

The Sudbury impact layer in the Paleoproterozoic iron ranges of northern Michigan, USA

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ABSTRACT

A layer of breccia that contains fragments of impact ejecta has been found at 10 sites in the Paleoproterozoic iron ranges of northern Michigan, in the Lake Superior region of the United States. Radiometric age constraints from events predating and postdating deposition of the breccia are ca. 1875 Ma and 1830 Ma. The major bolide impact that occurred at 1850 Ma at Sudbury, Ontario, 500–700 km east of these sites, is the likely causative event. The Michigan sites described here, along with previously described sites in Minnesota and Ontario, define an extensive ejecta-bearing deposit throughout the Paleoproterozoic iron ranges of the Lake Superior region that we refer to as the Sudbury impact layer. The layer at the sites in Michigan exhibits a range of thicknesses, lithologic characters, and sedimentary settings. The diversity of rock types and internal stratigraphic details of the layer imply that several different processes of transport and deposition are represented, but the detailed investigations needed to document them are incomplete. Many of the sites had been described and interpreted previously as products of common terrestrial processes, but the presence of relict shock-induced planar deformation features in quartz indicates that the breccia layer is in fact the product of an extraterrestrial impact. At most localities, this layer also contains relict fragments of altered devitrified glass and/or accretionary lapilli. One immediate use of the impact layer is as an ultraprecise time line that ties together the well-known stratigraphic sequences of the various geographically separated iron ranges, the correlation of which has remained controversial for many decades. The Sudbury impact layer most commonly lies at a horizon that records a significant change in

the character of sediments across the region. The impact layer marks the end of a major period of banded iron formation deposition that was succeeded by deposition of fine clastic rocks, commonly black shales. The impact may have produced regional, if not global, changes in the environment that resulted in this widespread synchronous change in sedimentation style.

INTRODUCTION

The major impact event at Sudbury, Ontario, has been studied in great detail for the past 40 years since its existence was first proposed by Dietz (1964). The time of impact is precisely dated at 1850 ± 1 Ma (Krogh et al., 1984; Davis, 2008), the age of impact-generated melts. Breccias and related rocks of the Onaping Formation near Sudbury were first interpreted as impact-related rocks soon after Dietz's proposal (French, 1967, 1970; Peredery, 1972) and are now widely accepted to be, at least in part, crater-filling material resulting from direct fallback of ejecta, slumping of initial crater walls, and resurgence of ocean water into the new crater (for recent summaries of various aspects of this large body of work, see Deutsch et al., 1995; Riller, 2005; Spray et al., 2004; Grieve and Theriault, 2000; Naldrett, 2003; Grieve, 2006). There is growing evidence that igneous rocks, including the Sudbury igneous complex, are largely the impact-generated melt sheet (Faggart et al., 1985; Grieve, 1994; Keays and Lightfoot, 1999; Theriault et al., 2002; Naldrett, 2003; Mungall et al., 2004; Zieg and Marsh, 2005; Grieve, 2006). The crater produced by this impact event, now largely destroyed by erosion and strongly modified by younger tectonic events, has been variously estimated to have a diameter between 150 km and 260 km (see summaries in Abramov and Kring, 2004; Grieve et al., 2008).

An important missing component of the Sudbury story has been information on the character and distribution of ejecta deposited beyond the

crater margin. The first documentation of the existence of such ejecta was by Addison et al. (2005), who described occurrences of ejecta-bearing breccias in the Gunflint iron range in Ontario and Mesabi iron range in Minnesota. In the past two years, following the descriptions of Addison et al., we have located the breccia layer at 10 sites in the Upper Peninsula of Michigan; the layer contains ejecta particles, including quartz grains with relict shock-induced planar deformation features (PDFs), together with spheres and shards of altered devitrified glass and accretionary lapilli. We term this the "Sudbury impact layer" and interpret the breccias to have formed rapidly, in part within hours, after the impact (Cannon et al., 2006; Kring et al., 2006; Cannon and Addison, 2007). The impact layer was also described by Pufahl et al. (2007) from two drill holes very near some of the localities described here. Many of the impact layer localities had been observed, mapped, analyzed, and described previously by numerous geologists, in some cases as much as 60 years ago. The previous interpretations ascribed volcanic or submarine slumping processes as the cause of the breccias, failing to recognize the widespread but sparse shock-induced PDFs in quartz grains.

The ejecta-bearing rocks described here, along with previously documented sites (Addison et al., 2005; Pufahl et al., 2007; Jirsa et al., 2008), define a regionally extensive field of ejecta from the Sudbury impact event that can reasonably be inferred to have originally covered roughly 100,000 km². The ejecta field lies between 500 and 900 km from Sudbury within an arc of 30° radially outward from Sudbury. The currently known ejecta field is likely to be the only representative of proximal to distal ejecta that is preserved, there being no other rocks of suitable age nearer than ~1200 km from Sudbury (Labrador Trough). This ejecta field is a significant addition to the still small inventory of known ejecta blankets from giant impacts on Earth. The study of these newly discovered sites of the Sudbury impact layer is in its infancy, and each site merits

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considerable additional detailed description and interpretation to decipher the processes represented in transport and deposition of the ejecta-bearing units and the implications for impact processes. Our studies to date have emphasized the mapping of the layer through the iron ranges of Michigan to determine its geographic distribution and stratigraphic position. In this paper, we present the first descriptions of 10 sites in Michigan at which we have identified the Sudbury impact layer by examination of outcrops and drill cores. We also provide general descriptions of the physical and geochemical character of the layer based on petrographic microscope examination and chemical analyses of impact layer materials. A diversity of ejecta-bearing rock types suggests that multiple processes of transport and deposition are probably represented across the region, but our studies and understanding of these rocks are too preliminary to attempt anything but the broadest genetic interpretations at this time.

GEOGRAPHIC DISTRIBUTION OF THE SUDBURY IMPACT LAYER

We have identified the Sudbury impact layer at 10 sites in northern Michigan that lie between 500 and 700 km from Sudbury, the presumed impact site, and within an arc of $\sim 15^\circ$ radially outward from Sudbury (Fig. 1). The sites define an area of roughly 30,000 km², in which the impact layer is inferred to be present beneath bedrock and glacial cover. The Michigan sites are separated from the impact site at Sudbury by the Midcontinent rift, which opened and partly closed at ca. 1.1 Ma. Distances from Sudbury to the Michigan sites may have been extended by a few tens of kilometers at most by that rifting event. We have not attempted to correct for that extension in the distances cited here.

The Sudbury impact layer is almost certainly much more extensive than the sites described here. The study of the layer is hampered by the physiographic nature of the region. The Precambrian rocks of Michigan are part of the peneplaned Canadian Shield, and the region is heavily mantled by Pleistocene glacial deposits. Bedrock exposures are sparse, particularly for units such as the Sudbury impact layer, which are not particularly resistant to weathering and glacial erosion. Only five of the sites reported here have been found in surface bedrock exposures. On the positive side, the iron ranges of the region have been mined and explored for more than 150 years, and extensive collections of mineral exploration drill core are available in public repositories or have been made available to us by mining companies, which has helped greatly in overcoming the lack of natural exposures. In two cases (McClure and Iron River–

Crystal Falls), the Sudbury impact layer is thick and extensive enough to constitute a mappable unit and, although not recognized as impact-related rocks at the time of mapping, appears as breccia beds on U.S. Geological Survey (USGS) 1:24,000 scale geologic maps (Puffett, 1974; Clark et al., 1975; James et al., 1968).

GENERAL CHARACTER OF THE SUDBURY IMPACT LAYER

The Sudbury impact layer in Michigan is a bed of breccia and related rocks. Because sites vary from a single drill hole or outcrop to clusters of drill holes and outcrops, the amount of information is quite variable between sites. Table 1 provides some general information about each site, and Figure 2 shows the stratigraphic setting of the impact layer at all known localities in the Lake Superior region. The impact layer in Michigan appears to have been deposited in a submarine to peritidal setting. At localities in the Baraga Basin and Dead River Basin (Fig. 1), the Sudbury layer lies on strata of shallow-water facies and possible supratidal deposits, including localities described previously (Pufahl et al., 2007). Elsewhere, deeper-water deposition is indicated by the low-energy nature of immediately underlying sediments, which most typically are banded iron formation, chert, or shale. Thickness and lithology vary considerably between sites, suggesting multiple processes of deposition or potentially variations in the amount of ejected debris carried to each locality. Five sites are documented as impact-related by widespread, though generally sparsely distributed, quartz grains displaying relict PDFs. Five additional sites have not yielded PDFs but contain breccias with many other features of the PDF-bearing sites and occur at approximately the same stratigraphic horizon as the PDF-bearing breccias. Spherules and flattened fragments of altered devitrified glass, probably formed from impact-generated melts, are also common, as are accretionary lapilli. The layer is as much as 40 m thick, but, more commonly, it is in the range of 5–10 m. Thickness at the Iron River locality could be as great as 150 m locally if the entire coarse clastic unit that contains verified ejecta at its base is considered to be impact-related. In some areas, the layer has not been recognized in drill holes that intersect the stratigraphic horizon at which the layer would be expected, indicating that deposition (or preservation) of the layer was discontinuous.

Shock Metamorphic Features

The most essential evidence of a link between the breccia beds described here and the Sudbury impact is the identification within quartz grains

of planar microstructures (PMs), which are interpreted to be highly annealed relicts of planar deformation features (PDFs) characteristic of a shock-metamorphic origin. Such PDFs indicate that the quartz grains experienced the extreme pressures and strain rates diagnostic of a hypervelocity impact (Grieve et al., 1996; French, 1998). We have identified single sets or multiple intersecting sets of these PMs interpreted to be annealed PDFs in quartz grains from five of the sites described here. These features originally formed as thin planar lamellae of shock-induced glass within the quartz grains. With time, the glass devitrified and recrystallized to quartz. During that process, the annealed PDFs became “decorated” with small fluid inclusions, so that they now appear as thin, regularly spaced planar zones of inclusions (decorated PDFs) within the host quartz grains (French, 1998). Examples of these features from the Sudbury impact layer at five localities in Michigan are shown in Figure 3. Some of the shocked quartz grains have subrounded shapes (Figs. 3D and 3F), indicating that they were derived from previously unmetamorphosed sedimentary rocks or sediments in the upper part of the target.

The decorated PDFs are expressed as planar zones of micrometer-scale inclusions, along which somewhat larger fluid inclusions are aligned. The best-preserved planar zones of inclusions are 1–2 μm in width and spaced 5–10 μm apart. Highly variable degrees of preservation are present, ranging from well preserved to poorly preserved. Thus, it is difficult to estimate the original abundance of PDF-bearing grains at various localities. At present, the abundance of well-preserved examples is very low, even at localities with the richest array of these features. Standard (2.5 \times 4 cm) thin sections that contain shocked quartz typically have 1–5 shocked grains among hundreds of grains that show no evident shock features. The shocked quartz grains have 1–3 sets of decorated PDFs, but single sets are most common.

Shock-induced PDFs in quartz form along favorable crystallographic orientations, which distinguish them from other types of planar microstructures that can form at lower pressures and strain rates. The crystallographic orientations (angles between pole to plane and quartz *c*-axis) of planar microstructures were measured on a universal stage using the methods of von Engelhardt and Bertsch (1969), Stöffler and Langenhorst (1994), and Grieve et al. (1996). Our measurements, combined with several on our thin sections by A.M. Therriault and R.A.F. Grieve (2007, personal commun.), provide polar angles for 33 sets of planes in 17 grains from 9 thin sections, with four sets of planes nonindexed. The total number of measured sets

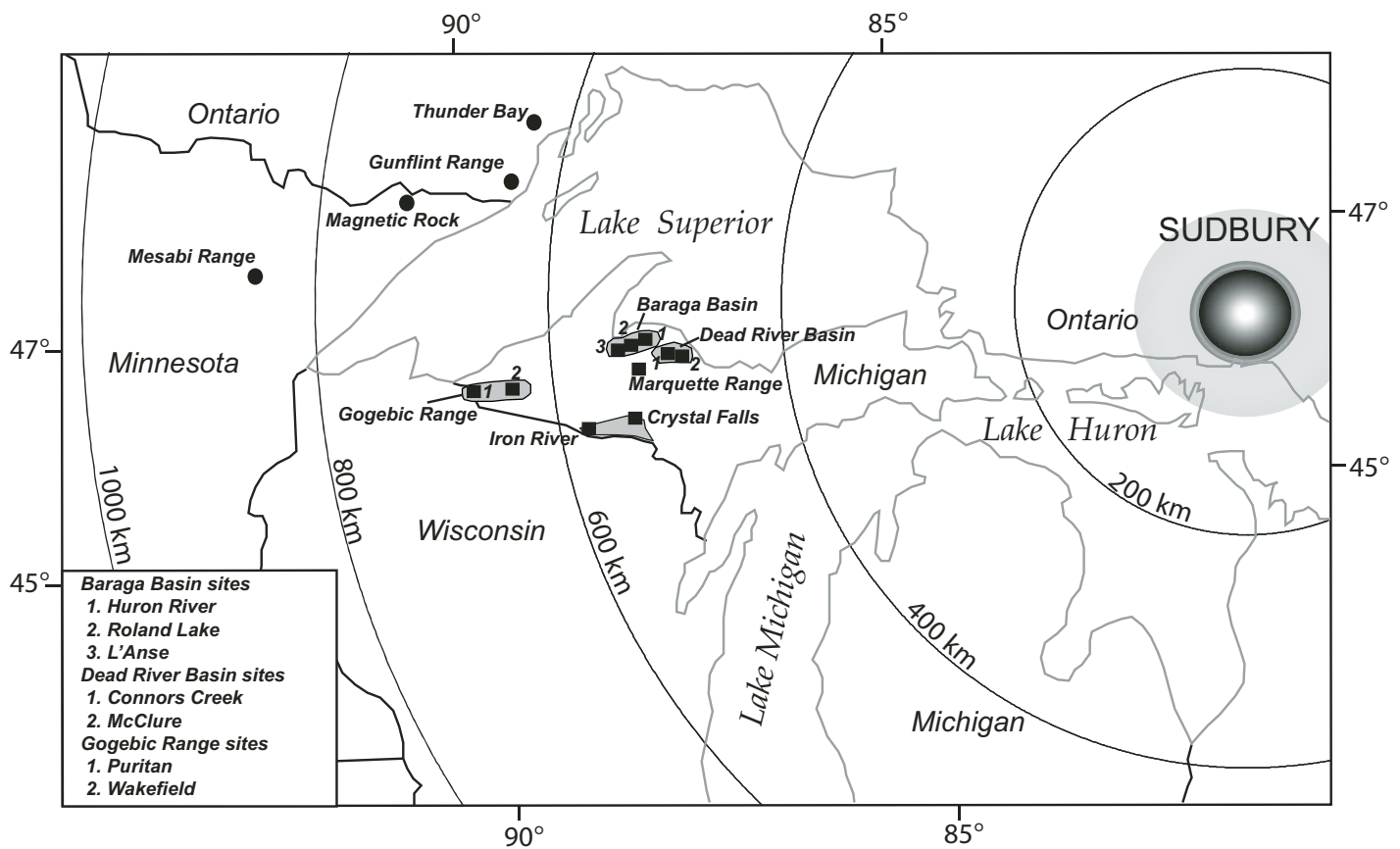


Figure 1. Map showing approximate locations for Sudbury impact-layer localities in Michigan and the surrounding region. Sites in Michigan are new localities reported here. Mesabi Range, Gunflint Range, and Thunder Bay sites are from Addison et al. (2005). Magnetic Rock site is from Jirsa et al. (2008). Concentric circles show distances from the presumed impact point near Sudbury, Ontario.

for each crystallographic orientation (symbol, Miller-Bravais index, polar angle) is $c(0001)$ (0°)—1 set, (no symbol) $\{10\bar{1}4\}$ (18°)—1 set, $\omega\{10\bar{1}3\}$ (23°)—12 sets, $\pi\{10\bar{1}2\}$ (32°)—6 sets, $\xi\{11\bar{2}2\}$ (48°)—2 sets, $s\{11\bar{2}1\}$ (65°)—2 sets, $\rho\{21\bar{3}1\}$ (73°)—1 set, $\tau\{31\bar{4}1\}$ (78°)—2 sets, $\chi\{51\bar{6}1\}$ (82°)—1 set, $a\{10\bar{1}0\}$ (90°)—1 set, nonindexed—4 sets. The orientations of these PMs coincide with common PDF orientations reported in the literature and support the interpretation that they are shock-induced. The most common sets are parallel to the low-index rhombohedral planes $\{10\bar{1}3\}$ and $\{10\bar{1}2\}$; most of the remaining sets are at higher angles and suggest relatively high shock levels. PDFs having the $\{10\bar{1}4\}$ rhombohedral orientation (Fig. 3B) are not mentioned in the standard references (Stöffler and Langenhorst, 1994; Grieve et al., 1996; French, 1998), but they are described elsewhere (e.g., Goltrant et al., 1992; Gurov and Koeberl, 2004). The $\{10\bar{1}4\}$ set of planar microstructures shown in Figure 3B has a polar angle of 18° , which was confirmed by repeated measurements and which does not re-

semble Boehm lamellae. While a more robust data set of PDF measurements would be needed for statistical analysis (Ferrière et al., 2008), these results are generally consistent with those of Pufahl et al. (2007) from part of the same area in that the most common orientations are $\{10\bar{1}3\}$ and $\{10\bar{1}2\}$, and most of the remaining orientations are at higher polar angles.

