Experimental Studies on Heated and Unheated He-Irradiated Olivine Grains at Moderate He-ion Fluences: Analogues to Radiation Damage in IDPs. D. J. Joswiak¹, D. E. Brownlee¹, D. J. Schlutter² and R. O. Pepin², ¹Dept. of Astronomy, University of Washington, Seattle, WA 98195, ²Dept. of Physics and Astronomy, University of Minnesota, Minneapolis, MN 55455. joswiak@astro.washington.edu

Introduction: Irradiation in the harsh environment of space from cosmic ray and solar wind bombardment can be a significant process inducing both chemical and textural changes in extraterrestrial materials. In lunar soils for example, solar wind implantation in olivine and ilmenite grains produces amorphous rims containing nanophase Fe-metal inclusions [1]. In IDPs with their high surface to volume ratios and naturally high exposure to radiation, both large and small scale irradiation textures are often seen. Perhaps the ultimate end-texture from irradiation is exhibited by GEMS (Glass with Embedded Metal and Sulfides) which are completely altered due to the complete erosion of the original mineralogy by incident radiation over long periods of time [2]. Small-scale minor features that have commonly been seen in IDPs include amorphous rims on grains, cosmic ray tracks in minerals and void or bubble structures near edges of grains.

Depending on a number of variables – time of exposure to solar wind or cosmic rays, orientation and position of grains relative to other grains in an aggregate IDP, astrophysical location and so on – minerals and other compounds can be affected very differently by incident radiation.

We subjected Fo90 olivine crystals to moderate He-ion fluences in the laboratory and have investigated the irradiation effects in subsequently heated and unheated grains. Investigation of heated samples is important because of the heating that all IDPs experience during atmospheric entry.

Analytical Techniques: Hand-picked olivine grains containing 0.28 wt% Ni were crushed and sieved into 10um and 50um size fractions. The grains were exposed to He ion fluxes at the University of Minnesota with a 5 mm diameter 4kV He ion beam for 48 hours while being vibrationally tumbled which ensured that the olivine grains were evenly irradiated. Approximate He-ion fluences for the 10um and 50um olivine size fractions were 1.5x10¹⁵ ions/cm² and 4.0x10¹⁴ions/cm², respectively.

To simulate atmospheric entry heating, some of the irradiated olivine grains were then placed in tungsten foils at the University of Washington and pulse-heated for 5–10s to 850 °C under vacuum (pressure=4x10⁻⁸ mbars). Heated and unheated irradiated olivine grains were micromotred and placed on standard Cu or Au TEM grids with ~10 nm thick C-films or were crushed and placed on holey carbon Cu Quantifoil grids which contain ~1.2 nm diameter holes evenly distributed in a ~12 nm thick C-film. The samples were examined with a 200 kV Tecnai F20 FEG STEM equipped with a Gatan imaging filter and EDAX EDX spectrometer.

Unheated Olivine Grains: The observable effects of He-irradiated rims on unheated olivine were considerably smaller and more subtle compared to heated grains. Amorphous rims containing < 5 nm voids were seen by TEM imaging (Figure 1) and occasionally dark patchy Fe-rich grains < 5nm in size were also observed which appeared to be noncrystalline based on lack of lattice fringes from high resolution imaging. More thorough analyses of these features are needed help to confirm these observations.

Heated Olivine Grains: Rim textures were much more dramatic in heated olivine grains compared to unheated grains. Bright-field and dark-field TEM imaging indicate that irradiated rims are largely amorphous with some crystalline regions, perhaps due to annealing from heating.

Conspicuous nanophase inclusions < 30 nm were observed within the amorphous rims (Figure 2) and were determined to be [\(\overline{1}\)]-FeNi metal from high resolution imaging combined with EDX spot analyses. The [\(\overline{1}\)]-FeNi metal inclusions were sometimes enclosed by thin Fe-rich rims; we presume that the rims observed on these inclusions are related to the growth and enlargement of the nanophase [\(\overline{1}\)]-FeNi metal inclusions during heating.

Prominent round to irregular < 100 nm voids were observed on the amorphous silicate irradiated rims. Evident size sorting with larger voids occurring near the outer rim edges is visible in Figure 2. Some elongate oval-shaped voids, near the outer margins of the rims, showed ‘necking’ in the centers of their long axes probably as a result of coalescence of smaller voids during void formation.

EDX spot analyses indicate that the rim compositions are Mg-rich silicate glass with significantly less Fe than the unaltered olivine. One EDX analysis of a amorphous rim was: O 71.1, Mg 12.9, Si 15.9, Fe 0.1, (normalized atom %). Lower O/Si and Mg/Si ratios were measured in the rim, compared to the unaltered olivine, indicating removal of O and Mg, presumably from sputtering during initial He bombardment.

Irradiated Rims in IDPs: In a recent report [3], we observed that stratospheric IDPs rich with implanted He contained numerous voids or bubbles along the rims of pyrrhotite grains which we attributed to the thermal migration of implanted He, perhaps during atmospheric entry heating. We have also observed voids in other phases in IDPs including olivine, pyroxene, hydrated minerals, glass and amorphous carbon.
In one pyroxene-rich IDP numerous voids and nanophase Fe-rich inclusions (probably magnetite) in the rims of Fe-bearing pyroxene were seen (Figure 3). The texture is very similar to that observed in our He-irradiated and heated olivine samples. Stepped He-release measurements [4], indicate that this IDP was heated to about 680 °C during atmospheric entry.

**Discussion and conclusions:** Recent work on He-irradiated ion-thinned olivine grains has shown that He ion fluences of \( \sim 10^{16} \) ions/cm\(^2\) or more are required to produce amorphous rims on olivine along with voids and nanophase metal [5] at room temperature. At fluences below \( \sim 10^{16} \) ions/cm\(^2\), the authors reported that little to no irradiation effects are observed in irradiated rims. Our unheated olivine grains were irradiated at He ion fluences just below the critical value of \( \sim 10^{16} \) ions/cm\(^2\) thus, it is not surprising that only minor irradiation textures were observed in the rims of our unheated grains.

In our heated samples, however, physical changes were very striking and included prominent voids, crystalline nanophase metal beads, rim amorphitization and removal of O and Mg atoms from the host olivine rim. This suggests that heating of olivine grains that have been subjected to radiation fluences less than \( \sim 10^{16} \) ions/cm\(^2\) will produce similar effects as olivine that has been irradiated at much higher fluences but not heated. In our unheated samples irradiated with only moderate He-ion fluences, a sufficient number of atoms and defects were not displaced by He implantation in the crystal lattice to form the amorphitized rim and produce voids and nanophase metal. At high temperatures, however, our olivine grains that were irradiated at moderate He-ion fluences show that atoms and defects diffuse through the crystal lattice toward the rims producing textures similar to those observed in unheated grains but with fluences \( > \sim 10^{16} \) ions/cm\(^2\).

Our data suggest that irradiation textures (amorphitized rims, voids and metal inclusions in grains) from He-ion fluences \(< \sim 10^{16} \) ions/cm\(^2\) will not be observable in IDPs unless strongly heated during atmospheric entry. Atmospheric pulse heating accentuates irradiation effects by enlarging He bubbles, producing Fe-rich inclusions and amorphitizing rims, a behavior opposite to other irradiation effects that are lost or minimized during atmospheric entry such erasure of solar flare tracks.