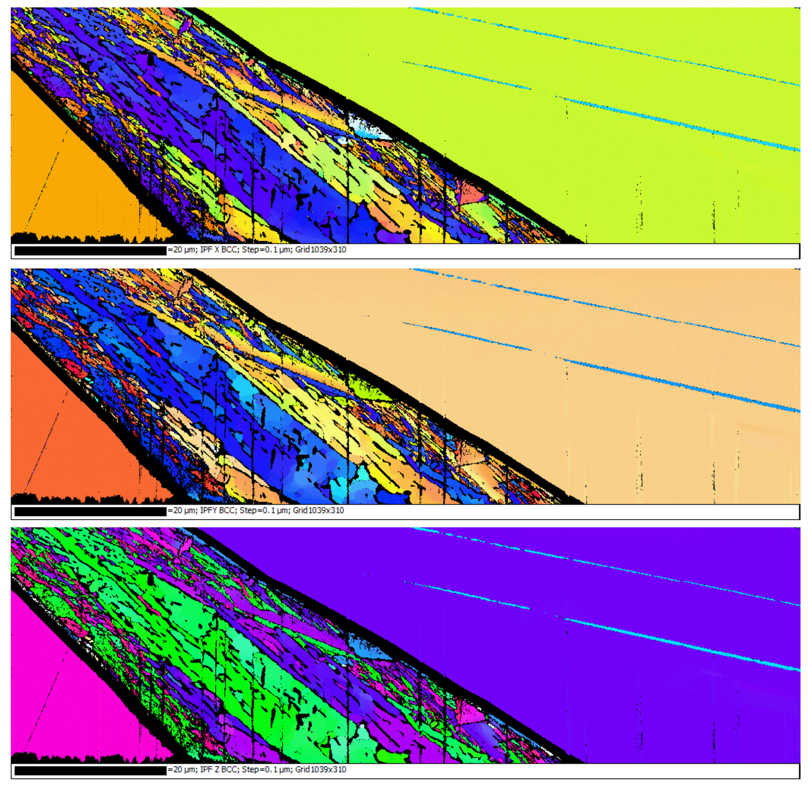
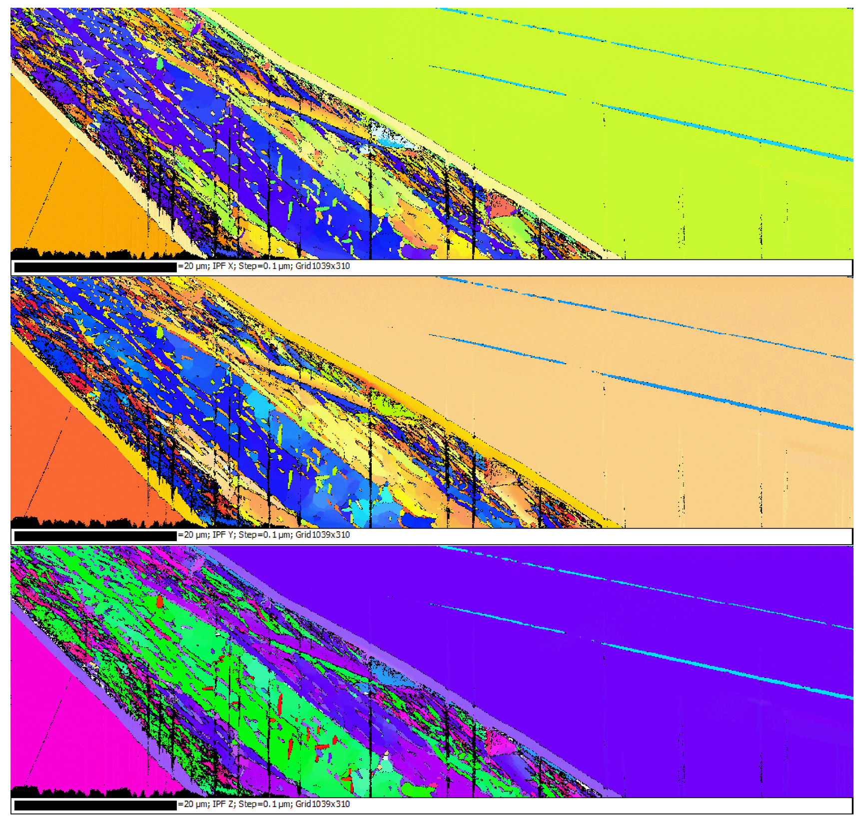


