doi:10.1017/S1431927617001568
Microsc. Microanal. 23 (Suppl 1), 2017
© Microscopy Society of America 2017

Multivariate Statistical Analysis of a Multimodal Diffraction and X-ray Spectral Series Data Set

Paul G. Kotula[1] and Mark H. Van Benthem[1]

[1.] Sandia National Laboratories, PO Box 5800, MS 0886, Albuquerque, NM 87185-0886, USA

Multivariate statistical analysis (MSA) methods comprise a number of powerful techniques for reducing high-dimension data to a more manageable and interpretable lower dimension solution [1-2]. Moving beyond spectroscopic data large image and diffraction series can now be acquired [2,3]. Previously the analysis with MSA of a 183 Gb diffraction series was described [2] and in this work we will describe a deeper look in to the same data set combined with simultaneously acquired X-ray spectral data at each real-space point.

Position-resolved diffraction data were acquired on a FEI Company Titan G2 80-200 operated at 200kV and equipped with three-condenser lenses and a probe spherical-aberration corrector. Diffraction patterns were acquired on a PNDetector direct electron camera [2, 4]. Figure 1 shows the results of a 256 by 256 real-space pixel convergent-beam diffraction data acquired from the Arltunga meteorite where each pattern consisted of 264 by 264 reciprocal-space pixels with 20 replicate patterns at each point for a data set of 183 Gb. The data were analyzed with Sandia's Automated eXpert Spectral Image Analysis (AXSIA) MSA software [3] such that the unfolded reciprocal-space pixels were the pixels and the real-space pixels were the channels. The specific analysis performed had the result of making the real-space (pixel) domain simple [5].

The resulting scree plot of the log-eigenvalues versus sorted eigenvalue index (Fig. 1A) showed a clear break-point at 8 factors and the solution showed both crystallographic phase and orientation information (Figs. 1Band C) but the orientation differences are washed out for the most part. The same data were re-analyzed with a rank-45 corresponding to the second break point in Fig. 1A. The X-ray spectral image data were also analyzed with AXSIA resulting in two factors, Fe and Ni, with the quantified Ni component shown in Fig. 2B. A cover overlay of the uppermost Kamacite (low Ni phase, bcc) factors (Fig. 2C) and at corresponding 6 factors (Fig. 2D) describe just the uppermost Kamacite region. The angular tilt resolved between the cyan and magenta factors is 0.03º. MSA has efficiently reduced over 65,000 dimensions to 45 while still retaining such sensitivity to small orientation changes. In this case there's enough redundancy in the data to make MSA a worthwhile approach to reducing the dimensionality which ultimately would allow us to understand the chemistry, phases and orientations within the sample more quickly and with greater sensitivity [6].

References:

[1] Kotula, P.G., Keenan, M.R. & Michael, J.R. (2003). *Microsc Microanal* **9**, 1–17.
[2] P.G. Kotula *et al*, *Microsc. Microanal.* **22** Suppl 3 (2016), p.482-483.
[3] Brewer, L.N., Kotula, P.G., & Michael, J.R. (2008) *Ultramicroscopy* **108**, 567-578.
[4] Ryll, H., et al., (2013) *Microscopy and Microanalysis* **19** 1160-1161.
[5] Keenan, M.R. (2009) *Surf. Int. Anal.* **41**, 79–87.
[6] Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the US Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

https://doi.org/10.1017/S1431927617001568 Published online by Cambridge University Press

