

# Nanoscale records of ancient shock deformation: Reidite ( $\text{ZrSiO}_4$ ) in sandstone at the Ordovician Rock Elm impact crater

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## ABSTRACT

The terrestrial record of meteorite impacts is difficult to decipher because unequivocal evidence of impact is increasingly destroyed with time by erosion, burial, and tectonics. Zircon survives these processes as a shocked mineral, and above 20 GPa transforms to reidite, a high-pressure  $\text{ZrSiO}_4$  polymorph diagnostic of impact. However, the utility of reidite has been limited by its occurrence; it has only been reported from three relatively young (<36 Ma) impact craters globally. Here we report a new occurrence of reidite in brecciated sandstone from the Ordovician Rock Elm impact crater in Wisconsin, United States. Electron backscatter diffraction mapping was used to identify reidite and microtwins within shocked zircons smaller than 50  $\mu\text{m}$  in diameter. Reidite occurs both as 200–500-nm-wide lamellar intergrowths and as nanoparticulate grains, and not only provides the first diagnostic evidence for ultrahigh-pressure shock metamorphism at Rock Elm, but is also the oldest reported occurrence of reidite. Considering its small size, and the ubiquitous presence of detrital zircon in siliciclastic rocks, reidite may be more common in the rock record than has been reported but has potentially gone undetected. The recognition that nanoscale reidite can be preserved over deep time within zircon in shock-metamorphosed sandstone presents new opportunities for investigating Earth's impact record, as it could potentially preserve nanoscopic evidence of impact events much older than the one that formed Rock Elm. Given that shocked zircons have been shown to survive sedimentary cycling, the identification of reidite within zircons in siliciclastic rocks could facilitate investigating the impact chronology over much of the geological time scale, as the oldest terrestrial minerals known are detrital zircons.

## INTRODUCTION

Reidite is a tetragonal  $\text{ZrSiO}_4$  polymorph with scheelite structure (space group  $I4_1/a$ ) that was first reported from high-pressure experiments (Reid and Ringwood, 1969). A range of experimentally determined pressures for zircon-reidite equilibrium at ambient temperature have since been reported (e.g., Kusaba et al., 1985; Knittle and Williams, 1993). At high temperatures, reidite is unstable; it reverts to zircon above 1200 °C (Kusaba et al., 1985). Naturally occurring reidite forms when zircon is shock metamorphosed (Glass et al., 2002), and has only been reported from three impact structures where it occurs in ejecta and melt-bearing breccia. Reidite was first described in distal ejecta from the 35.5 Ma Chesapeake Bay impact structure in northeastern USA (Glass and Liu, 2001; Wittmann et al., 2009). It has since been found at the 15.1 Ma Ries crater in Germany (Gucsik et al., 2004; Wittmann et al., 2006) and at the 0.05 Ma Xiuyan crater in China (Chen et al., 2013). Of significance to this study is that, in crater environments, reidite has been found only in crystalline clasts incorporated in melt-bearing impact breccia; it has not been reported in other target lithologies. Here we report an occurrence of sub-micrometer-size reidite in sandstone breccia from Rock Elm, a deeply eroded, Middle Ordovician impact structure in Wisconsin, United States. These results are the first documented occurrence of reidite in a

sedimentary target rock, and constitute the oldest known reidite in the geological record.

## GEOLOGICAL BACKGROUND

Rock Elm is a 6.5-km-diameter impact crater in Paleozoic target rocks in western Wisconsin (Fig. 1A). The complex impact crater is formed in Upper Cambrian and Lower Ordovician sedimentary rocks, with an impact age of 470–450 Ma constrained by unconformably overlying Middle Ordovician basin-fill sediments (Cordua, 1985; French et al., 2004). The central uplift consists of an ~1-km-diameter circular area of poorly exposed isolated blocks of bedded sandstone and conglomerate interpreted to be the Mount Simon Formation, an Upper Cambrian cratonic sequence that is dominantly mature quartz arenite (Lovell and Bowen, 2013). Breccia from the central uplift has been described (Cordua, 1985; French et al., 2004), however no in situ outcrops have been reported. The breccia is comprised of lithic clasts of variable size and composition within a matrix of sand-sized particles. French et al. (2004) reported shocked quartz grains with planar fractures that indicate pressures of 5–10 GPa, confirming an impact origin for the Rock Elm breccia.