Fig. 5. FCC+BCC



STEM/XEDS

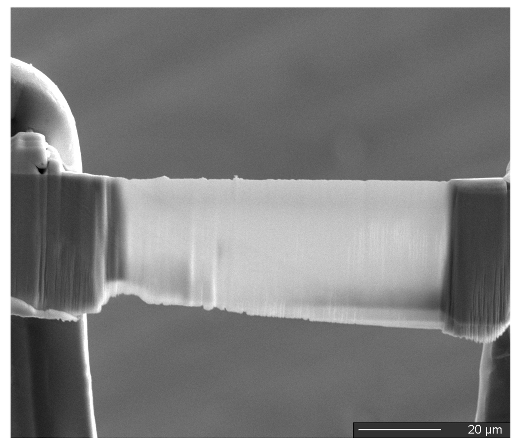


Fig. 6. (a) SEM image of meteorite after further PFIB milling to electron transparency.

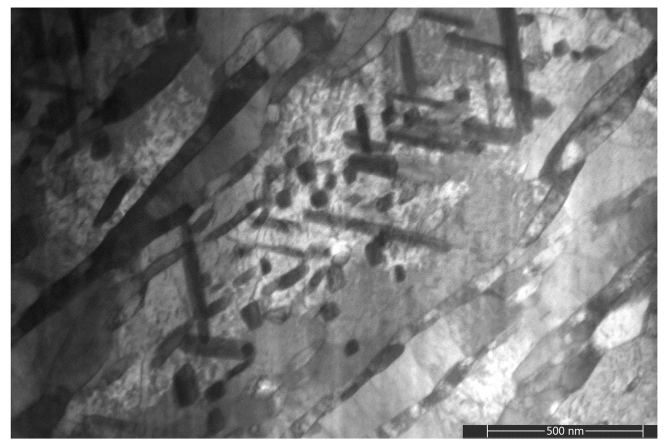


Fig. 6. (b) 30 keV bright field STEM image

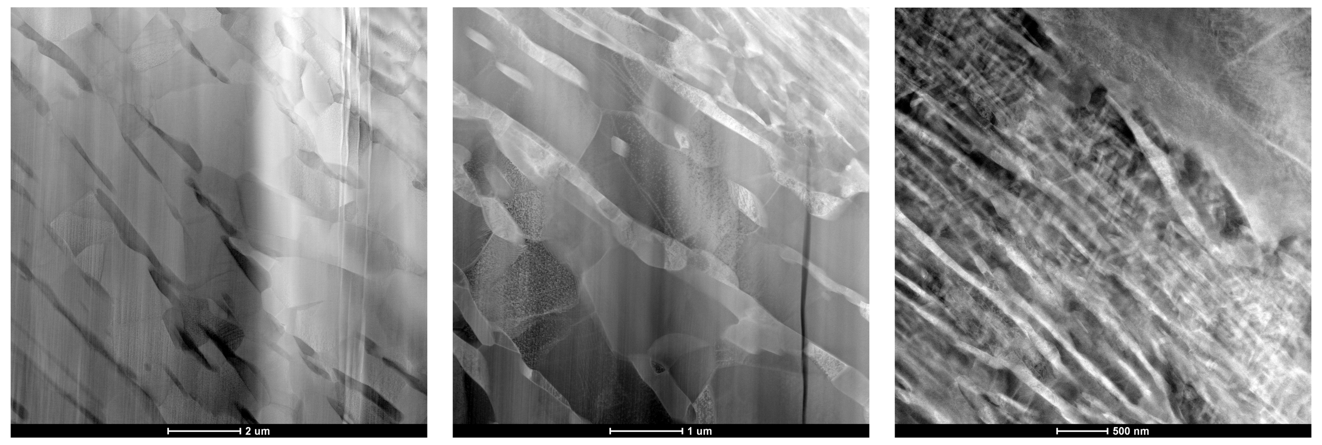


Fig. 7. 300 keV HAADF STEM images with successively higher magnification.