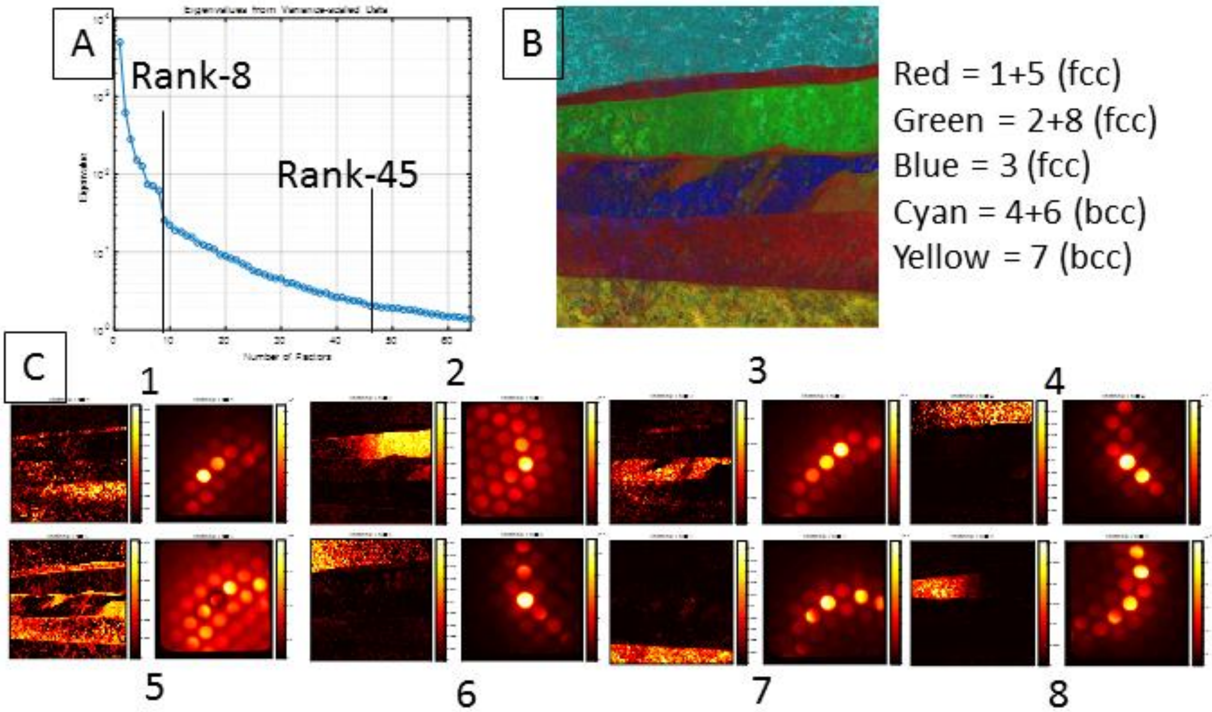

Figure 1. A. First 64 eigenvalues showing two break points. B. Color overlay of the component images from the 8-factor model of the data. C. Respective component images as real-space/reciprocal space image pairs.

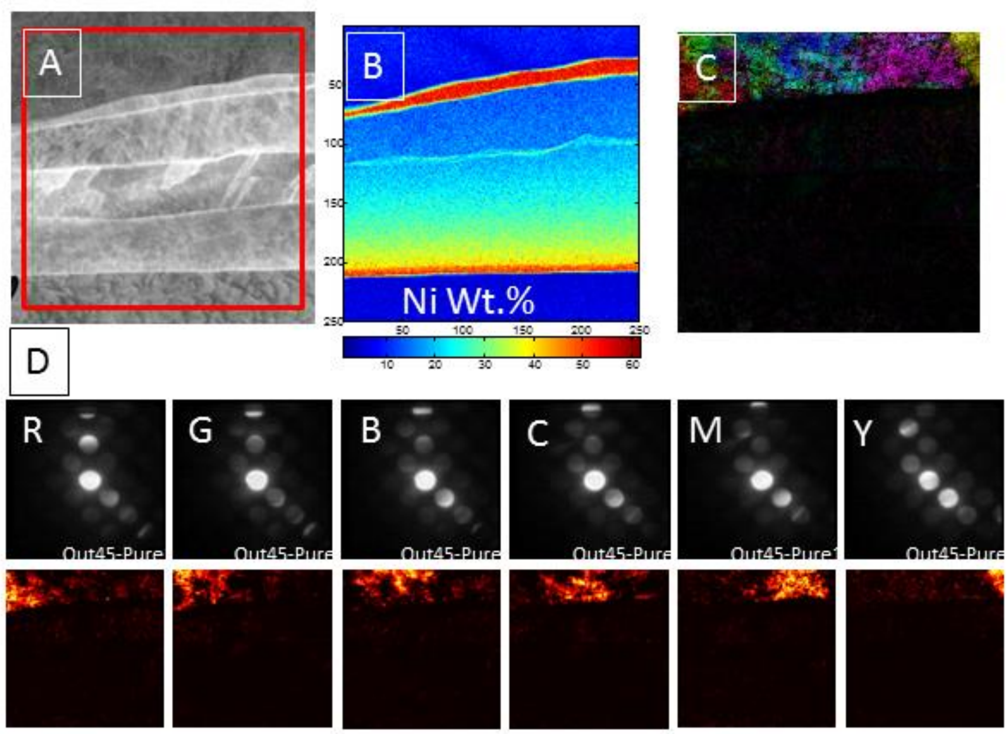

Figure 2. A. HAADF image of the analysis region. B. MSA analysis output (Ni factor) quantified to Ni weight percent. C. Color overlay with respective colors shown in the reciprocal space/real space image pairs in D of a set of factors corresponding to the topmost Kamacite from a 45-factor model of the data.

https://doi.org/10.1017/S1431927617001568 Published online by Cambridge University Press