Accretionary Lapilli

Concentrations of accretionary lapilli are found in the Sudbury layer at six of the sites in Michigan (see Table 1; Fig. 4), and they also are found widely at sites in Ontario (Addison et al., 2005) and Minnesota (Jirsa et al., 2008). They are clearly of impact origin, because shocked quartz is found within them, and they occur in beds interlayered with other ejecta-bearing lithologies. We consider the lapilli to be a key characteristic of the Sudbury layer. Although in themselves not fully diagnostic of impact origin, their occurrence is so closely correlated to definitive impact features that their occurrence

is one critical aspect in the search for the Sudbury layer because the lapilli are easily recognizable in outcrops and drill core. We know of no previous descriptions of lapilli within any other Paleoproterozoic strata in Michigan, so their occurrence appears to be restricted to the impact layer.

The lapilli range up to 2 cm in diameter, but they are more typically from 0.5 to 1.0 cm in diameter. In most occurrences, they are dispersed in a matrix of finer-grained material in beds a few tens of centimeters thick. Less commonly, they form beds of densely concentrated lapilli (Fig. 4A). Many lapilli display concentric zones shown by coarser and finer fragments (Fig. 4B). The size of the fragments ranges from silt to very fine material that is unresolvable with a petrographic microscope. Rarely, grains as coarse as fine sand occur. Lithic fragments form the cores of some lapilli. X-ray diffraction analysis of lapilli from the Connors Creek and McClure localities indicates that they are predominantly quartz (45%–60%) and dolomite (15%–30%). Smaller percentages of microcline,

The Sudbury impact layer in northern Michigan, USA

TABLE 1. SUMMARY OF SOME FEATURES OF THE SUDBURY IMPACT LAYER AT SITES IN NORTHERN MICHIGAN

Locality	Lat./Long.	Distance (km)	Thickness (m)	Underlying	Overlying	PDF	Lapilli	Altered glass
Dead River Basin								
McClure	46°33'N, 87°33'W	480	40	Iron formation	Black slate	Single and multiple sets	Common near base	Vesicular shards
Connors Creek	46°38'N, 87°51'W	500	7	Chert	Chert	Single and multiple sets	Common in lower half	Heterolithic shards
Baraga Basin								
Huron River	46°52'N, 88°05'W	530	5–26	Chert, granite	Argillite	Single and multiple sets	Common in thin beds	Shards and spherules
Roland Lake	46°59'N, 88°11'W	540	4.5	Chert	Black slate	Unconfirmed	30 cm bed	Shards and spherules
L'Anse	46°43'N, 88°21'W	550	2.5	Chert-carbonate	Sheared argillite	Unconfirmed	20 cm bed	Pumice and shards
Marquette Range	46°32'N, 88°03'W	530	15	Chlorite schist	Chlorite schist	Unconfirmed	50 cm bed	Shards, spherules
Crystal Falls	46°06'N, 88°19'W	540	0–50	Iron formation	Argillite, chert	Single and multiple sets	None	Shards, spherules
Iron River	46°05'N, 88°38'W	580	0–150	Iron formation	Argillite, chert	Single and multiple sets	None	Shards, spherules
Gogebic Range								
Wakefield	46°29'N, 89°57'W	640	40	Iron formation	Ferruginous argillite	Unconfirmed	None	Shards?
Puritan	46°28'N, 90°10'W	660	~1	Gray argillite	Brown argillite	Unconfirmed	Probable	Shards, spherules

Note: Underlying—rock type immediately below impact layer. Overlying—rock type immediately above impact layer. PDF—planar deformation feature.

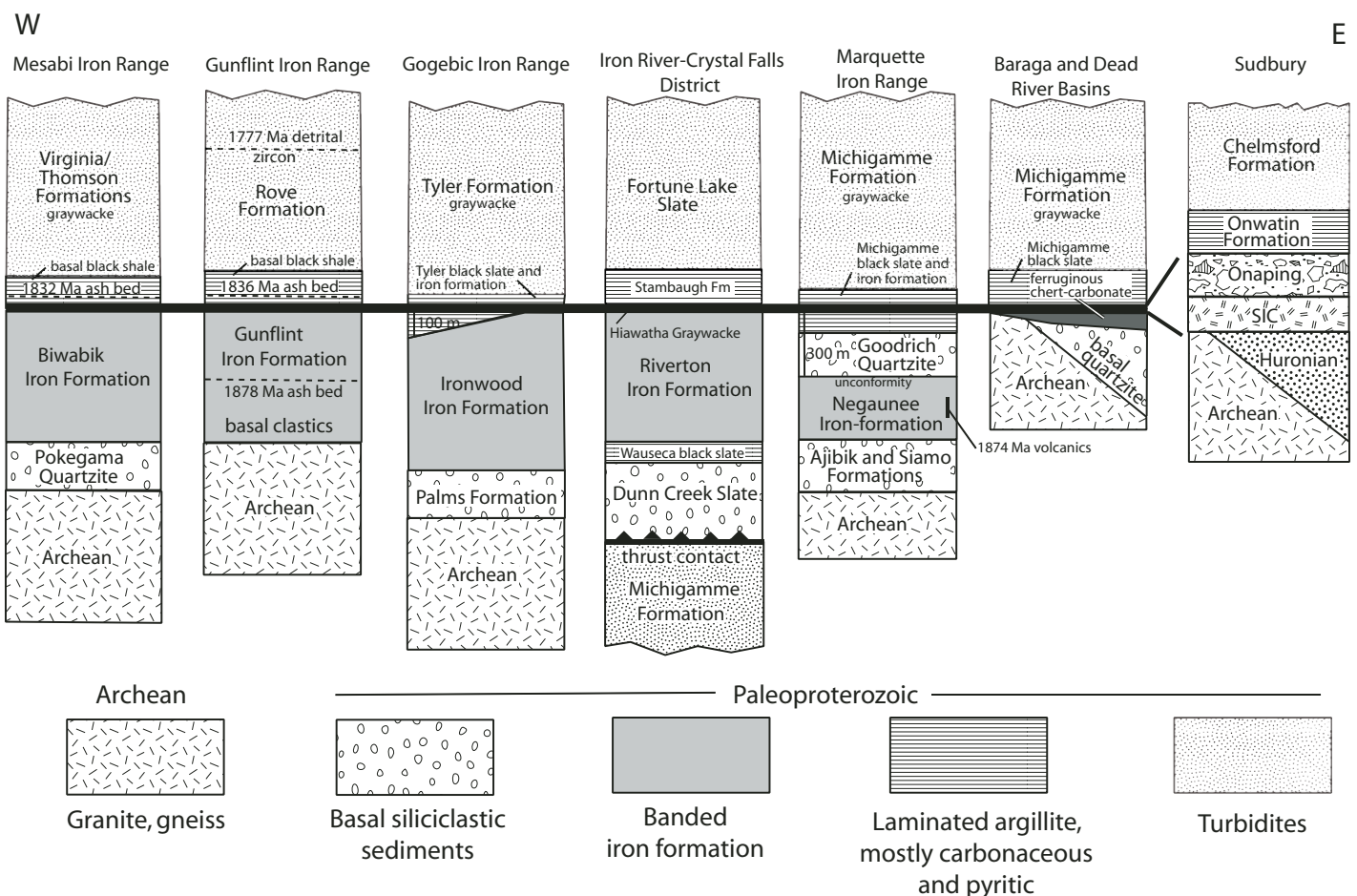


Figure 2. Correlation diagram of stratigraphy in and near the iron ranges of the Lake Superior region based on the time marker provided by the Sudbury impact layer (heavy black line) that formed at 1850 Ma. Radiometric ages for the Gunflint and Mesabi Ranges are from Addison et al. (2005) and Fralick et al. (2002). Radiometric age of the Marquette Range Supergroup is from Schneider et al. (2002).

plagioclase, muscovite, and chlorite make up the rest of the lapilli. It is not clear to what extent the dolomite is a primary mineral in the lapilli rather than a secondary replacement.

Except for the quartz grains with PDFs found in some lapilli and their very broad geographic

distribution, ranging from ~480 km to 730 km distance from Sudbury, the lapilli are similar to volcanic lapilli formed in clouds having 15–25 wt% water (Kring *et al.*, 2006). The lapilli appear to have been produced in a vapor-rich cloud of impact ejecta. Repetitions of lapilli-

rich beds at some sites, local thickness variations, and mixing with ripped-up local materials indicate reworking of lapilli-rich deposits under high-energy conditions.

Similar lapilli have been observed in debris produced by other large impact cratering events, including the Ries (Graup, 1981), Alamo (Warne *et al.*, 2002), Popigai (Masaitis, 2003), Tookoonooka (Bron, 2008), and Chicxulub craters (Ocampo *et al.*, 1996; Pope *et al.*, 1999) and in a proximal ejecta blanket in Scotland (Amor *et al.*, 2008). At the Ries (24 km diameter) crater, accretionary lapilli were found in impact melt breccias that were deposited within the crater (Graup, 1981). Accretionary lapilli in ejecta from the Chicxulub crater are up to 2 cm in size and, like those around Sudbury, were deposited ~550 km from the crater center (Salge *et al.*, 2000).

Stratigraphy and Age

All of the localities in Michigan lie within the lower part of the Baraga Group or partly equivalent Paint River Group. Those within the Baraga Group vary from being the basal unit of the group (Huron River) to ~500 m stratigraphically above the base (Marquette Range) (Fig. 2). This relationship establishes that the early phases of Baraga Group deposition were diachronous within relatively short distances. The Baraga Group is the youngest of three groups comprising the Marquette Range Supergroup (Cannon and Gair, 1970). The Baraga Group lies over the Menominee Group, the major iron-bearing sequence of the Lake Superior region, or, where the Menominee Group is absent, it lies directly on Neoproterozoic basement rocks, such as at Huron River, L'Anse, McClure, and Connors Creek localities. Figure 2 also shows the extent of the Sudbury impact layer in Ontario and Minnesota at the equivalent stratigraphic position.

A critical aspect of our interpretation that the breccia layer in Michigan is a record of the Sudbury impact event requires us to establish independent time constraints showing that the layer was deposited within a permissible window of time to be correlative with the 1850 Ma Sudbury impact. The best available constraints are at the sites in Ontario and Minnesota described by Addison *et al.* (2005) (Fig. 2). In the Gunflint Range in Ontario, the age of deposition of the Sudbury impact layer is constrained by a U/Pb zircon age of 1878 ± 1.3 Ma (Fralick *et al.*, 2002) from a volcanic ash bed in the Gunflint Formation 105 m below the ejecta layer (Addison *et al.*, 2005). An ash bed in the Rove Formation ~6 m above the Sudbury impact layer yielded a U/Pb zircon age of 1836 ± 5 Ma (Addison *et al.*, 2005). Thus, the radiometric age range of ~40 Ma

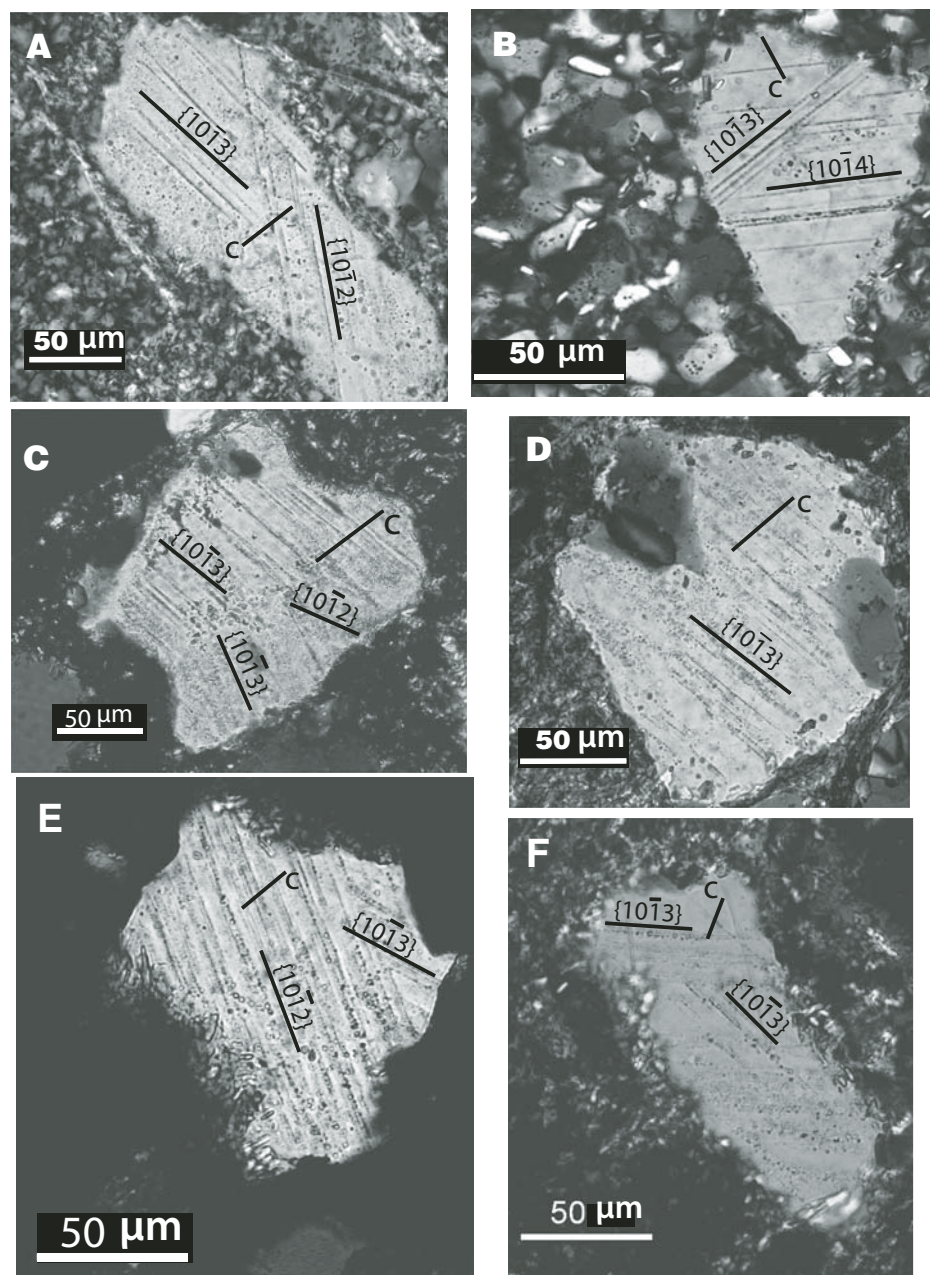


Figure 3. Examples of relict planar deformation features (PDFs) in quartz grains. (A) Baraga Basin (outcrop at Huron River). (B) Baraga Basin (drill core near Huron River). (C) Dead River Basin (McClure site outcrop). (D) Dead River Basin (Connors Creek site outcrop). (E) Hiawatha breccia drill core near Iron River. (F) Hiawatha breccia drill core near Crystal Falls. All photographs are in cross-polarized light. Miller-Bravais crystallographic indices of PDF planes and *c*-axes of quartz grains determined by universal-stage measurements are shown in flat-stage view. Indexing methods are after von Englehardt and Bertsch (1969), Stoffer and Langenhorst (1994), and Grieve *et al.* (1996).