## SAMPLE AND METHODS

Breccia sample 13RE07 was collected in 2013 as an ~25 cm loose block from a low mound

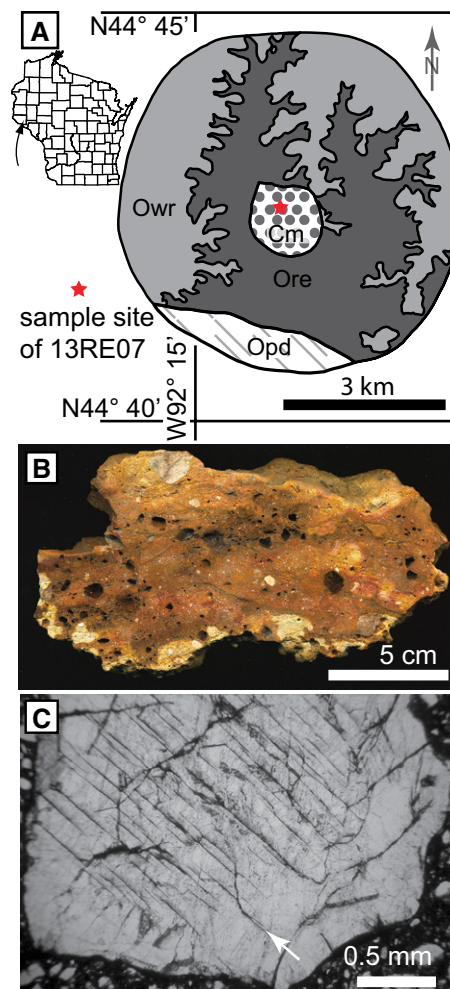


**Figure 1.** A: Simplified geologic map (after Evans et al., 2007) showing sampling location for Rock Elm breccia sample 13RE07 (Wisconsin, United States). Cm—Cambrian Mount Simon Formation sandstone. Middle Ordovician units, from oldest to youngest, are Opd (Prairie du Chien Group dolomite), Owr (Washington Road sandstone), and Ore (Rock Elm shale). B: Cut rock slab of breccia sample 13RE07. C: Quartz grain with planar fractures, sample 13RE07 (after Arana and Cavosie, 2014).

(10 cm high) of soil and rock fragments in the central uplift (GPS coordinates: 44°43.027'N; 92°13.822'W). The breccia contains deformed, <0.5 cm mixed sedimentary lithic clasts, shocked quartz, and minor alkali feldspar sand grains in an opaque reddish matrix, with voids ranging from sand-sized porosity to 3 cm vugs

(Fig. 1B). Shocked quartz grains contain one to four orientations of parallel planar fractures (Fig. 1C), similar to those described by French et al. (2004). A total of 135 zircons larger than 10  $\mu\text{m}$  in diameter was documented in one polished petrographic thin section using scanning electron microscopy; most preserve oscillatory growth zoning, occur in the matrix, and are interpreted as detrital in origin. Electron backscattered diffraction (EBSD) mapping revealed the presence of reidite in two zircons within a  $3.5 \times 0.5$  mm clast, and a zircon with microtwins nearby in the matrix. For details of the methods and analytical conditions, see the GSA Data Repository<sup>1</sup>.

## RESULTS

### Shock-Twinned Zircon

Zircon 36 (Fig. 2A) is a  $10 \times 15$   $\mu\text{m}$  anhedral fragment. High-spatial resolution EBSD mapping using 50 nm step size identified five parallel sub-micrometer-wide microtwin lamellae on the right side of the grain (green in Fig. 2A). The microtwins are related to the host zircon by  $65^\circ$  misorientation about  $\langle 110 \rangle$ , which results in the sharing of a  $\{112\}$  orientation between the two domains (see the Data Repository). Despite occurring in an area of high fracture density, the microtwins are not visible in atomic number contrast imaging (see the Data Repository).

