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STEM EDS/EELS for Phase Analysis of Deep-Mantle Rock Assemblages Supported by Machine Learning

Hui Chen[1*], Farhang Nabiei[1], James Badro[2,3], Duncan T. L. Alexander[1] and Cécile Hébert[1]

[1.] Electron Spectrometry and Microscopy Laboratory (LSME), IPHYS, EPFL, Lausanne, Switzerland
[2.] Earth and Planetary Science Laboratory (EPSL), IPHYS, EPFL, Lausanne, Switzerland
[3.] Institute de Physique du Globe de Paris, Sorbonne Paris Cité, Paris, France
* Corresponding author: hui.chen@epfl.ch

Scanning transmission electron microscopy (STEM) is now a powerful technique employed in the study of minerals and rocks. In addition to providing high-resolution images, STEM generates a variety of additional signals that are often employed for analyzing geological samples. The two most prominent techniques among them are energy-dispersive X-ray spectroscopy (EDS) and electron energy-loss spectrometry (EELS). EDS is a relatively simple and robust technique to do chemical analysis. EELS is a more challenging technique but is in principle capable of not only measuring elemental composition, but also giving information about chemical bonding and oxidation state, band structure, etc. These two methods are both applicable for the quantitative chemical analysis of mineral phases which do not overlap across the thickness of the TEM sample. In the scenario where phases instead overlap in the projected volume, 2D EDS/EELS is insufficient to obtain accurately the composition of every single phase. Although in principle this problem can be tackled using electron tomography, this technique drastically complicates the data acquisition, and the minerals may experience severe sample degradation under the extensive beam exposure required to record the tomogram.

Here we instead look to address this problem via use of data processing algorithms developed by the computer science community. From the perspective of statistics, counts generated from a characteristic phase have a featured spatial distribution, which acts as a blind source to be unmixed via model-constrained "learning". Among many different statistical approaches, we adopt a non-negative matrix factorization (NMF) technique [1], because it applies the assumption of non-negative values in the spectra and cardinalities of chemical components, which are always positive in actual data.

In this paper, the starting material is a synthetic pyrolite glass doped with Nd, Sm, Hf, Lu, and U (0.3 wt.% for each). Four samples were made by compressing the pyrolite to the pressure range of 46 GPa to 88 GPa in diamond anvil cell (DAC). The samples were molten by double-sided laser heating and then slowly cooled down below the solidus temperature before quenching. Thin sections for STEM analysis were made by focused ion beam (FIB) lift-out technique from the recovered samples after decompression.

EDS maps, which are given in Figure 1, were obtained on a FEI Tecnai Osiris equipped with 4 SDD EDS detectors. Further electron microscopy data processing was done using Hyperspy [2], an open source Python package for multi-dimensional data analysis. In a first step, principal component analysis (PCA) based on singular value decomposition (SVD) has been applied to the EDS data. The explained variance scree plot in Figure 2 indicates three main features. Then, NMF was implemented on the PCA denoised data to perform a decomposition. Loadings (i.e. 2D image in navigation space) and factors (i.e. 1D spectrum in single space) of the main components are displayed in Figure 2. The corresponding quantification results are calculated and reported in the figure. The phase compositions obtained from

NMF segmentation are similar to the composition of bridgmanite, ferropericlase (with metallic iron mixed in), and Ca perovskite, respectively [3]. On the other hand, the algorithm failed to separate metallic iron from the ferropericlase. We are currently evaluating the ability of EELS to fulfill this differentiation by measuring the distribution of electronic states of iron, in order to achieve a more accurate phase segmentation.