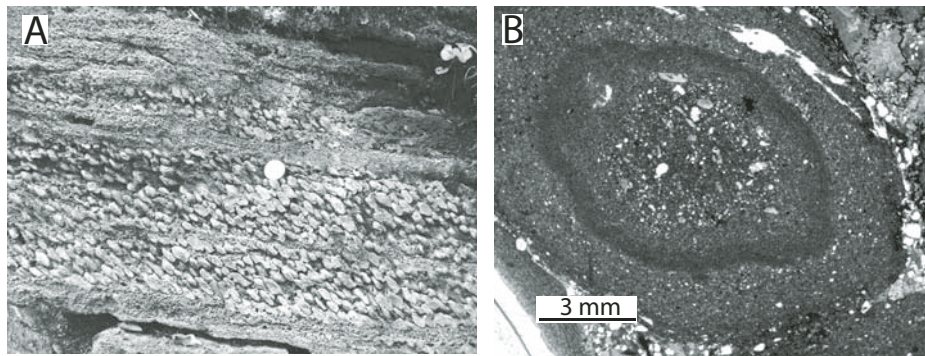


Figure 4. Accretionary lapilli from the Connors Creek locality. (A) Beds of stacked lapilli interlayered with finer-grained beds consisting of mixtures of clastic quartz grains and impact glass particles. Lapilli are deformed by regional tectonic events and elongated in the direction of regional cleavage. Coin is 2.5 cm diameter. (B) Thin section photomicrograph of lapilli showing complex internal zonation of coarser- and finer-grained layers.

between underlying and overlying ash beds includes the 1850 Ma age of the Sudbury impact.

In Michigan, there is a similar age constraint. A U/Pb zircon age of 1874 ± 9 Ma for volcanic rocks that are lateral equivalents of the Negaunee Iron Formation was reported by Schneider et al. (2002), providing a maximum age for the overlying Sudbury impact layer. An upper geochronological boundary is provided by the age of secondary xenotime within basal Baraga Group rocks. Xenotime with $^{207}\text{Pb}/^{206}\text{Pb}$ ages as old as 1800 Ma (<10% discordant) was reported by Vallini et al. (2007). An older minimum age can be inferred from the approximate age of metamorphism, which recrystallized the entire stratigraphic section at ca. 1830 Ma (Schneider et al., 2004; Holm et al., 2007). Because the Sudbury layer is succeeded stratigraphically by many kilometers of turbidites, all of which were deformed and metamorphosed by 1830 Ma, it is reasonable to infer that the Sudbury layer in Michigan was deposited well before the 1830 Ma metamorphism.

Lithology

Details of the lithologic nature of the Sudbury impact layer and enclosing strata are given later in the descriptions of individual sites. In this section, we provide a general description emphasizing the considerable variations in character of the layer from site to site. Figure 5 shows the generalized character of the impact layer and the immediately underlying and overlying beds for all 10 sites discussed here. All known occurrences of the impact layer in Michigan are parts of marine sedimentary sequences. Judging by the enclosing strata, water depths at the time of impact varied from tidal to deep water across the region. Thus, varied interaction between the

ejecta and seawater at the sites of deposition may have been a dominant factor in determining the nature of the layer. Variable seafloor slopes may also have been important as controls on gravity-driven submarine debris flows.

In general, the impact layer is dominated by breccias in which chert is the most abundant clast type. The layer most commonly lies on banded iron formation or an equivalent, variably ferruginous chert-carbonate unit, so there was a readily available local source for the abundant chert. Chert clasts are generally angular and vary in size up to about a meter. Most sites show a distinct layering of coarser and finer breccia beds and, less commonly, poorly sorted sandstone, particularly toward the top of the unit. However, at the McClure locality, the 40-m-thick layer is massive and graded from coarse breccia at the base to sandstone at the top, thus appearing to record a single depositional event. Spherules and shards of devitrified glass are common and make up from a few percent to as much as 50% of the rock. These are all highly altered to chlorite, sericite, and carbonate minerals. A common, but not universal, feature is an abundance of accretionary lapilli as much as 2 cm diameter. These are commonly concentrated in distinct beds from ~10 to 50 cm thick.

Most occurrences of the impact layer appear to be hybrid rocks in which relatively local substrate, such as quartz and chert sand grains and larger chert clasts, is intermixed with impact-generated fragments, largely millimeter- to centimeter-scale altered glass particles along with sparse shock-metamorphosed quartz grains. Secondary alteration to carbonate minerals, and, less commonly, chert or chalcedony, is widespread and, where strongly developed, obscures much of the primary texture of the layer. Although parts of northern Michigan have

undergone moderate- to high-grade metamorphism, all of the sites reported here, with the exception of the Marquette Range site, lie in areas of low metamorphic grade; most show lower greenschist or subgreenschist facies. Regional deformation has also affected the layer, in places producing internal deformation and flattening along a regional cleavage direction, but seldom, if ever, has deformation been intense enough to obliterate primary structures.

Thickness

Figure 5 shows the approximate thickness of the impact layer across Michigan. These thicknesses are somewhat interpretive. Although the lower contact of the impact layer is universally sharp, the upper contact of the layer is not clearly defined at all sites. At many sites, coarse breccias of the lower part of the formation grade upward into finer-grained rocks with decreasing amounts of clearly impact-related grains. In all cases, this gradation eventually leads to laminated fine-grained shale and argillite that are clearly postimpact units. We have generally considered the contact of coarse clastic rocks and overlying laminated fine clastics to mark the upper limit of the impact layer. By this criterion, the layer is locally as much as 150 m thick near Iron River, but it is generally considerably thinner. Minimum thickness is probably zero, since we have examined several drill holes that almost surely crossed the impact horizon but contained no identifiable breccias or other indications of ejecta. We observed no regional trends in thickness changes across the 200 km width of this study area, suggesting that local settings for deposition and preservation were more important than distance from Sudbury in determining the thickness of the layer.

Paleogeography of the Region at 1850 Ma

The Sudbury impact at 1850 Ma occurred in an active tectonic belt, within which both pre-impact and postimpact strata are preserved. A long history of geologic study of this belt, augmented by recent precise geochronology, allows inferences to be made concerning the geography of the region at the time of impact. An understanding of this paleogeography may be one key in interpreting the regional effects of the impact as recorded in the lithologically diverse impact layer.

At Sudbury, there are no immediately pre-impact rocks preserved. The Sudbury structure is surrounded by strata of the Huronian Supergroup, older than 2.2 Ga, and its Archean basement. The impact-related rocks of the Onaping Formation, however, are conformably overlain

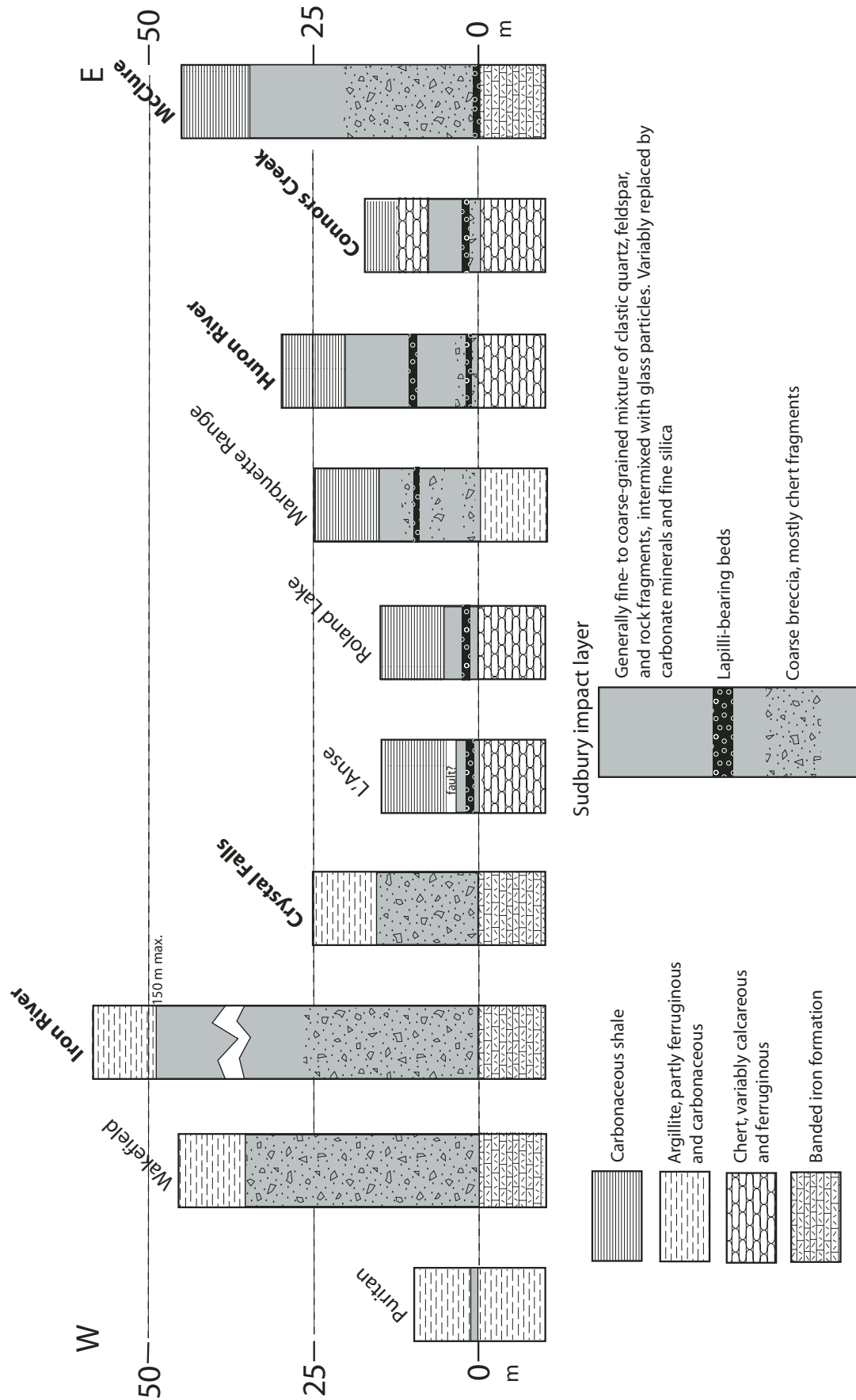


Figure 5. Cross sections of the Sudbury impact layer and enclosing strata at 10 sites in Michigan. Sites labeled in bold are those at which quartz grains with relict planar deformation features (PDFs) have been identified.

by a marine sedimentary sequence of black shales of the Onwatin Formation and, in turn, by turbidites of the Chelmsford Formation (Pye et al., 1984). Also, parts of the Onaping Formation contain abundant fragments of carbonaceous argillite, indicating that this lithology was present in the area now occupied by the crater at the time of impact (Mungul et al., 2004). These relationships indicate that the impact was immediately followed by sedimentation in an anoxic starved basin of sufficient depth to be entirely below wave base (Long, 2004). Based on paleocurrent analysis (Long, 2004), this basin is likely to have been part of a pre-impact regional foreland basin rather than a local depression caused by the impact. Thus, the impact seems most likely to have occurred in a marine setting with water depth in excess of wave base.

The ejecta sites described in this paper, as well as those reported previously (Addison et al., 2005; Jirsa et al., 2008), all share a similarity with the Onaping Formation: they are succeeded by sediments consisting wholly or partly of black shale followed by turbidites, suggesting that all of the known ejecta layer was deposited into a regionwide sedimentary basin about which a good deal of detailed information is known. This basin is well established to have been a continental margin foreland basin that was developing north of northward-accreting island-arc terranes now preserved in Wisconsin (see Schulz and Cannon [2007] for a recent review).

The ejecta in the Gunflint Range in Ontario lies on the Gunflint Iron Formation, possibly along a cryptic hiatus between the two formations, and it is overlain by black shales of the Rove Formation (Addison et al., 2005). Deposition of the ejecta may have been subaerial. Along strike at the Magnetic Rock site in Minnesota (Fig. 1), coarse breccias of the impact layer contain folded and contorted masses of chert from the Gunflint Iron Formation. This suggests that the Gunflint Iron Formation was at least partly un lithified at the time of impact, and that there is little or no hiatus there. Thus, the north shoreline of the advancing foreland sea may have lain between the Minnesota and Ontario sites at 1850 Ma. On the Mesabi iron range, ~150 km southwest of the Magnetic Rock site, ejecta occurs at the top of the Biwabik Iron Formation and is overlain by black shales of the Virginia Formation. No hiatus has been documented or suggested between the two formations on the Mesabi Range, and a conformable and somewhat gradational contact of the Biwabik and Virginia Formations is widely recognized (e.g., Lucente and Morey, 1983). However, the iron formations beneath the Sudbury impact layer in both the Gunflint and Mesabi Ranges contain

abundant shallow-water facies, indicating that deposition of the Sudbury layer occurred in a nearshore, shallow-water setting.

The Michigan sites exhibit some stratigraphic differences probably related to their position somewhat nearer to the accreting southern arc terrane and thus in a more tectonically active part of the basin. The Huron River, Roland Lake, L'Anse, and Connors Creek occurrences, as well as nearby occurrences described previously (Pufahl et al., 2007), are in peritidal settings similar to the Ontario and Minnesota occurrences. At all other Michigan sites, the Sudbury layer lies on even-bedded rocks, commonly iron formation and, less commonly, laminated argillite and black shale, all of which appear to indicate a deeper-water setting. Therefore, deposition of the Sudbury layer in Michigan seems to have occurred in more varied settings than at the Minnesota and Ontario sites, accounting for at least part of the variability in lithologies and thicknesses.

DESCRIPTION OF LOCALITIES

We found the Sudbury impact layer at 10 localities across the Upper Peninsula of Michigan (Fig. 1). These localities in the Dead River Basin, the Baraga Basin, the Marquette iron range, the Iron River–Crystal Falls iron district, and the Gogebic iron range are described next and are summarized in Table 1. The lithology of the impact layer and enclosing strata is shown in Figure 5.

Baraga Basin

We have divided occurrences of the Sudbury impact layer in the Baraga Basin (Fig. 1) into three geographically separate groups and discuss each separately. These localities span a distance of ~40 km east-west along the basin. The stratigraphy within the basin consists of Paleoproterozoic strata of the Baraga Group, composed entirely of various informal stratigraphic units of the Michigamme Formation, which lies unconformably on Neoarchean granitic rocks. The Michigamme Formation is dominated volumetrically by graywacke turbidites that make up the upper part of the formation. The basal parts of the Michigamme vary in both lithology and thickness throughout the basin and consist of an assemblage of basal quartzite overlain by iron formation and equivalent variably ferruginous banded chert-carbonate, argillite, black shale (Cannon, 1977; Klasner et al., 1979), and the newly recognized Sudbury impact layer. The impact layer occurs at precisely the same stratigraphic position at all of these localities. It lies directly on a unit of

chert, which is variably interbedded with carbonate beds and is also variably ferruginous, although nowhere to the extent of being a true iron formation. The impact layer is overlain by laminated shale, mostly carbonaceous black shale.