### Reidite-Bearing Zircons

Zircon 9 is a  $30 \times 40$   $\mu\text{m}$  subhedral grain with irregular form. The upper margin is cusped and contains narrow tubular embayments, the right side of the grain preserves prism and pyramid faces, and the left side appears to have partially disaggregated (Figs. 2B and 3A). The host grain preserves crystallographic misorientation of up to  $25^\circ$  from the mean orientation, principally as a combination of sub-planar deformation bands and isolated fragments (Fig. 3C). The dispersion of crystallographic poles for the host grain has a complex pattern, but is predominantly around  $\langle 001 \rangle$  (Fig. 3E). The misorientation of isolated blocks likely results from rigid rotation due to fracturing, whereas deformation bands are low-angle boundaries formed by the stacking of dislocations. The  $\langle 001 \rangle$ -parallel misorientation axis is consistent with dislocation slip by  $\{100\}\langle 010 \rangle$  (Leroux et al., 1999; Timms et al., 2012).

Two sets of parallel sub-micrometer lamellae on the right side of the grain are brighter in atomic number contrast imaging than the host zircon, yield elemental spectra (by energy dispersive spectroscopy) consistent with  $\text{ZrSiO}_4$ , and produce EBSD patterns that index as reidite

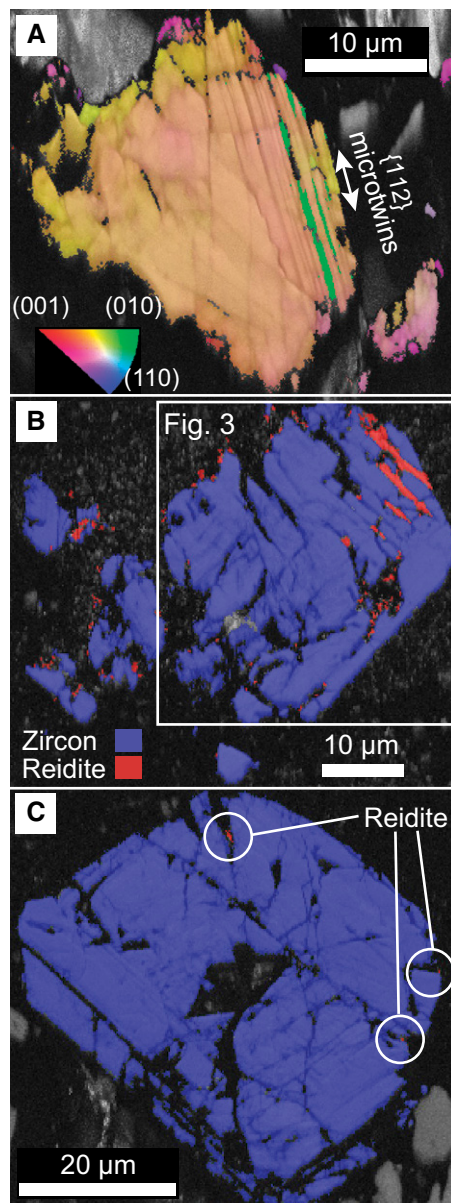


**Figure 2. Shocked zircons in Rock Elm breccia sample 13RE07 (Wisconsin, United States).** A: Map of zircon 36, showing crystallographic orientation of host zircon and microtwin lamellae (with an inverse pole figure color scheme; microtwin lamellae are green). B: Phase map of zircon 9. C: Phase map of zircon 10. Data for A are superimposed on a grayscale map of electron backscattered diffraction pattern quality (band contrast).

(Figs. 2B and 3). The lamellae are non-luminescent in cathodoluminescence, and cross-cut growth zoning in the host zircon (see the Data Repository). The lamellar reidite occurs in two distinct planes (1 and 2 in Fig. 3D) and shows systematic relationships with the host zircon. Reidite crystallographic poles show tight clusters, such that each lamellae set shares its  $\{001\}$  with a different  $\{110\}$  in the host zircon, and a  $\{110\}$  for each reidite set aligns with  $\{001\}$  of the host zircon (Fig. 3E).