References:

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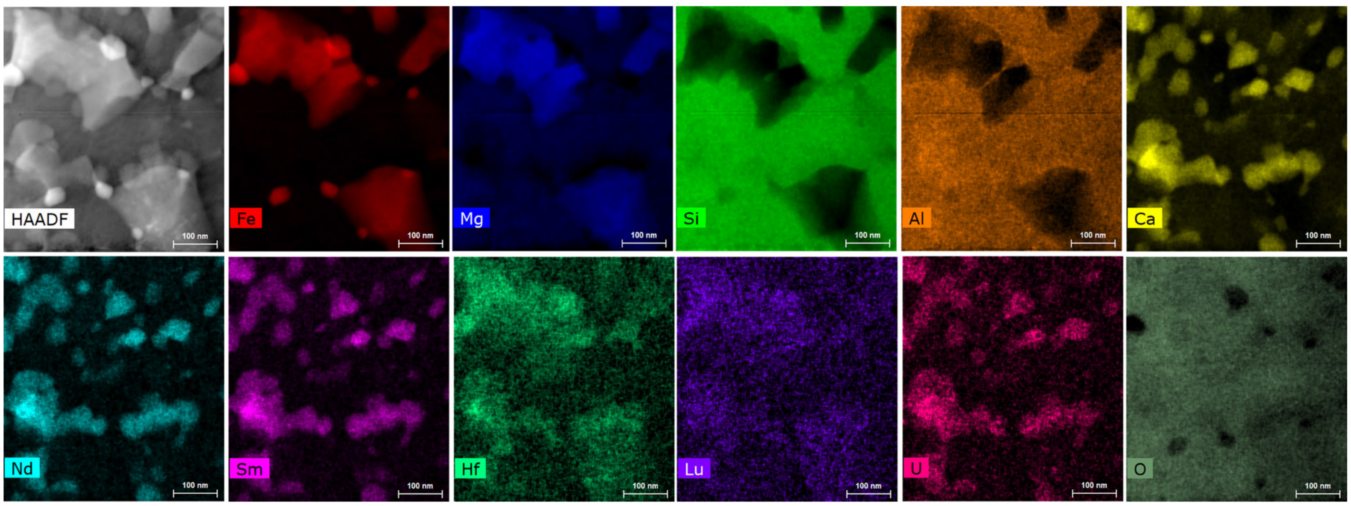

Figure 1. HAADF image and EDS raw elemental maps of the 46 GPa sample consisting of bridgmanite, ferropericlase, Ca perovskite minerals and metallic iron particles.

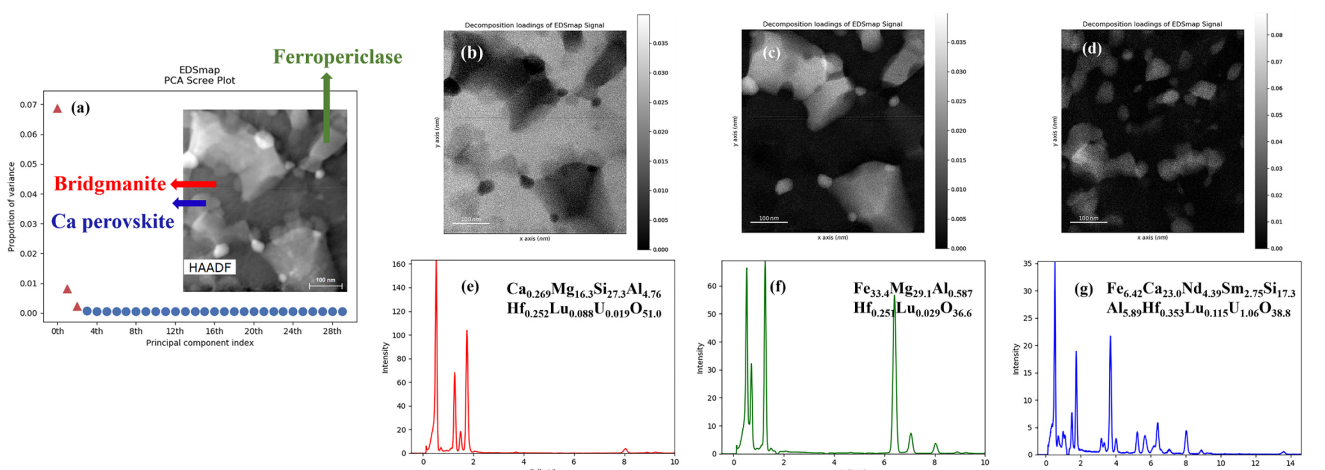

Figure 2. (a) PCA scree plot of STEM-EDS data, (b)-(d) representive component loadings and (e)-(g) component factors of NMF decomposition. Insets give the corresponding quantified composition for each component.

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