Huron River

The Huron River locality (Fig. 1, Baraga Basin locality 1) consists of outcrops in the bed of the Huron River at the “lower falls” and a few additional sporadic outcrops extending ~15 km to the east, both in the river bed and along the south flank of the Huron Mountains. Detailed maps of these outcrop areas are presented by Shaw (1974), who considered the Sudbury impact layer to be volcanoclastic rocks. A detailed description of outcrops on the Huron River was presented by Kalliokoski and Lynott (1987), who identified silicified chert breccia, which we herein document as the Sudbury impact layer. Three mineral exploration drill holes, all within about a kilometer of the lower falls outcrop area, provide a section through the layer, and most of the following description is based on studies of those drill cores.

The thickness of the layer varies from 5 to 26 m between the holes, indicating a substantial variation in initial thickness over short distances. In all three holes, the Sudbury layer is overlain by black, red, and green argillite, with an apparently conformable contact. The layer lies either directly on Archean granitic rocks or on as much as 2 m of bedded chert-carbonate and phosphatic rocks and underlying pebble conglomerate (Kalliokoski and Lynott, 1987). Locally, small stromatolite mounds occur in chert beds immediately below the ejecta layer. It appears that at the time of impact, the Huron River area was experiencing an initial phase of marine transgression, and a thin and discontinuous layer of marine chemical sediments had been deposited along with sporadic basal pebble conglomerate over the Neoarchean basement rocks. Thus, there may have been little or no interaction between ejecta and seawater at this site. The Sudbury layer may have been deposited in part subaerially, assuming that the thickness of the layer exceeded water depth at the time of impact and deposition.

The Sudbury layer at Huron River is a well-bedded sequence of coarser- and finer-grained breccia. Clast size varies from sand or finer, to more than 3 cm in drill core; in outcrop, clasts of chert as large as 0.5 m are present locally. Chert forms by far the greatest percentage of the larger clasts. Intense silicification of much of the layer has destroyed much of the primary texture and in places results in a rock that is nearly all fine-grained silica. Limonite staining produced by secondary oxidation also obscures

primary features in many samples. Nevertheless, a rich array of relict spherule and shard forms is evident (see Figs. 6A, 6B, and 6C). Clastic grains of quartz are relatively rare compared to many other sites, making up only a few percent of the rock. However, the percentage of these grains that contain PDFs (Figs. 2A and 2B) appears to be higher than at other localities. A typical abundance of PDF-bearing quartz grains is a few grains per standard thin section. Accretionary lapilli up to ~2 cm in diameter are concentrated in distinct beds up to 70 cm thick (Fig. 6D). One drill hole contains two such beds ~12 m apart stratigraphically, whereas only a single bed is present in other drill holes and outcrops.

Coarse, chert-rich breccias near the base of the Huron River section indicate that a high-energy event, possibly a ground surge, dismembered relatively local bedrock to form the breccia beds. However, many of the finer-grained and bedded parts of the section, which preserve lapilli and abundant altered glass particles, appear to have been made up largely of ejecta before the intense secondary alteration, and they may represent deposition from a turbulent ejecta cloud after the initial ground surge.

Roland Lake

The Sudbury impact layer near Roland Lake is known only from multiple closely spaced exploration drill holes. The description here is based on a single hole characterized by the thickest and most lithologically complete section. The Sudbury layer is 4.5 m thick and lies with a sharp contact on an underlying chert-carbonate unit. In thin section, the contact shows minor disconformity where the Sudbury layer has eroded and truncated fine-bedding lamination of chert layers. It is overlain by a sharp contact with pyritic black slate. The drill hole ended in the basal quartzite that makes up the lowest unit of the Baraga Group in this part of the basin and did not reach the Archean basement. Based on the section that was intersected and a typical thickness of the basal quartzite, the Sudbury layer is a minimum of ~40 m above the base of the Baraga Group.

In thin section, many features similar to those illustrated above for the Huron River locality are preserved, although a pervasive replacement by carbonate minerals obliterates much of the finer-scale texture. An array of altered glass spherules and shards is present. Shards as much as a centimeter long occur in some layers and are readily identified in hand specimens as black aphanitic angular fragments. Accretionary lapilli ~1 cm in diameter are abundant about a meter above the base of the unit within an ~30-cm-thick bed. Quartz grains are rare, and none with PDFs has been identified.

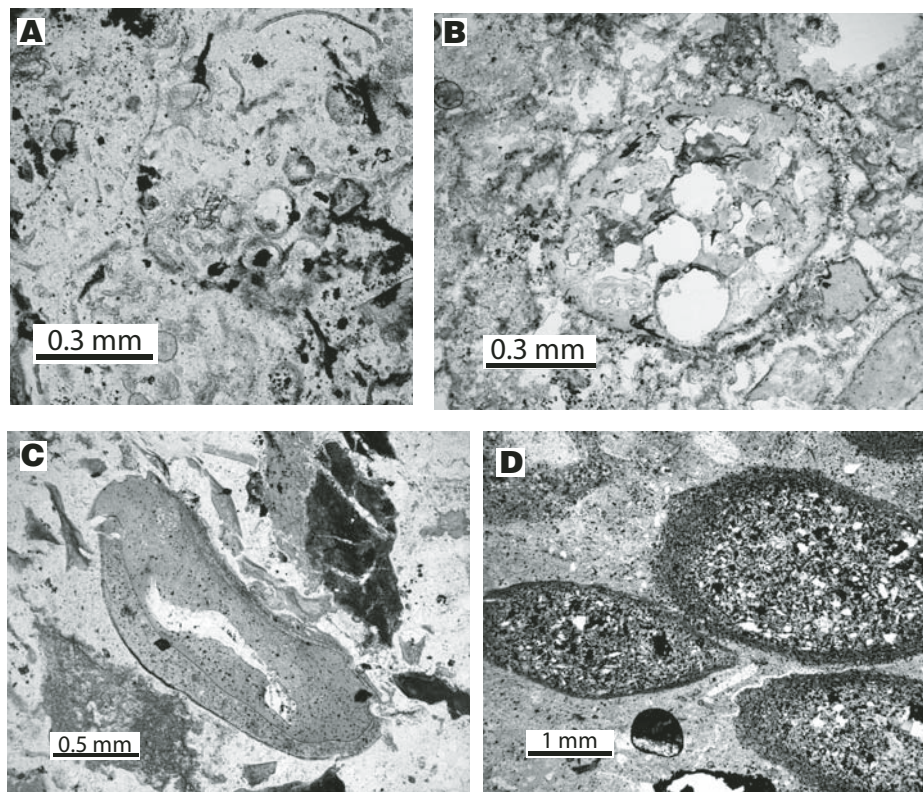


Figure 6. Microscopic features for the Huron River locality in the Baraga Basin. (A) Spherules and thin curved plates in matrix of secondary silica. Plates may be spalled margins of larger spherules or broken bubble walls. (B) Sphere-in-sphere structure. (C) Flattened spherule showing rim and core of different mineralogic composition. Elongate light area in center of spherule is now quartz, possibly filling original vesicle. Note that inner zone appears to break through outer zone on upper left. (D) Accretionary lapilli flattened parallel to bedding. All photographs are in plane polarized light.

The Roland Lake section appears to have little or no coarse breccia, indicating that, if a ground surge did cross this area, it did not deposit material here. All of the material appears to represent a layered unit of ejecta rich in glass and lapilli, which we interpret to be deposited from a turbulent ejecta cloud.

L'Anse

The Sudbury impact layer near the village of L'Anse was observed in an exploration drill hole that intersects a complete and lithologically varied sequence of ejecta-bearing beds. The Sudbury impact layer here is ~2.5 m thick, but the top is highly sheared, so the original thickness may have been greater. The layer lies on a banded chert-carbonate rock with traces of disseminated pyrite in some beds. The base of the Sudbury impact layer is somewhat indistinct in an ~10-cm-thick zone, across which an upward increase in abundance of fine altered glass particles occurs in a cherty matrix. Above the contact zone, there is 0.4 m of polymict altered

glass breccia with individual glass fragments to ~2 cm in diameter. Many are vesicular and variably flattened (Fig. 7C). Only a few percent of quartz sand grains occur. This is overlain by 0.5 m of bedded pumice, in which individual beds show a distinct imbrication of the pumice fragments (Fig. 7A). The pumice fragments are very angular (Fig. 7B) and are held in a siliceous matrix. Interbeds consist of rounded quartz sand grains in a similar siliceous matrix. This unit grades upward to a 0.2 m bed rich in ~1-cm-diameter accretionary lapilli. Above the lapilli, and composing the remainder of the layer, there is a normally graded unit, ~1.5 m thick, consisting of polymict altered glass breccia similar to the basal unit except for the inclusion of ~20% rounded quartz sand grains. The size of altered glass fragments decreases upward from ~1 cm near the base to a few millimeters near the top. The upper contact of the recognizable Sudbury layer is a zone of strongly sheared rock, ~0.2 m thick, which grades upward to a schist in which quartz sand grains are suspended in a matrix of

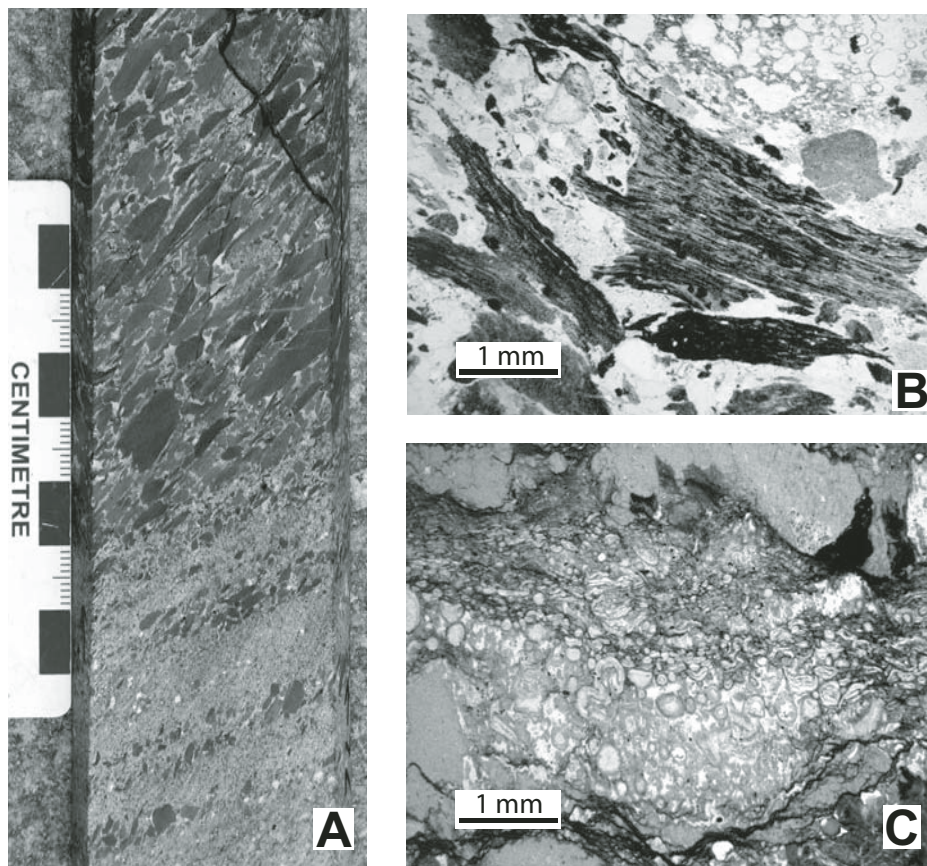


Figure 7. L'Anse locality, Baraga Basin. (A) Drill core with pumicelike fragments (dark clasts) showing imbrication within clast-rich beds. (B) Photomicrograph of pumicelike fragments from the bed shown in A. (C) Clast of highly vesicular siliceous material from lower polymict glass breccia. Vesicles grade from nearly spherical in bottom of clast to flattened in top.

fine quartz and sericite. Planar deformation features have not been recognized here.

The absence of coarse breccias, including any containing local chert clasts, suggests relatively low-energy deposition and little incorporation of underlying strata. However, apparent imbrication of glass fragments as well as incorporation of rounded quartz sand grains of possible local derivation indicate some degree of directional flow and interaction with the land surface during deposition. The abundance of altered glass and accretionary lapilli in distinct beds indicates that most of the section preserved in the drill hole represents ejecta with relatively minor dilution from locally derived material.

Dead River Basin

Knowledge of the Sudbury impact layer in the Dead River Basin comes from two localities, both of which consist of natural rock exposures. Additional occurrences in two drill holes were reported by Pufahl et al. (2007).

Connors Creek

The Connors Creek locality in the western part of the Dead River Basin (Fig. 1) consists of two outcrops ~200 m apart along strike. Outcrops are sparse in the vicinity, but the ejecta layer appears to be ~150 m or less stratigraphically above the base of the Michigamme Formation based on its distance from an exposure of the basal unconformity of the Michigamme and underlying Archean granite, assuming a constant dip of the strata. The layer appears to be ~7 m thick and displays a complex internal stratigraphy (Fig. 8). It is both underlain and overlain by chert that contains features indicative of peritidal conditions similar to those reported in nearby drill holes (Pufahl et al., 2007).

Three distinct units are defined based on physical and mineralogical characteristics (Fig. 8). The basal unit is a coarse chert breccia, ~1 m thick, containing chert clasts up to ~1 m in longest dimension. The chert is identical to immediately underlying bedded chert, and the fragments are most likely slightly displaced and

transported rip-ups of the underlying unit. The matrix of the breccia is a mixture of fragments with multiple lithologies of nearly aphanitic material that vary from felsic (sericitic) to mafic (chloritic). Clast size ranges up to a few millimeters. Some clasts, particularly the more chloritic compositions, contain internal spherical structures, apparently remnants of vesicles, and appear to be metamorphosed devitrified mafic glass. Sand-sized chert grains are also abundant. Quartz sand grains are relatively rare in contrast to their abundance in overlying units.

A middle unit, about a meter thick, consists of well-bedded material devoid of large clasts but rich in accretionary lapilli up to 2 cm in diameter. The lapilli are concentrated in several beds (Fig. 4A) separated by finer-grained multilithic microbreccia consisting of nearly aphanitic angular grains with a variety of compositions as indicated by a varying amount of very fine-grained sericitic and chloritic minerals. Grain size ranges up to a few millimeters. The groundmass contains abundant rounded to subangular quartz grains. Secondary carbonate alteration has destroyed much of the primary texture in the matrix. The lapilli commonly display internal layering of coarser- and finer-grained bands, indicating a complex growth history (Fig. 4B). Some are broken and now occur as angular fragments.

The uppermost unit, ~5 m thick, consists of a basal unit of breccia containing sparse clasts of black chert as much as a meter in long dimension dispersed in a multilithic matrix. Accretionary lapilli are abundant near the base, but they are dispersed rather than occurring in discrete beds as in the middle unit. The finer matrix consists of rock fragments similar to the middle unit and an abundance of sand-sized rounded quartz grains and lesser chert grains (Fig. 8B). The breccia grades upward into sandstone similar to the matrix of the breccia. In one outcrop, the upper unit displays an erosional contact with the middle bedded unit, along which lapilli-rich beds of the middle unit are truncated at the base of the upper unit (Fig. 8C). The second outcrop appears to consist only of the upper unit, so the basal breccia unit and middle lapilli-rich unit may have been completely eroded in the 200 m between the two outcrops. In this outcrop, the sandstone consists of several meter-scale beds marked by a thin layer of lapilli and angular chert at their base and planar cross-bedded sand above (Fig. 8A). The upper unit is overlain by chert identical to that which underlies the ejecta layer. The thickness of this overlying chert cannot be determined because of lack of exposure, but it is at least several meters thick. Based on relationships nearby along strike, the chert unit is succeeded by black slate and in turn by graded-bedded graywacke.