In addition to lamellae, a nanoparticulate form of reidite is present lining the outer margin of the grain and within irregular, cusped voids within the host zircon (Figs. 2B and 3D). These grains were calculated to have a mean diameter of  $192 \text{ nm} (\pm 115 \text{ nm, one standard deviation})$ . Crystallographic analysis shows a scattered orientation distribution, where the poles of  $\{001\}$  only broadly cluster around poles to  $\{110\}$  in the host zircon; a significant portion of the nano-reidite grains shows no systematic relationship to the zircon (Fig. 3E).

Zircon 10 is a  $40 \times 50$   $\mu\text{m}$  blocky subhedral zircon that contains reidite in three small domains (Fig. 2C). EBSD mapping of the largest domain ( $\sim 0.5 \times 2$   $\mu\text{m}$ ) shows that the reidite is in a single orientation, and has the same relationship to the host grain as in zircon 9 (see the Data Repository). The small size of the reidite occurrences precludes identification of habit, although an array of grains in uniform orientation is consistent with lamellar form.

## DISCUSSION

### Electron Backscatter Diffraction: A New Method to Identify Reidite

Reidite has previously been documented in shocked zircon using transmission electron microscopy (TEM) (Leroux et al., 1999), X-ray diffraction (Glass and Liu, 2001), and Raman spectroscopy (Wittmann et al., 2006; Chen et al., 2013). As shown here, EBSD is an additional method for identifying reidite, and can provide highly spatially resolved crystallographic data collected in situ from sub-micrometer domains in a thin section. The Rock Elm data confirm crystallographic relations between zircon and reidite determined previously in experimentally shocked grains measured by TEM, where  $\{100\}_{\text{Zrn}} \parallel \{112\}_{\text{Red}}$  and  $[001]_{\text{Zrn}} \parallel \langle 110 \rangle_{\text{Red}}$  (Zrn is zircon; Red is reidite) (Leroux et al., 1999).

### An Occurrence of Sub-Micrometer Granular Reidite

Sub-micrometer granular reidite has not previously been described in nature, however it has been documented in zircon experimentally shocked at 60 GPa (Leroux et al., 1999). The various occurrences of nanoscale granular reidite in zircon 9 (Fig. 3D) are remarkably similar, whether on exterior surfaces, lining cusped margins and interior voids, or lining nearby disaggregated zircon fragments. The presence of two distinct forms of reidite within a single shocked zircon implies that multiple transformation mechanisms operated simultaneously, or alternatively, that two separate shock events are recorded in this grain. Either scenario is constrained to have occurred over the millisecond time scales that high-pressure conditions were present during the passage of the shockwave (Melosh, 1989). The lamellar form of reidite

<sup>1</sup>GSA Data Repository item 2015116, analytical methods, EBSD conditions, and additional images, is available online at [www.geosociety.org/pubs/ft2015.htm](http://www.geosociety.org/pubs/ft2015.htm), or on request from [editing@geosociety.org](mailto:editing@geosociety.org) or Documents Secretary, GSA, P.O. Box 9140, Boulder, CO 80301, USA.