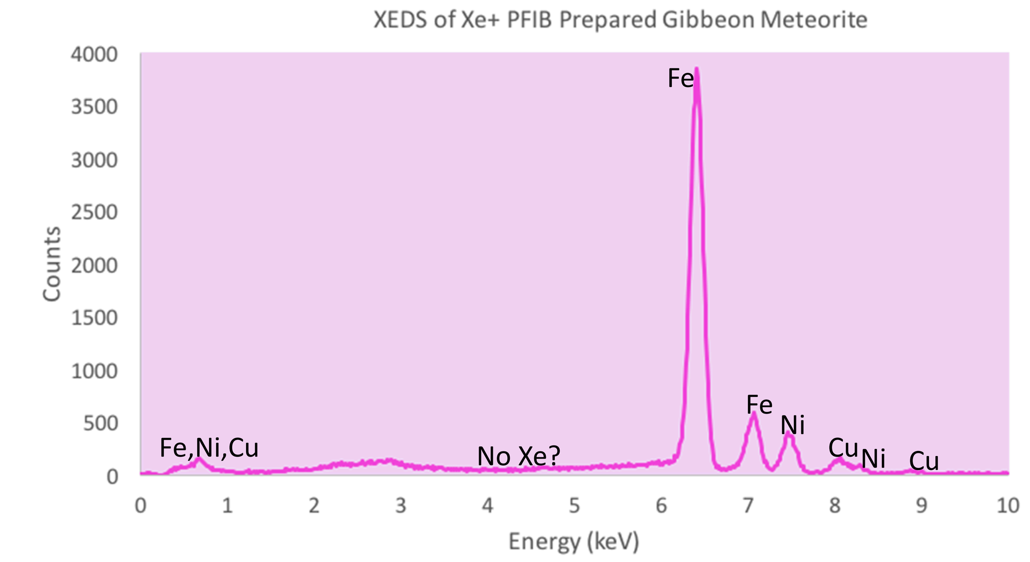


Fig. 8. XEDS of the meteorite showing the native Fe and Ni and the Cu grid. Note there is no detectable Xe from the PFIB milling in this spectrum.

STEM/XEDS Results

The specimen was further thinned by PFIB milling to electron transparency using 30 keV and then 5 keV Xe^+ (Fig. 6a). The specimen was a bit too thick for 20-30 keV transmission Kikuchi diffraction but was thin enough to obtain 30 keV STEM (Fig. 6b), 300 keV high angle annular dark field (HAADF) STEM images and XEDS as shown in Figs. 7 and 8. The Fe and Ni peaks are from the meteorite and the Cu peaks are fluorescence from the gird. Note, however, there are no detectable Xe peaks from the PFIB milling.

Summary and Conclusions

A large (> 100 µm) site specific specimen prepared by PFIB was manipulated via EXLO and analyzed using EBSD, STEM, and XEDS. EXLO eliminates the need for EBSD on a sample edge. EXLO to new grids allow for additional FIB milling after lift out and STEM analysis without the need for a carbon film support.

References

[1] S.V. Prasad, J.R. Michael, and T.R. Christenson, *Scripta Mater,* 48, (2003) 255–260.
[2] S.V. Prasad, et al., *J Microelectromech Syst,* 18, (2009) 695–704.
[3] T.L. Matteson et al., *J Electronic Materials,* 31(1), (2002) 33-39.
[4] L.A. Giannuzzi and J.R. Michael, *Microsc Microanal* 13(2), (2007) 926CD.
[5] L.A. Giannuzzi & N. Smith, *Microsc Microanal,* 17(2), (2011) 646–647.
[6] L.A. Giannuzzi et al., *Microsc & Microanal*, 21, (2015), 1034-1048.

Sandia National Laboratories

Sandia National Laboratories is a multi-program laboratory operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Company, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

TM