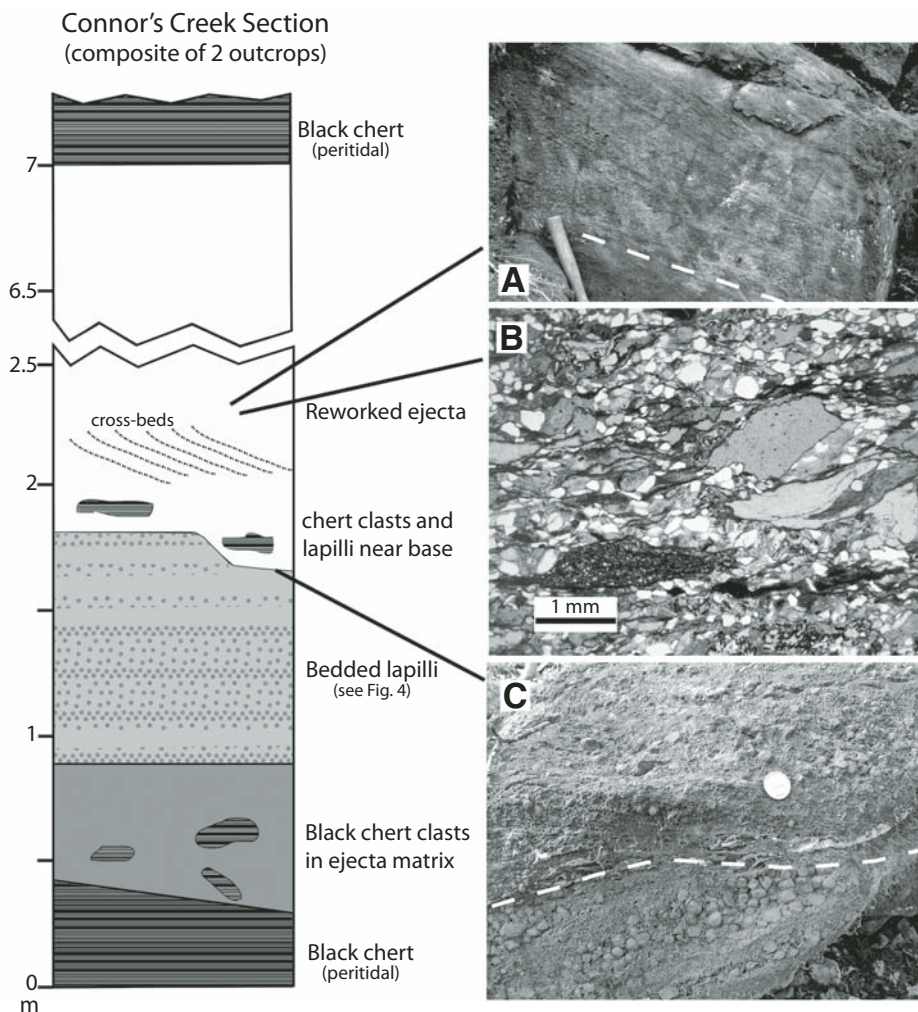


Figure 8. Stratigraphic section of Connors Creek outcrop, Dead River Basin, and typical textures of units. (A) Planar cross-bedded coarse-grained sandstone. Dashed line indicates base of cross-bedded unit. Accretionary lapilli are scattered in lower few centimeters of bed. Hammer handle is shown for scale. (B) Photomicrograph of upper sandstone unit showing multiple compositions of altered glass particles and abundant quartz grains (white). (C) Erosional contact (dashed white line) between upper reworked unit and middle bedded lapilli unit. Coin is 2.5 cm in diameter.

A very sparse population of quartz grains with PDFs has been found. These grains and the abundance of accretionary lapilli and multi-lithic altered glass fragments all serve to confirm this unusual lithologic assemblage as the Sudbury impact layer. At least two depositional processes are represented here. The basal chert breccia seems clearly to be composed of ripped up clasts of the underlying bedded chert surrounded by finer-grained ejecta particles, and it is likely to be a ground surge deposit. The middle bedded lapilli unit is more problematic. The high concentration of lapilli in discrete beds is unprecedented in other occurrences in Michigan. The abundance of rounded quartz grains and rounded sand-size chert grains throughout

the matrix precludes a strictly air-fall deposition and suggests significant intermixing with more local material. The bedded lapilli may record waning phases of a ground surge. The upper cross-bedded unit is also unique to this locality. The erosional contact with the bedded lapilli, the thickly bedded character, and planar cross-beds suggest deposition from a high-velocity flow, possibly one or more tsunami surges across the shallow tidal environment in which the Connors Creek material was deposited.

McClure

The McClure locality consists of a group of outcrops from which a complete stratigraphic section can be pieced together. The Sudbury im-

act layer here is distinct from other localities in several aspects. First, it is unusually thick, having a stratigraphic thickness of ~40 m. Second, it is devoid of internal layering, being a single graded unit varying from basal breccia to upper sandstone. Third, it contains an unusually high percentage of relict glass fragments, particularly in the lower half of the layer.

The Sudbury layer at the McClure locality lies on an unnamed banded iron formation, a member of the Michigamme Formation and the lateral equivalent of the bedded chert that underlies the Connors Creek layer. Its base is ~300 m stratigraphically above the basal unconformity with Archean volcanic rocks. The area has been mapped in detail (Puffett, 1974; Clark *et al.*, 1975), and the Sudbury layer is thick and continuous enough to be mapped as a unit for ~10 km along strike (mapped as chert conglomerate and breccia). We have divided the Sudbury layer here into two lithologic types, a lower chert breccia unit and an upper sandstone unit. The lowermost portion of the lower breccia consists of reoriented slabs of the underlying iron formation that are as much as a meter in longest dimension. The matrix surrounding the slabs consists of poorly sorted, matrix-supported rock fragments up to ~2 cm long and sand-sized rounded grains of quartz and lesser chert. Some of the rock fragments may be altered devitrified glass. The matrix is very fine-grained and highly chloritic. Accretionary lapilli are generally sparsely distributed throughout the matrix in the basal 1–2 m, although they are locally abundant. The basal beds grade upward into matrix-supported breccia in which chert is the most abundant clast type, but several other rock types are present, including quartzite and rhyolite (Puffett, 1974) (Fig. 9A). Both the percentage and size of the clasts gradually diminish up-section over ~20 m. The matrix is notable in its content of particles of devitrified, commonly vesicular, mafic glass, now composed largely of chlorite (Figs. 9B and 9D). These glass particles make up 30%–40% of the matrix. Many glass particles have complex shapes characteristic of volcanic fiamme and may have been sufficiently hot to have been deformed plastically shortly after deposition. Rounded sand-sized quartz grains are also abundant and make up ~15% of the matrix. These grains occur in a groundmass of nearly aphanitic quartzo-feldspathic material that is remarkably uniform through the lowermost 25 m of the layer. These percentages remain the same throughout the chert breccia unit, although the percentage of large clasts diminishes up-section.

The upper sandstone unit, ~18 m thick, is massive dark gray sandstone with rare chert clasts up to a few centimeters in diameter. The

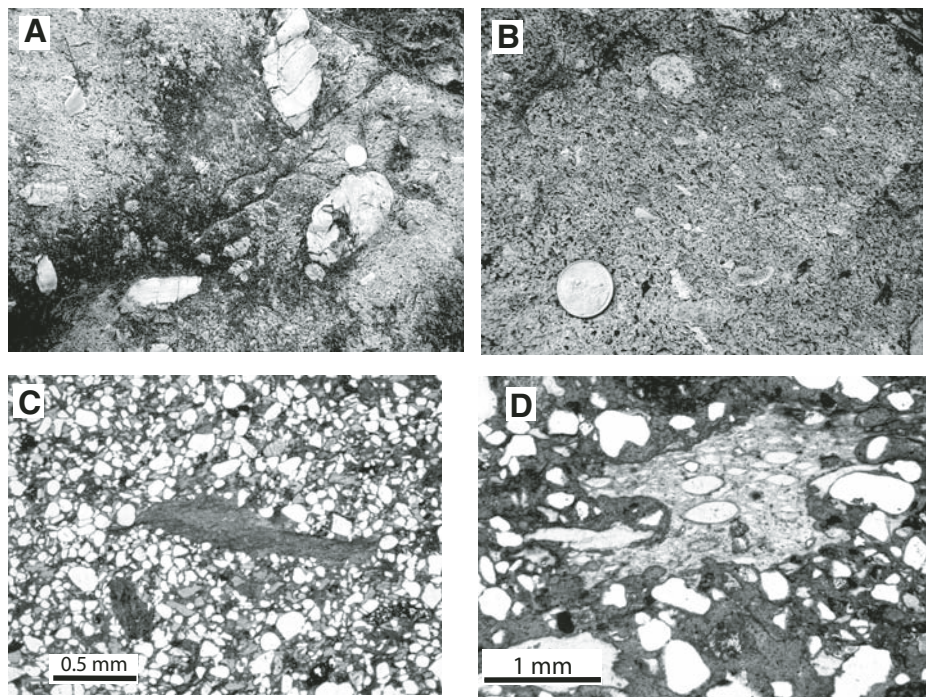


Figure 9. Outcrop and microscopic features of the McClure locality, Dead River Basin. (A) Large chert clasts (light colored) in matrix of finer fragmental material. Coin is 2.5 cm in diameter. (B) Close-up of breccia matrix showing scattered lapilli (upper center) and abundant small chloritic fragments (darker gray to black, negative weathering masses). Coin is 2 cm in diameter. (C) Photomicrograph of upper sandstone showing clast-supported framework of quartz and minor feldspar grains and fragments of altered devitrified glass, now mostly chlorite. (D) Flattened altered vesicular glass particle, now mostly chlorite, in aphanitic matrix, which also contains abundant quartz grains. C and D are in plane polarized light.

abundance of relict glass particles is less than in the lower unit and is typically ~20%, whereas the quartz content increases to 30%–40% as mostly rounded sand-size grains. The percentage of groundmass remains nearly unchanged from the lower unit and is typically ~40% of the rock. The groundmass appears to differ from the lower unit: it is an aggregate of fine clastic particles rather than the uniform aphanitic material of the lower unit (Fig. 9C). The lithologic change from lower to upper unit occurs between our samples spaced ~3 m apart stratigraphically, but we observed no sharp contact in outcrop, and the change appears to be gradational. The top of the upper unit appears to be conformable with overlying laminated black slate.

The Sudbury impact layer at the McClure site differs from all other sites in several respects. First, it appears to be a single graded depositional unit, ~40 m thick, that varies from coarse chert breccia at the base to sandstone at the top with none of the internal layering observed in outcrops. Second, it has abundant glass particles that have a grain size larger than glass particles at other sites, up to ~2 cm long. These

particles commonly show fiammelike shapes, unique to the McClure site, suggesting that they may have been hot during deposition. Third, accretionary lapilli are only in the very basal part of the section, indicating that they arrived at the site with the first impact material rather than occurring within the impact sequence as shown at other sites.

The depositional setting of the McClure site appears to have been in deeper water than Connors Creek and sites in the Baraga Basin. The underlying iron formation is even-bedded and was apparently deposited below wave base, in contrast to the shallow to peritidal setting of other sites. Thus, McClure may record a higher degree of interaction between seawater and ejecta. The abundance of large chert clasts, as well as abundant rounded sand grains of quartz, chert, and feldspar, indicates that vigorous erosion of underlying units by the ejecta accounts for the intermixing of ejecta and local material, probably by a ground surge. This mixing could have been generated by the high velocity of the ejecta, despite the deeper-water setting of the site. Alternatively, the mixing may have

occurred in a neighboring shallower area with material then carried away from the crater into the deeper water of the site. The single, thick, graded bed of material may indicate gradual settling of a single surge of material through a sediment- and ejecta-laden water column.

Marquette Iron Range

The single known locality for the Sudbury layer in the Marquette iron range is an exploration drill hole near the town of Michigamme (Fig. 1). This hole penetrated ~15 m of graywacke and breccia that contains particles of altered glass and has a single bed, ~0.5 m thick, which is rich in accretionary lapilli. As with the localities in the Baraga Basin and Dead River Basin, the layer is within the lower part of the Michigamme Formation, although details of the stratigraphy differ. The locality lies within the staurolite zone of regional metamorphism (James, 1955), and all rocks are strongly recrystallized. Quartz grains in particular are recrystallized, so that any shock metamorphic features that might have been present have been annealed. Biotite is abundant, and garnet occurs in some beds. Rarely, small grains of staurolite are seen in thin sections. The layer is underlain by laminated chloritic and carbonaceous slate containing abundant pyrite. The basal contact is sharp (Fig. 10A). Based on surface mapping (Klasner and Cannon, 1978), the Sudbury layer appears to be from 500 to 600 m stratigraphically above the Neganee Iron Formation and separated from it by conglomerate and quartzite of the Goodrich Quartzite, and graphitic and pyritic slate and iron formation of the basal part of the Michigamme Formation. The drill hole also intersects ~60 m of chloritic and pyritic black slate and argillite above the Sudbury layer. The upper contact marks the transition from massive graywacke of the Sudbury layer to the finely laminated overlying unit.

The Sudbury impact layer consists of thick bedded greywacke, in which sand-sized grains are dominantly quartz, lesser chert fragments, and little or no feldspar. The matrix is recrystallized and rich in biotite. Grain:matrix ratios vary considerably, so that both clast-supported and matrix-supported units are interlayered. The basal 50 cm contain abundant intraclasts of black mudstone similar to the immediately underlying strata. Many of these clasts are highly contorted, indicating that they were not indurated when incorporated into the Sudbury impact layer. Beds of breccia are interlayered in the graywacke. These consist of variable amounts of larger clasts, with maximum diameter occasionally greater than the 3.5 cm diameter of the drill core (Fig. 10C). Clast abundance