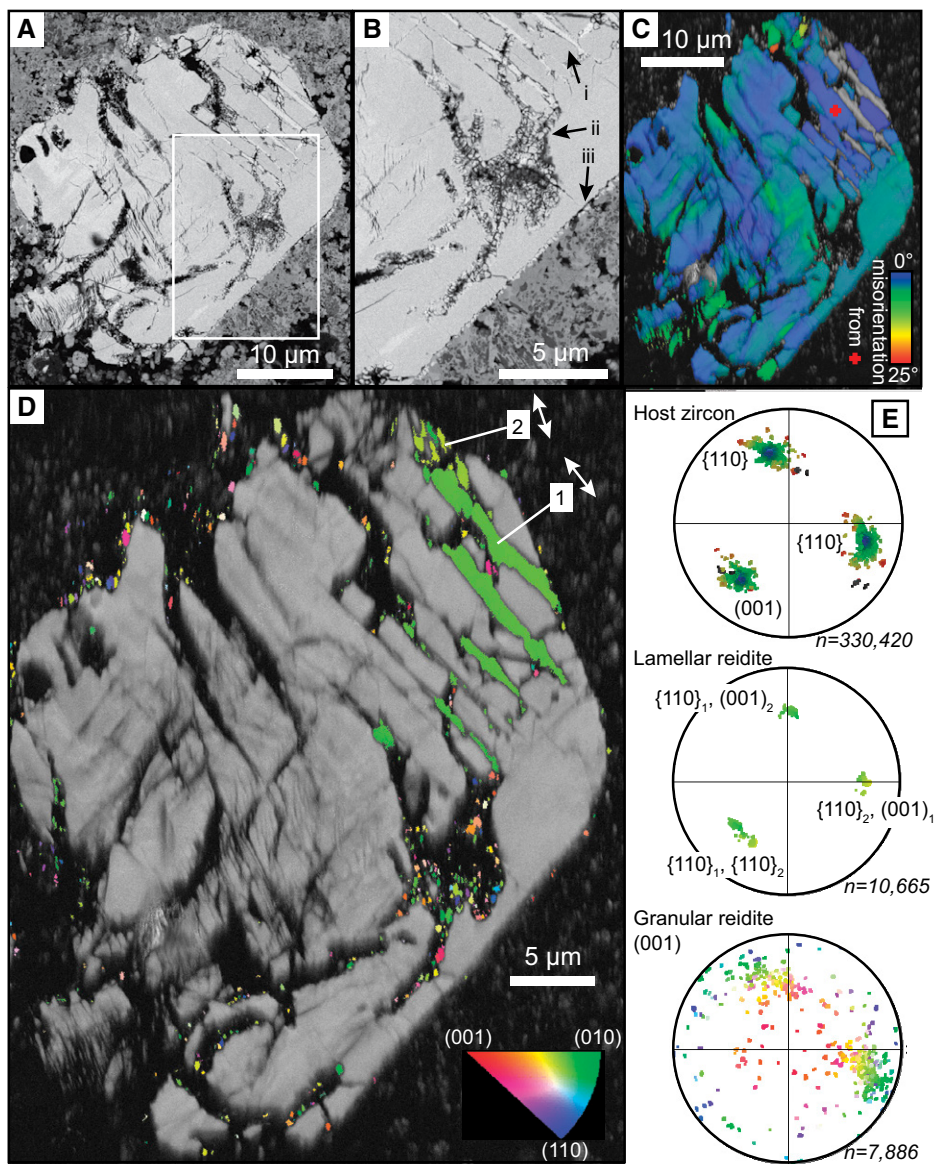


**Figure 3.** Images and crystallographic data of reidite-bearing zircon grain 9 (shown in Fig. 2B), Rock Elm breccia sample 13RE07. **A:** Atomic number contrast image. White box shows area shown in B. **B:** Detail of A showing (i) lamellar reidite, (ii) nano-granular reidite in voids, and (iii) nano-granular reidite decorating the external surface. **C:** Map showing crystallographic misorientation across the host zircon, relative to a reference point (red cross). **D:** Map showing crystallographic orientation of reidite grains and lamellae (with an inverse pole figure color scheme; zircon is gray in this image). **E:** Pole figures for the host zircon and reidite forms (all are lower hemisphere, equal area stereographic projections). Data for C and D are superimposed on grayscale maps of electron backscattered diffraction pattern quality (band contrast). Color scheme in E correlates to C (host zircon) and D (reidite).

and its topotactic relation with the host zircon are consistent with a shear-dominated phase transformation (Kusaba et al., 1986), whereas the formation mechanism of the nano-granular reidite appears most consistent with a reconstructive transformation mechanism that relies on nucleation and growth of new grains from the host zircon (Dutta and Mandal, 2012). Further exploring the variable response of zircon during dynamic high-pressure polymorphism is beyond the scope of this contribution, and is the focus of ongoing work.

