COMPOSITIONAL AND MICROSTRUCTURAL EVOLUTION OF OLIVINE UNDER MULTIPLE-CYCLE PULSED LASER IRRADIATION AS REVEALED BY FIB/FIELD-EMISSION TEM.

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**Introduction:** The use of pulsed laser irradiation to simulate the short duration, high-energy conditions characteristic of micrometeorite impacts is now an established approach in experimental space weathering studies [1, 2]. The laser generates both melt and vapor deposits that contain nanophase metallic Fe (npFe\textsuperscript{0}) grains with size distributions and optical properties similar to those in natural impact-generated melt and vapor deposits [3]. There remains uncertainty, however, about how well lasers simulate the mechanical work and internal (thermal) energy partitioning that occurs in actual impacts [4]. We are currently engaged in making a direct comparison between the products of laser irradiation and experimental/natural hypervelocity impacts. An initial step reported here is to use analytical SEM and TEM to attain a better understanding of how the microstructure and composition of laser deposits evolve over multiple cycles of pulsed laser irradiation.

**Experimental Methods:** We irradiated pressed-powder pellets and a single crystal of San Carlos olivine (Fo\textsubscript{90}) with sequential rastered pulses of a GAM ArF excimer laser [5]. SEM and TEM results are reported here for pressed-powder (PP) pellets irradiated with a single rastered scan (1-scan PP) and 99 rastered scans (99-scan PP), and a single crystal (SC) irradiated with 6 scans (6-scan SC). The surfaces of the samples were characterized by SEM imaging after irradiation and areas were selected for FIB cross sectioning for TEM study using an FEI Quanta dual-beam electron/focused ion beam instrument. FIB sections were characterized using a JEOL2500SE analytical field-emission scanning transmission electron microscope (FE-STEM) optimized for quantitative element mapping at <10 nm spatial resolutions.

**Results:** In the SEM, the 99-scan PP sample shows a complex, inhomogeneous, distribution of laser-generated material, largely concentrated in narrow gaps and larger depressions between grains. Local concentrations of npFe\textsuperscript{0} spherules 0.1 to 1 \(\mu\)m in size occur within these deposits in SEM back-scatter images. Fig. 1 shows bright-field STEM (BF-STEM) images of a FIB cross-section of a one of these deposits that continuously covers the top and sloping side of an olivine grain. The deposit has 3 microstructurally distinct sub-layers composed of silicate glass with varying modal fractions and size distributions of npFe\textsuperscript{0} spherules, along with nanocrystalline silicate material (Fig. 1). A relatively thin (50-300 nm) topmost surface layer has a high-concentration of npFe\textsuperscript{0} spherules 5-20 nm in size (Fig. 1a). Element mapping shows the layer to be enriched in Fe by a factor of 2.5 relative to the olivine substrate, with Mg and Si depleted by 20% and 10%, respectively. This is compositionally complementary to the underlying, middle layer of the deposit that is depleted in Fe, enriched in Mg and has a much lower npFe\textsuperscript{0} concentration. A third layer of nanocrystalline olivine occurs at the substrate interface.

On the FE-STEM scale, the layers of laser-generated material on the 1-scan PP and 6-scan SC samples are microstructurally and compositionally less complex than the 99-scan PP sample. The layers continuously cover their respective olivine grains and have uniform widths (125-150 \(\mu\)m) across their respective FIB sections. The layer on the 1-scan PP sample is slightly wider on average (~150 \(\mu\)m) than on the 6-scan SC (~125 \(\mu\)m, Fig. 2). Conventional bright-field and Z-contrast STEM imaging shows the layers in both samples to be similarly composed of <10 npFe\textsuperscript{0} spherules in a silicate glass matrix (Figs. 2, 3). The layers in both samples exhibit a distinctive “nanostratigraphy” in their

![Image](https://ntrs.nasa.gov/search.jsp?R=20160003147)
modal npFe⁰ concentrations and grain size (Figs. 2, 3). This npFe⁰ microstructural nanostratigraphy is not mirrored in strong variations in major element concentrations determined from compositional profiles extracted from X-ray EDX spectrum images (Fig. 3). The bulk compositions integrated over the layer volumes do, however, differ from the underlying olivine in showing significant Si-depletion relative to Mg+Fe. FE-STEM electron energy loss (EELS) spectroscopy analyses are currently ongoing to correlate both bulk and nanoscale variations in Fe oxidation state within the layers.

**Discussion:** We interpret the nanoscale microstructural and compositional variations in the 1-scan PP and 6-pulse SC samples to reflect very short time scale sequential deposition and mixing of melt and vapor-generated material as the pulsed laser beam tracks over the sample. The vapor component in particular, along with small scale thermal variations, likely account for the nanostratigraphic variations in npFe⁰ concentration and grain size. For the 99-scan PP, the more complex microstructural/compositional layering reflects longer timescale deposition on an irregular surface that causes build up of thicker, more inhomogeneous and complex deposits. FE-STEM results suggest the topmost layer in the 99-scan PP is a vapor deposit, underlain by a thick-

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