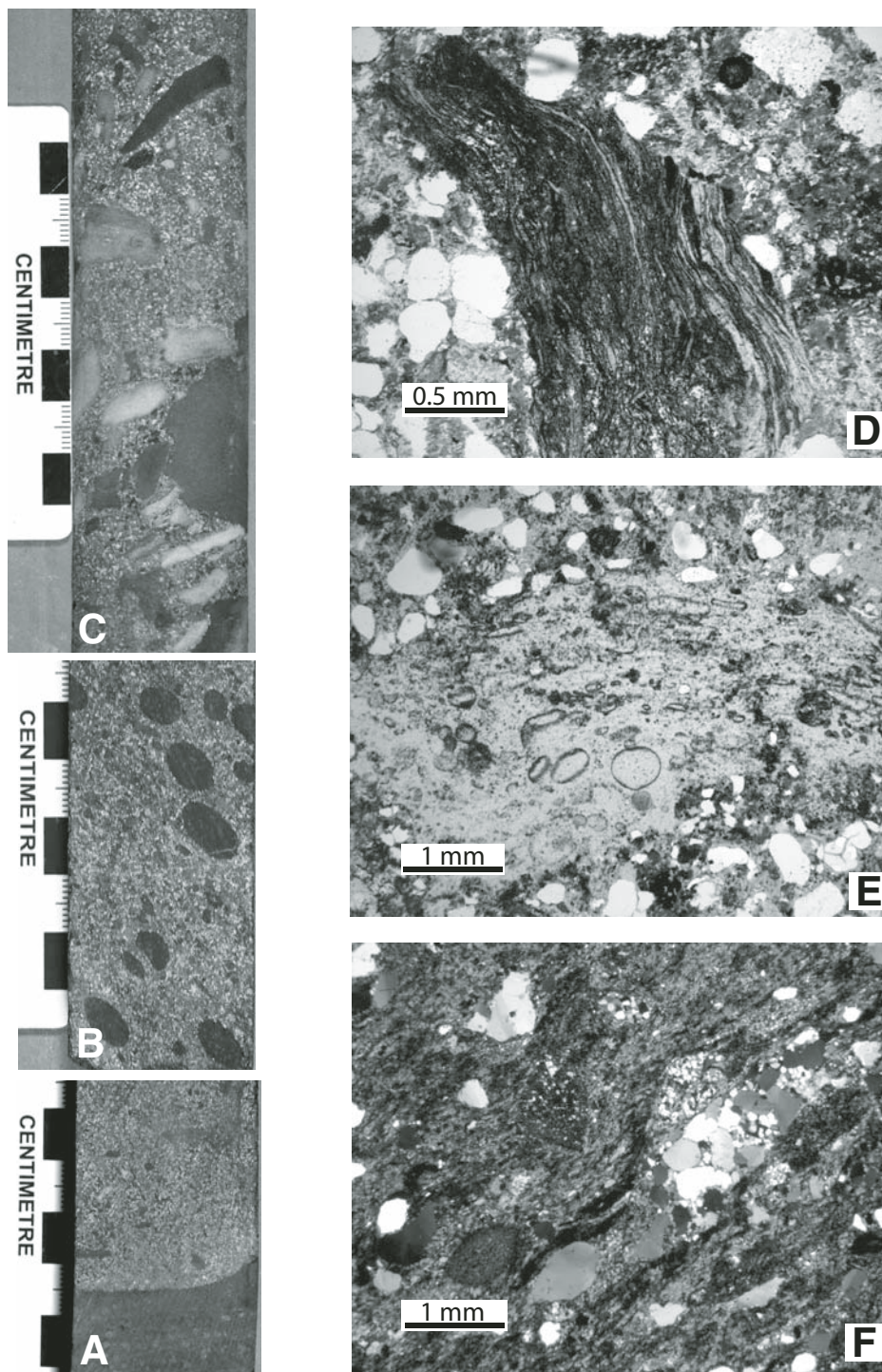


Figure 10. Marquette Range locality. (A–C) Segments of drill core containing complete stratigraphic section of the Sudbury layer. (A) Basal contact of Sudbury impact layer with chloritic, carbonaceous slate. Basal unit is quartz-rich graywacke containing abundant intraclasts of underlying bed. (B) Lapilli-bearing middle unit. (C) Conglomeratic bed containing clasts of chert (lighter clasts) and argillite (darker clasts). (D) Pumicelike fragment in graywacke. (E) Fragment of altered vesicular glass showing gradation from spherical to flattened vesicles. (F) Possible altered glass spherules (fine-grained dark particles in graywacke matrix). Note also small clast of orthoquartzite (right of center).

ranges from a few percent to as much as 50%. These larger clasts are dominantly chert with lesser amounts of black mudstone. Accretionary lapilli are abundant in an ~0.5-m-thick bed ~10 m above the base (Fig. 10B). Particles of altered glass from 1 to 10 mm in long dimension are abundant in some beds, particularly near the middle of the layer. Although many are largely recrystallized to biotite, some retain primary textures showing flow-banded or flattened pumicelike textures and relict vesicles (Figs. 10D and 10E). There are also sparse millimeter-scale spherical grains consisting of fine-grained chlorite and biotite that may be recrystallized relict glass spherules (Fig. 10F). The upper 2 m of the Sudbury layer are made up of graywacke that lacks clasts larger than sand size, but, like the lower units, this interval contains particles of altered glass.

Because the Sudbury impact layer here is underlain by evenly laminated marine strata, deposition of the impact material must have involved considerable interaction with seawater. However, the abundance of chert clasts and rounded quartz grains suggests erosion and incorporation of relatively local material. The incorporation of un lithified intraclasts of mudstone in the basal beds of the layer indicates that the immediately underlying sediments were eroded and incorporated into the impact layer. This could be accomplished either by vigorous erosion by a ground surge energetic enough to scour the sea bottom beneath a considerable depth of water, or, perhaps more likely, by turbidity flows generated by collapse and mobilization of nearby sediment accumulations by impact-induced earthquakes. The graywacke-like lithology of much of the impact layer here is consistent with deposition from turbidity flows, although we did not observe a consistent graded relationship internal to the impact layer.

Iron River–Crystal Falls District

The Iron River–Crystal Falls district (Fig. 1) is a former iron mining district covering ~800 km². It is a complexly deformed synclinal structure containing the Riverton Iron Formation and enclosing sedimentary strata (James *et al.*, 1968). We found indications that a distinctive breccia unit, part of the Hiawatha Graywacke, is a Sudbury-related breccia. The breccia contains a suite of sparse but widespread (generally 1–5 grains per thin section) quartz grains that have relict PDFs (one and rarely two sets) as well as shards and spherules of altered devitrified glass.

The Hiawatha Graywacke was described as long ago as 1899 (Clements and Smyth, 1899) and was mapped and described in detail during an extensive USGS study of the district in the

1940s and 1950s (James et al., 1968). Based on James' description, the basal breccia unit of the Hiawatha Graywacke is widespread but not ubiquitous in the district and was observed in numerous underground iron mines and related exploration drill holes as well as in rare outcrops. The breccia consists of angular fragments of chert set in a matrix of dark gray graywacke (Fig. 11A). Fragments are commonly several centimeters long, but, locally, slabs are as much as 0.7 m long. The chert is clearly derived from the underlying iron formation, and although siderite is not common in the fragments, it is abundant in the matrix. All gradations can be found from rocks consisting dominantly of chert debris to normal graywacke containing scattered chert fragments, or to graywacke containing none. The typical thickness of the Hiawatha Graywacke ranges up to ~20 m but has been interpreted to be as much as 150 m locally (James et al., 1968).

A disconformable contact between the breccia and underlying Riverton Iron Formation, at least locally, was well documented by James et al. (1968), who interpreted the breccia to have formed by submarine slumping of partly consolidated iron formation within a tectonically active basin. In the eastern part of the area (Crystal Falls), the Hiawatha Graywacke is entirely breccia. In the west (Iron River), the breccia appears to grade upward into graywacke and slate that make up the bulk of the Hiawatha unit. The top of the impact-influenced rocks is not well constrained in the Iron River area. We tentatively interpret the entire Hiawatha Graywacke to be related to the impact event. The Hiawatha represents an interval of coarse clastic sedimentation within an otherwise low-energy environment of chemical and fine-grained clastic sediments, suggesting that the entire high-energy sedimentation record may be a short interval of energetic deposition triggered by the Sudbury impact.

In our re-examination of the Hiawatha breccia, we concentrated on a set of drill holes near the Hiawatha Mine (abandoned), near Iron River, Michigan, and a single drill hole and a set of outcrops near Crystal Falls, Michigan (Fig. 1). We also reexamined an extensive set of thin sections from earlier USGS studies in the district in the 1940s. Pervasive oxidation related to the paleoweathering responsible for the iron ores of the district hinders some aspects of petrographic examination because of the nearly ubiquitous presence of secondary iron oxides. Also, secondary replacement by carbonate minerals is widespread and obliterates much of the primary textures in some samples. Nevertheless, several significant aspects of the breccias are indicative of impact-related deposition. In addition to

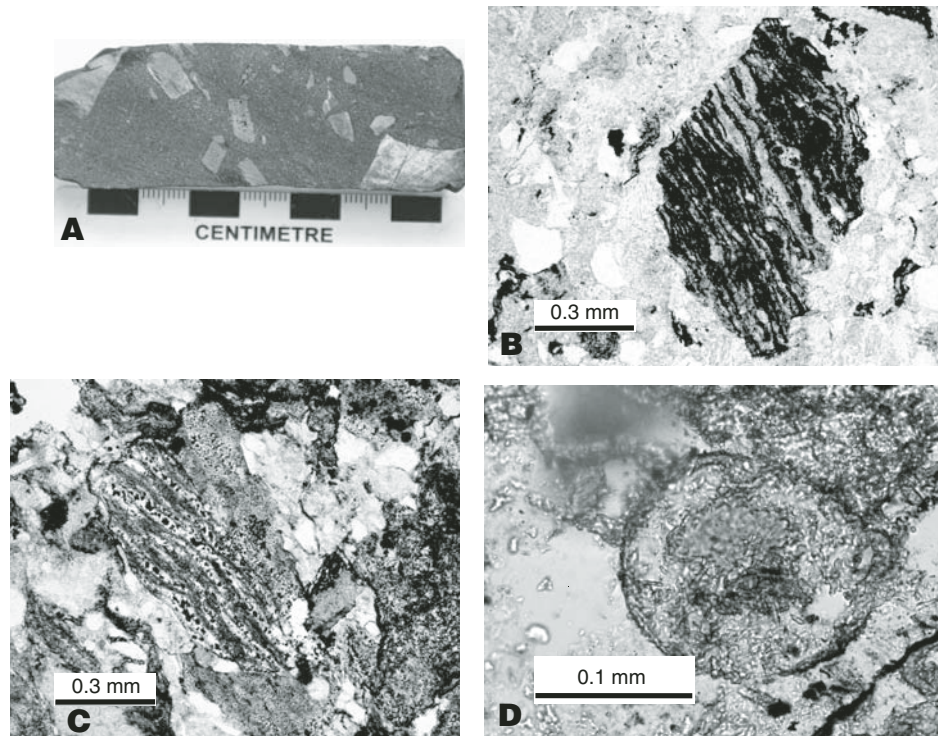


Figure 11. Hiawatha locality, Iron River–Crystal Falls district. (A) Section of drill core showing matrix-supported breccia of angular chert fragments in clastic matrix. (B–C) Particles of altered devitrified glass in clastic-quartz-rich matrix. (D) Spherule consisting of chlorite, quartz, and carbonate mineral. All photomicrographs are in plane polarized light.

the widely distributed shock-metamorphosed quartz grains, an important component of many samples is altered devitrified glass particles that constitute from a few percent to more than 50% of the breccia matrix (Figs. 11B and 11C).

Most altered glass particles range from one to a few millimeters in diameter, but particles up to 2 cm occur. Internally, the particles are very fine-grained and highly clouded with secondary opaque minerals, but generally they show varying degrees of flattening of original textures. Relict submillimeter-scale spherical particles are also present (Fig. 11D). Some show relict internal compositional zones and may have been spheres of impact-generated melt.

The Hiawatha Graywacke appears to be a deep-water deposit in a sequence of evenly bedded, commonly reduced sediments. It is also an anomalous high-energy deposit within an otherwise very low-energy environment. The chert breccias were originally interpreted to be a submarine debris flow generated by a strong earthquake (James et al., 1968). We concur with this interpretation but expand it to propose that the slump was triggered by the powerful earthquake(s) generated by the Sudbury impact. The seemingly ubiquitous incorporation of shocked quartz grains and impact glass particles

within the breccia indicates that the debris flow was active when ejecta arrived in the area and that the ejecta material was incorporated into the active flow.

Gogebic Iron Range

The search for the Sudbury layer in the Gogebic iron range is hampered by a nearly complete lack of bedrock exposures along the appropriate stratigraphic horizon. The range has been intensively mined for iron ore, mostly from underground mines now long-abandoned, so mine workings provide little help in the search. Exploration drill holes are also sparsely preserved, but they do provide a small amount of material, some of which contains rock types that may be genetically related to the Sudbury impact. Because the Gogebic Range lies between aforementioned ejecta-bearing localities to the east and localities in the Mesabi and Gunflint Ranges to the west, it seems likely that the Sudbury layer does occur in strata of the range that are temporally equivalent to those other localities. Next, we present descriptions of two localities at which unusual rock types, possibly impact related, occur within the stratigraphic interval in which the Sudbury layer would be

expected. We did not identify shock metamorphic features at either locality, so the evidence of a link to the Sudbury impact is circumstantial.

Puritan

The suspected Sudbury impact layer at the Puritan locality is known only from thin sections contained in an archive of work performed in the 1960s and described in a later report (Schmidt, 1980). Schmidt used two cores from holes drilled from the 24th underground level of the Puritan iron mine to define details of the stratigraphy of the lower part of the Tyler Formation (equivalent to the Michigamme Formation to the east), which is otherwise nearly totally concealed by glacial deposits throughout the region. We were unable to relocate the drill core or samples of it, and the mine is long-abandoned, so our knowledge is limited to examination of these few thin sections and Schmidt's generalized description. Schmidt described the layer as "lithic tuff containing fragments 2–3 mm in diameter" (Schmidt, 1980, p. 73) but gave no other details.

According to Schmidt (1980), in a stratigraphic section of the lower part of the Tyler Formation, this "lithic tuff" layer is 100 m above the contact of the Tyler with the underlying Ironwood Iron Formation. The Tyler Formation below the lithic tuff consists of a few meters of basal conglomerate that grades up into argillite and siltstone. The lithic tuff layer occurs within a unit of dark-red-brown argillite. The 45 m of section immediately above the lithic tuff layer contain ~20 m of iron formation of various lithologies, mostly faintly bedded carbonate iron formation interbedded with ferruginous argillite. This unit is overlain by pyritic black slate that eventually passes upward into a thick sequence of turbidites.

Schmidt's identification of this unit as "lithic tuff" is consistent with its content of fragments of altered devitrified glass with a wide variety of compositions. Our reexamination of these few thin sections found numerous features similar to those in well-documented Sudbury layer occurrences to the east, described previously. Metamorphic grade in this area is exceptionally low, so original textures are remarkably well preserved. Compared to most other localities, the Puritan material is exceptionally rich in glass particles of multiple compositions. Spheres and shards of altered devitrified vesicular glass (Fig. 12A) and quenched glass particles with crystallites of plagioclase (Fig. 12B) are common. Many glass particles are aphanitic and appear optically isotropic (Fig. 12D), whereas others are well recrystallized to rosettes of chlorite. Many are highly vesicular. Many glass fragments have overgrowths of very fine-