### Shock History of the Rock Elm Breccia

An inherent difficulty in field investigations of Rock Elm breccia is the inability to observe contact relations with the Mount Simon Formation sandstone. Regardless, the presence of shocked quartz (French et al., 2004; Fig. 1C) and the shock-twinned zircon and reidite reported here provide incontrovertible evidence of high-pressure deformation that links breccia formation to the impact. The conditions under which deformation twins form in zircon have not been experimentally determined, however

shock-twinned zircon has been reported only from impact environments (Moser et al., 2011; Timms et al., 2012; Erickson et al., 2013a, 2013b; Thomson et al., 2014). In experiments, planar microstructures are pervasive at 20 GPa (Leroux et al., 1999), and so it follows that shock-twinned zircons record pressures of at least 20 GPa. Reidite represents the highest-pressure phase thus far identified at Rock Elm. While stable at pressures as low as 20 GPa in static experiments (Knittle and Williams, 1993; van Westrenen et al., 2004), in shock recovery experiments reidite forms between 30 and 40 GPa (Kusaba et al., 1985; Leroux et al., 1999). In crater environments, reidite has been reported only in melt-bearing impact breccias (Wittmann et al., 2006, 2009; Chen et al., 2013) that record shock stage II to III conditions, corresponding to pressures from 35 to 60 GPa (Stöffler and Grieve, 2007). We thus tentatively assign shock stage II conditions (35–45 GPa) to breccia sample 13RE07, as it is the minimum stage for which the conditions necessary to form reidite occur (Stöffler and Grieve, 2007).

### A New Locality and the Oldest Occurrence

Rock Elm represents the fourth impact structure where reidite has been documented, and with a minimum age of 450 Ma (French et al., 2004), has by far the oldest reidite known. Of the 185 confirmed impact structures in the Earth Impact Database (Spray and Elliott, 2015), 154 craters (83%) are younger than Rock Elm, and thus could feasibly preserve reidite if zircons were present in the target rocks. Reidite has not been reported from the largest known terrestrial impact structures, such as the Vredefort Dome (South Africa) and Sudbury (Canada), where large volumes of rock were exposed to higher shock pressures than at Rock Elm. One possible reason for the paucity of reidite at these sites may be the rapid reversion to zircon at temperatures above 1200 °C (Kusaba et al., 1985). Post-impact thermal conditions may inhibit widespread preservation of reidite in basement rocks at large impact structures. Reidite may thus be preferentially preserved in impact breccias typically found in the higher levels of impact structures, such as at Ries (14.5 Ma, 24 km diameter) and Chesapeake Bay (35.5 Ma, 85 km diameter), and also at relatively small and/or young impact craters, as exemplified by Xiuyan (0.05 Ma, 1.8 km diameter).

### Implications for Preservation of Reidite in Sediments and Rocks

Given the ubiquitous occurrence of detrital zircon in siliciclastic rocks, these findings highlight the preservation potential of reidite in shocked sandstone, a reservoir not generally recognized for preservation of ultrahigh-pressure shock records. For ancient impact structures that have since eroded, reidite may be preserved in

detrital shocked zircons that have been eroded and transported, and now reside in younger sedimentary rocks. Shocked zircons with diagnostic shock microstructures are ubiquitous in modern alluvium eroded from complex craters, such as the Vredefort Dome (Cavosie et al., 2010; Erickson et al., 2013b), Sudbury (Thomson et al., 2014), and Santa Fe (southwest United States) (Lugo and Cavosie, 2014) impact structures. With the recognition that Hadean detrital zircons with ages as old as 4.4 Ga are preserved (Valley et al., 2014), reidite may also provide a record of even earlier impact bombardment. Identification of reidite in ancient detrital zircons could provide ground-truth evidence from the terrestrial record of early impact events on Earth.

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