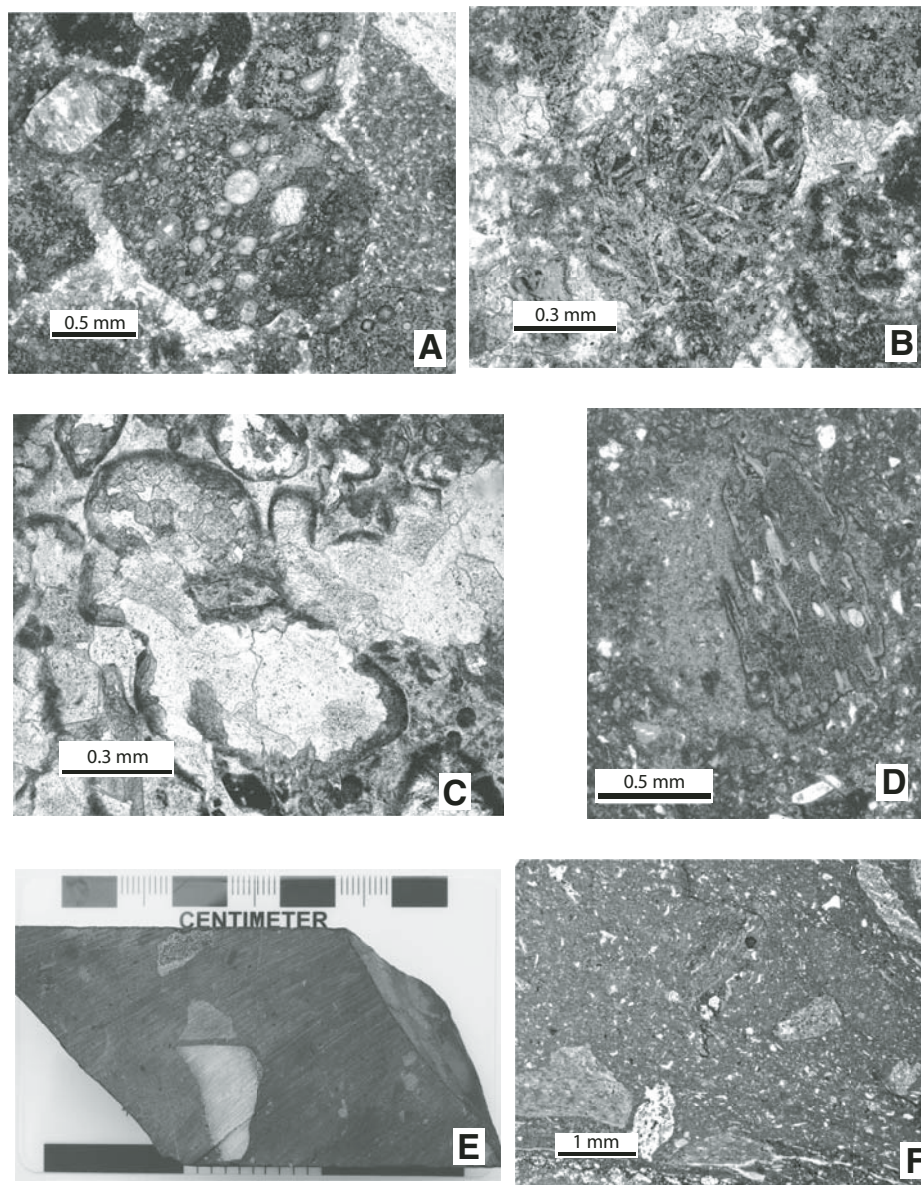


Figure 12. Photomicrographs of Gogebic Range localities. (A–D) Puritan locality. (E–F) Wakefield locality. (A) Sphere of highly vesicular altered glass. Vesicles are filled with chlorite. (B) Sphere containing numerous small plagioclase laths, possibly quenched melt droplet. (C) Spherules largely replaced by coarse quartz and carbonate. Possible broken bubble walls. (D) Delicately preserved vesicular glass particle encased in accreted fine-grained dust. (E) Drill core of breccia at Wakefield containing angular fragments of oolitic jasper (upper center) and gray and white chert. (F) Photomicrograph of breccia matrix showing variety of lithic fragments in fine matrix of chlorite and carbonate. All photomicrographs are in plane polarized light.

grained particles (Fig. 12D), apparently material accreted during transport. Possible altered glass spherules (Fig. 12C) also occur and are replaced by combinations of quartz, chlorite, and carbonate minerals. Quartz grains are extremely sparse, being generally less than 1% of the rock. Probable accretionary lapilli occur in one of the thin sections.

The Puritan section, if impact related, appears to be nearly entirely ejecta. It differs from previously described occurrences in having no clearly locally derived component. It is similar in that regard to some of the distal ejecta described in the Gunflint and Mesabi Ranges (Addison *et al.*, 2005). The Puritan locality may be the only representative in our suite of ejecta

sites that is sufficiently distal from the impact site to be composed nearly entirely of airborne ejecta that was deposited into a relatively quiet marine setting.

Wakefield

Two drill cores from near Wakefield, Michigan, contain an unusual breccia composed of fragments of iron formation and chert in a matrix of chlorite and carbonate (Fig. 12E). It is notable for large fragments, as much as ~10 cm long in drill core, of oolitic jasper. In detail, the matrix contains abundant small rock fragments with composition similar to surrounding structureless matrix (Fig. 12F). The breccia is devoid of bedding. One drill core provides a complete stratigraphic section of the breccia bed totaling ~35 m. The breccia lies on the Ironwood Iron Formation and is overlain by laminated ferruginous argillite, probably basal beds of the Tyler Formation. The breccia contains an ~3-m-thick unit of laminated ferruginous argillite near the middle, indicating that the entire breccia unit contains evidence of two episodes of breccia deposition.

Iron assays appended to the original mining company drill log show that the breccia is exceptionally iron-rich. Iron grades are 25–30 wt% Fe, identical to the underlying Ironwood Iron Formation. Clastic quartz grains are extremely rare. This suggests that the breccia is largely a disaggregated mass of the Ironwood that was broken and fluidized before complete lithification, perhaps by seismically generated submarine slumping, similar to the Hiawatha Graywacke. The Wakefield breccia bears similarities to recently discovered breccia at the Magnetic Rock locality in Minnesota (Jirsa et al., 2008). That breccia appears to be composed nearly entirely of disaggregated Gunflint Iron Formation, on which it lies. At the Magnetic Rock locality, the Gunflint breccia is overlain by a thin (<1 m) unit of ejecta containing accretionary lapilli, altered glass shards, and sparse shock metamorphic features in quartz grains. Such an ejecta bed is not evident in the Wakefield section.

Geochemistry of the Impact Layer

To further characterize the Sudbury impact layer and allow comparison with ejecta materials at Sudbury, we analyzed selected samples from several northern Michigan locations for major and trace elements, including rare earth elements (REE). Also, because in the past some of the occurrences of the impact layer have been interpreted as volcanic in origin, we compared the composition of the impact-layer samples with felsic volcanic rocks from both below

(Hemlock Formation rhyolite; Schneider et al., 2002) and above (Virginia Formation felsic ash; Hemming et al., 1995) the layer.

The geochemical analyses of the impact-layer samples are presented in Table 2. Analytical methods are discussed in the Appendix. Samples include: (1) accretionary lapilli from the Connors Creek (sample 2) and L'Anse (sample 10) locations; (2) black, altered devitrified glass fragments from the L'Anse drill core (samples 11–12); (3) altered vitric-rich breccia from the Marquette Range drill core (samples 3–6); and (4) matrix from chert breccia in the Iron River–Crystal Falls district (samples 7–9). In addition, accretionary lapilli from the Thunder Bay location described in Addison et al. (2005) were analyzed for comparison (sample 1). The samples all have high loss on ignition (LOI = 2.12–18.78), and two accretionary lapilli samples have particularly high CO₂ content (Table 2). As a result, major elements were recalculated to 100% volatile free; the two accretionary lapilli samples with high CO₂ were recalculated to 100% volatile free after removal of carbonate (dolomite + calcite) (Table 2).

Major Elements

The major-element compositions of most of the impact-layer samples, particularly the accretionary lapilli and altered vitric-rich breccia samples, are similar (Fig. 13); however, most element concentrations tend to vary inversely with increasing SiO₂ content. Sample 7, matrix from the chert breccia at the base of the Hiawatha Graywacke in the Iron River–Crystal Falls district, has low SiO₂ (60.28 wt%) and high FeO (24.2 wt%) and P₂O₅ (1.43 wt%) contents, which probably reflect the presence of a significant component of the Riverton Iron Formation that directly underlies the breccia (James et al., 1968). The two black, altered glass fragments from the L'Anse drill core (samples 11 and 12) have very high SiO₂ contents (~95 wt%) and concomitantly low concentrations of all other elements (Table 2); the high SiO₂ may reflect significant secondary silicification. The other samples have similar compositions over a range of SiO₂ from ~71 to 84 wt% (Fig. 13). A distinctive characteristic of all the impact-layer samples is low CaO and Na₂O contents (Fig. 13). Compared to the North American Shale Composite (NASC; Gromet et al., 1984) and Post-Archean Average Australian Shale (PAAS; Taylor and McLennan, 1985), two compositions often taken to approximate average upper continental crust, the impact-layer samples mostly have higher SiO₂, Fe₂O₃ + FeO, and MgO, and lower Al₂O₃, CaO, Na₂O, K₂O, and TiO₂ contents (Fig. 10).

Trace Elements

The trace-element concentrations of the impact-layer samples tend to decrease in abundance with increasing SiO₂ content (Table 2). However, the samples mostly have similar ratios of relatively immobile trace elements (e.g., Zr/TiO₂, Zr/Nb, Th/Hf, Sc/Yb; see Table 2). Ratios tend to be most variable in the highest SiO₂ samples, probably because many trace-element abundances are often near their detection limits (see Appendix). Although the absolute concentrations of the REEs are variable, chondrite-normalized patterns are similar for all impact-layer samples (Fig. 14). All the patterns are light (L) REE enriched and slightly heavy (H) REE depleted (chondrite-normalized La/Yb ratio [La/Yb]_n ranging from ~8 to 20) and have no to moderately negative Eu anomalies (Eu/Eu* = 0.65–0.94). Compared to the NASC and PAAS shale composites, the impact-layer samples have similar LREE-enriched patterns but generally lower overall REE concentrations, mostly smaller negative Eu anomalies, and slightly more depleted HREE (Figs. 14A and 14B).

Extended chondrite-normalized trace-element patterns are similar for most of the impact-layer samples and are similar to the NASC and PAAS shale composites, except for more prominent negative anomalies of Ba, Sr, and Ti, and no to only slightly negative P anomalies (Figs. 14C and 14D). Two samples from the Iron River–Crystal Falls district have patterns distinct from the other samples (Fig. 14D). Sample 7, which is enriched in Fe and P, has a large positive P anomaly, depletions of Ba, Rb, and K, and a large positive Th anomaly (Fig. 14D). In contrast, sample 9 is relatively enriched in Ba, Rb, and K, and has a prominent negative Th anomaly. The variable Th anomalies for these two samples probably reflect secondary mobility of Rb and K, since their Th content is similar. Both of these samples also have distinctly negative Zr-Hf anomalies (Fig. 11D). All of the impact-layer samples have similar crustal K/Rb ratios (mean 294) except for sample 7 (K/Rb = 28), but they have relatively high U/Th ratios (0.46–7.43; mean = 1.94 versus 0.26 for average upper crust; Taylor and McLennan, 1985), which probably reflect secondary redistribution of uranium (Lev et al., 2000). In addition, accretionary lapilli samples 1 and 10 are anomalously enriched in Y, V, and Cr (Table 2). These enrichments may reflect precipitation from seawater under appropriate redox conditions (Tribouillard et al., 2006) and/or prior enrichment of source materials. Sample 6, the most iron-rich sample, also is enriched in V.

TABLE 2. GEOCHEMISTRY OF THE SUDBURY IMPACT LAYER SAMPLES (Cont.)

Location	1	2	3	4	5	6	7	8	9	10	11	12
Sample	Gunflint GF Lapilli	Connors Creek CC Lapilli	Marquette 27-7-128	Marquette 27-7-126	Marquette 27-7-59	Marquette 27-7-11	Iron River CF-01	Iron River CF-02	Iron River DL92-2-449	L'Anse BIC-9ULap	L'Anse BIC-8	L'Anse BIC-7L
K/Rb	310	293	293	322	293	555	28	166	261	202	307	257
U/Th	1.37	0.43	0.46	1.03	0.77	0.58	0.71	0.60	2.10	2.73	7.43	6.68
Zr/TiO ₂	370	296	331	264	331	374	225	387	224	332	288	520
Sc/Yb	7.40	7.34	6.85	9.64	8.16	9.24	9.49	4.09	7.76	6.85	5.36	3.66
Zr/Nb	17.1	11.9	12.4	13.8	10.5	9.9	10.3	9.9	9.6	11.7	12.1	20.0
Th/Hf	1.40	2.19	2.23	2.38	2.10	2.21	2.94	2.19	2.21	2.18	2.33	1.80
[La/Yb] _n [†]	10.42	19.76	13.10	14.21	17.94	7.97	15.67	9.17	18.69	20.06	11.85	8.31

Note: All trace elements are in ppm except Au (which is in ppb). LOI—loss on ignition.

*Recalculated to 100% volatile free (samples 1 and 2 recalculated volatile free after removal of secondary carbonate [dolomite + calcite]).

[†]Chondrite-normalized ratio (chondrite values are from Nakamura, 1974).

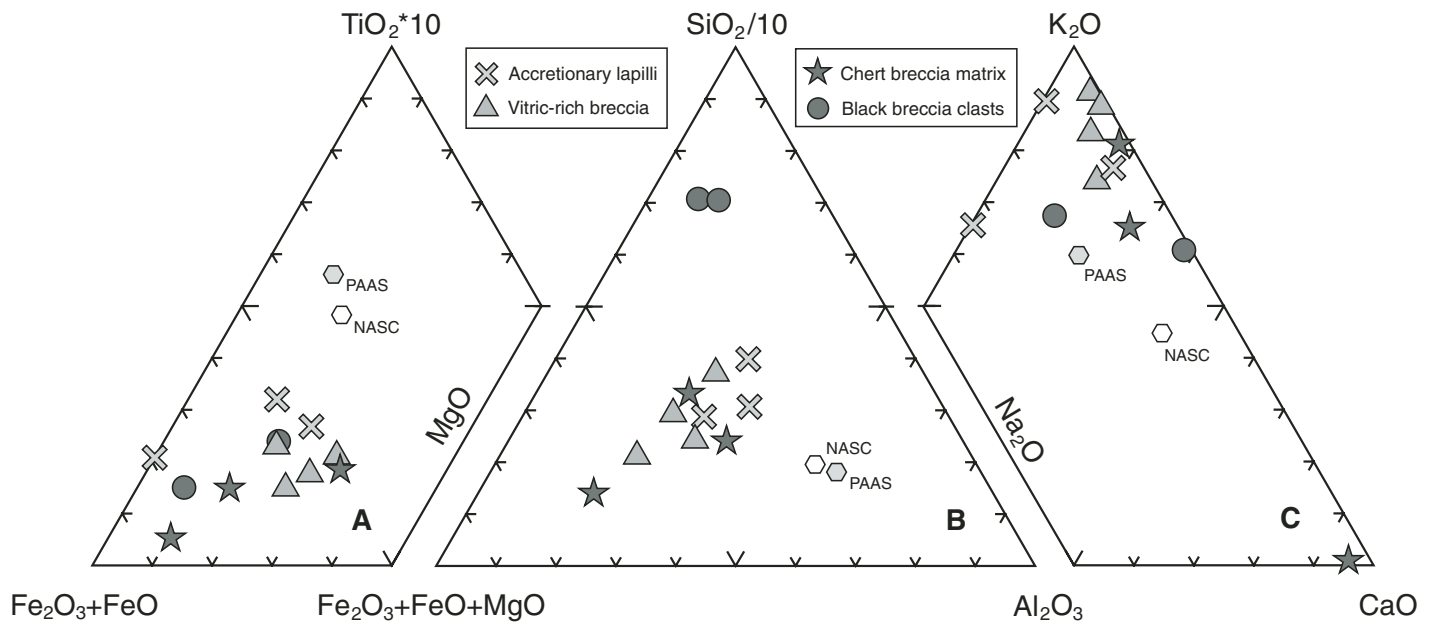


Figure 13. Major-element compositions of the samples analyzed from the Sudbury impact layer plotted in ternary diagrams. (A) Relative $(\text{Fe}_2\text{O}_3 + \text{FeO})\text{-TiO}_2 (\times 10)\text{-MgO}$; (B) $(\text{Fe}_2\text{O}_3 + \text{FeO} + \text{MgO})\text{-SiO}_2$ (divided by 10) $\text{-Al}_2\text{O}_3$; (C) $\text{Na}_2\text{O-K}_2\text{O-CaO}$. North American Shale Composite (NASC; Gromet et al., 1984) and Post-Archean Average Australian Shale (PAAS; Taylor and McLennan, 1985) are plotted for comparison.

Discussion of the Geochemical Data

As described already, the Sudbury impact layer is composed of three main components: (1) ejecta material likely derived directly from the impact site at Sudbury in the form of particulate matter (shocked quartz and other clastic material) and impact melt (altered melt spherules and shards); (2) material derived locally from the substrate to the impact layer, mostly consisting of chert, iron formation, and shale/greywacke; and (3) a secondary alteration component mostly consisting of carbonate or silica replacement. Given the potential for compositional variability resulting from these three sources, it is noteworthy that most of the impact-layer samples, although varying widely in SiO_2 content, have overall similar compositional characteristics, particularly with respect to their trace elements (Fig. 14).

Thus, increasing SiO_2 , attributable to increased detrital quartz (both shocked and unshocked) and/or secondary silicification, mainly serves to dilute abundances. Local substrate contributions are perhaps most clearly evident in the compositions of the samples from the Iron River-Crystal Falls district, whose relatively high SiO_2 and iron contents may reflect local additions of detrital quartz and iron formation. However, the overall similarity in composition of the accretionary lapilli and vitric-rich breccia samples, both of which are likely composed of material derived largely from the ejecta plume itself, suggests that the composition of these materials primarily reflects that of the source materials at the site of impact.

One of the distinctive compositional characteristics of the impact-layer samples is their low CaO , Na_2O , Sr , and Ba contents (Figs. 13 and 14). The cations Ca , Na , and Sr are dominantly

hosted by plagioclase in crustal rocks, while Ba is dominantly in feldspar and biotite. Their low abundance in the impact-layer samples thus suggests that they either have low feldspar content (i.e., composition is a primary characteristic of the source material) or any contained feldspar has been strongly altered and the cations removed in solution (i.e., composition is a secondary feature unrelated to the source). Support for the first alternative is provided by the generally low to absent content of plagioclase grains or feldspar-bearing rock fragments in the samples (see previous descriptions), although if they were very fine grained, they may not be readily detected. Secondary alteration is a possibility and is observed to have variably affected felsic volcanic rocks both above and below the impact layer (see following discussion). However, unaltered feldspar is commonly observed in clastic metasediments (arkosic greywackes) overlying

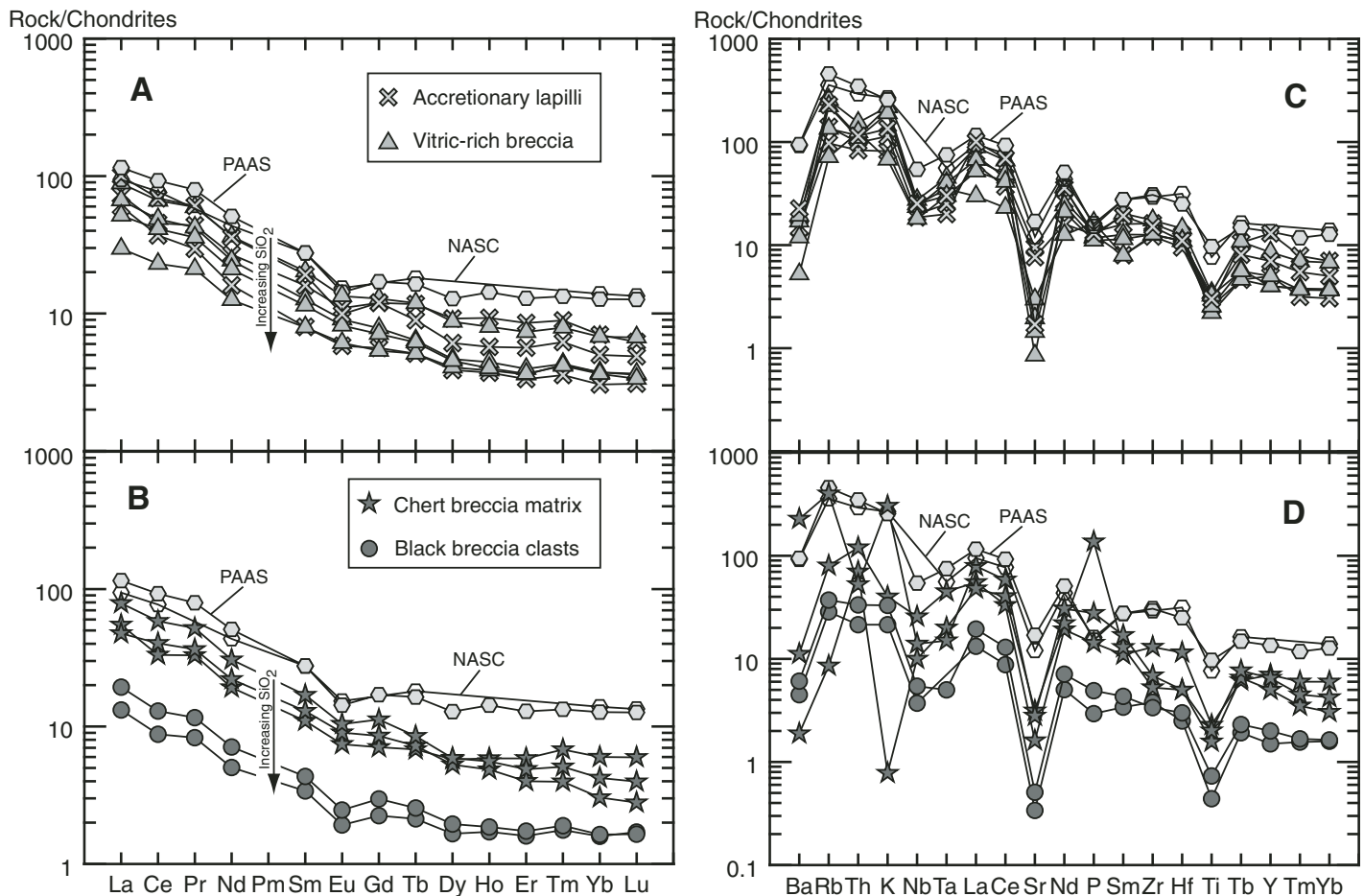


Figure 14. Chondrite-normalized rare earth element (REE) (A–B) and extended trace-element (C–D) patterns for the Sudbury impact layer samples. Note that the chondrite-normalized REE and extended trace-element patterns are similar for most of the samples, while sample abundances tend to decrease with increasing SiO₂ content. North American Shale Composite (NASC; Gromet et al., 1984) and Post-Archean Average Australian Shale (PAAS; Taylor and McLennan, 1985) are plotted for comparison. Normalizing values for REEs in A and B are from Nakamura (1974), and normalizing values for extended trace elements in C and D are from Thompson et al. (1984).

the impact layer (Ojakangas, 1994), suggesting that widespread feldspar alteration has not occurred. In addition, secondary alteration tends to be variable in intensity, as is observed with the felsic volcanic rocks that underlie and overlie the impact layer, resulting in variable compositions. Thus, although secondary feldspar alteration cannot be fully discounted for the impact layer, we tentatively conclude that low plagioclase content is a primary feature and reflective of the source materials at the impact site.

Comparison with the Sudbury Onaping Formation and Implications for the Nature of the Ejecta Source Material

The Sudbury impact is inferred to have occurred in a shallow marine basin in the foreland of the Penokean orogen (Mungall et al., 2004). This is supported by the ubiquitous presence

of carbonaceous mudstone clasts in the upper kilometer of the fallback Onaping Formation (Bunch et al., 1999). The Onaping Formation is exposed in the central basin of the Sudbury structure and records the history of crater-fill emplacement and crater collapse related to the Sudbury impact event (Ames et al., 2002). The Onaping Formation is mostly composed of a lower suevitic fallback breccia containing 60–90 wt% devitrified glass and igneous lithic fragments (Sandcherry Member) overlain by a series of plume collapse units consisting of lenticular devitrified glass bubble-wall shards (~30%) and lithic clasts dominated by metasedimentary fragments (Dowling Member). Ames et al. (2002) showed that the units of the Onaping Formation are similar in composition, particularly with respect to relatively immobile trace elements (e.g., consistent Zr/TiO₂ ratios), and they are characterized by enrich-

ment of large ion lithophile elements (LILE) and LREEs, and prominent negative Nb and Ti anomalies. The andesitic composition of the least altered vitric fragments and aphanitic dikes (61.6 wt% SiO₂, 4.28 wt% MgO, 60 ppm Ni) is considered to be most representative of the bulk composition of the initial quenched impact melt and is consistent with a crustally derived melt (Ames et al., 2002).

The compositions of the northern Michigan impact-layer samples are compared with those of the Onaping Formation in Figures 15 and 16. For comparison, data are also included for rhyolites in the Hemlock Formation that are lateral equivalents of the Negaunee Iron Formation below the impact layer (Schneider et al., 2002) and for ash beds interlayered in the younger Virginia Formation in Minnesota (Hemming et al., 1995). The ash beds in the Virginia Formation are probably equivalent to previously dated ash beds in

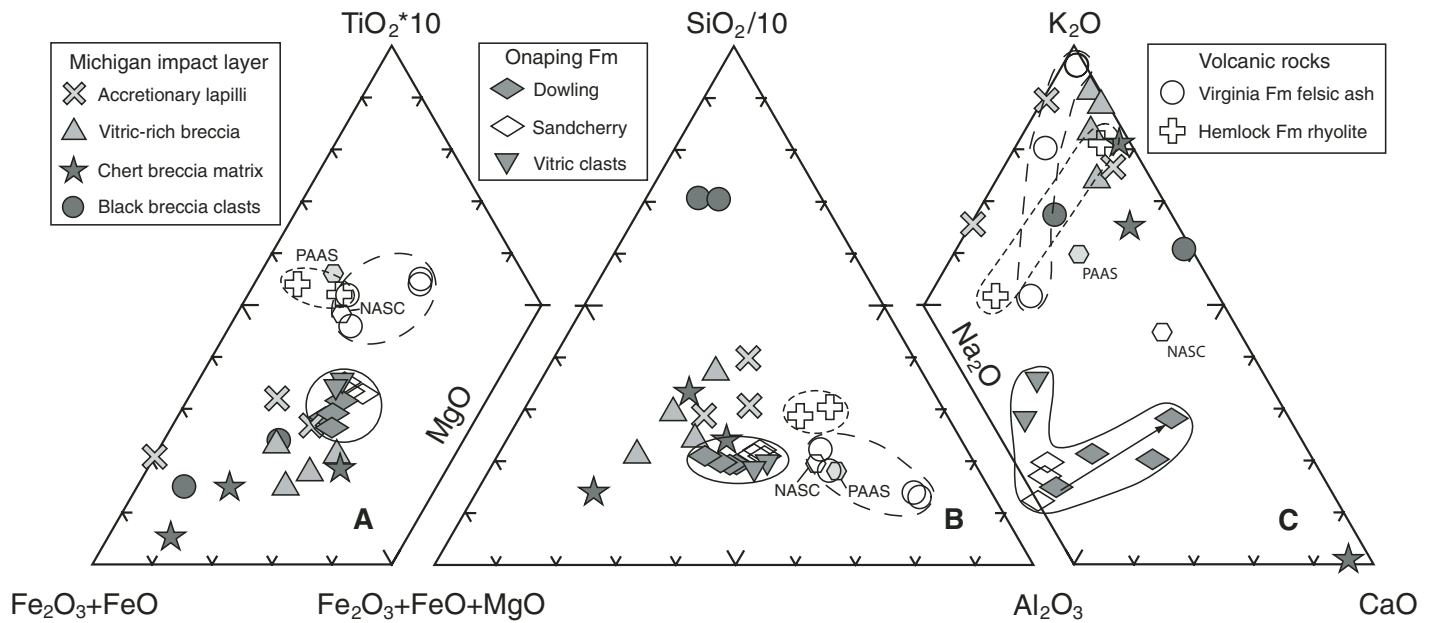


Figure 15. Ternary diagrams showing the major-element compositions of samples from the Sudbury impact layer, felsic ash samples from the Virginia Formation, Minnesota (Hemming et al., 1995), rhyolite from the Hemlock Formation, Michigan (Schneider et al., 2002), and units of the Onaping Formation, Sudbury, Ontario (Ames et al., 2002). (A) Relative $(\text{Fe}_2\text{O}_3 + \text{FeO})\text{-TiO}_2 (\times 10)\text{-MgO}$; (B) $(\text{Fe}_2\text{O}_3 + \text{FeO} + \text{MgO})\text{-SiO}_2$ (divided by 10) $\text{-Al}_2\text{O}_3$; (C) $\text{Na}_2\text{O-K}_2\text{O-CaO}$. Note: arrow in C shows trend of decreasing Na_2O and CaO from the lower to upper units of the Dowling Member of the Onaping Formation (Ames et al., 2002); see text for discussion. North American Shale Composite (NASC; Gromet et al., 1984) and Post-Archean Average Australian Shale (PAAS; Taylor and McLennan, 1985) are plotted for comparison.

the Rove Formation based on their similar chemistry (P. Fralick, 2008, personal commun.). With respect to major elements, the impact-layer samples have higher SiO_2 , $\text{Fe}_2\text{O}_3 + \text{FeO}$, and K_2O , similar to slightly higher MgO , and lower Al_2O_3 , TiO_2 , and especially CaO and Na_2O compared to the Onaping samples (Fig. 15). Compared to the volcanic rocks, the impact-layer samples are generally distinct from the ash samples (impact-layer samples have mostly higher SiO_2 , $\text{Fe}_2\text{O}_3 + \text{FeO}$, MgO , and lower TiO_2 contents, but both have very low CaO and Na_2O contents) and have higher $\text{Fe}_2\text{O}_3 + \text{FeO}$ and MgO and lower Al_2O_3 , CaO , and TiO_2 contents than the rhyolite (Fig. 15). It is important to note that, stratigraphically, the Onaping Formation shows an upward decrease in CaO and Na_2O abundances, trending toward the composition of the impact-layer samples (Fig. 15C).

The REE abundances of the impact-layer samples are significantly lower than those of either the ash or rhyolite samples; however, the abundances overlap those of the Onaping Formation and have similar chondrite-normalized patterns (Fig. 16A). Note that because of the dilution of trace-element abundances with increasing SiO_2 in the impact-layer samples, only two of the samples with the highest abundances are plotted for comparison. The extended chondrite-normalized trace-element patterns of the impact-

layer samples also show little similarity to the volcanic rocks, but they are very similar to those of the Onaping Formation, with the exception of more prominent negative Ba and Sr anomalies (Fig. 16B). The marked compositional differences between the impact-layer samples and the felsic volcanic rocks, particularly in trace elements, strongly suggest that the impact layer was not a product of such volcanism. However, their compositional similarity to the Onaping Formation supports the interpretation that the impact layer largely represents ejecta material derived from the Sudbury impact.

Mungall et al. (2004) suggested that the Sudbury impact melt was dominantly derived from the lower crust based on an observed enrichment in transition metals and on models of cratering dynamics and impact melting. Models of crater excavation in layered continental terranes composed of sedimentary sequences and underlying crystalline basement (Grieve and Cintala, 1992; Kring, 1995; Pierazzo et al., 1997, 1998; Kring, 2005) predict that debris from both the sedimentary and crystalline layers is ejected during crater formation, but that the bulk of the impact melting occurs at depth in the silicate basement. This is supported by studies of some Cenozoic impact craters in layered continental terranes, which show that melt and breccia clasts at the impact site are dominantly

derived from the deeper, crystalline basement in the crater, whereas at greater distances, the ejecta material is dominated by near-surface sedimentary lithologies (Kring, 2005). The brecciated, melted, and/or vaporized target rocks and impactor that were ejected from the crater at Sudbury are inferred to have partially collapsed back onto the melt sheet (the Sudbury igneous complex) to form the Onaping Formation. Mungall et al. (2004) also observed that the matrix of the Dowling Member is generally similar in composition to that of the contained carbonaceous metasedimentary clasts (e.g., both are characterized by low concentrations of Al_2O_3), and it could best be modeled by taking 30% glass shards (inferred lower-crustal impact melt) and 70% carbonaceous mudstone (upper crust) with a small added chondrite component to account for enriched platinum group element (PGE) content. We note, however, that the overall trace-element composition of the Middle Dowling Member modeled by Mungall et al. (2004) is also essentially identical to that of the least altered devitrified glass shards interpreted as representative of the Sudbury impact melt (Ames et al., 2002). The overall results of the crater impact modeling studies predict that the upper-crustal portion of the shock-melted target rock volume at Sudbury would be ejected away from the crater area and would

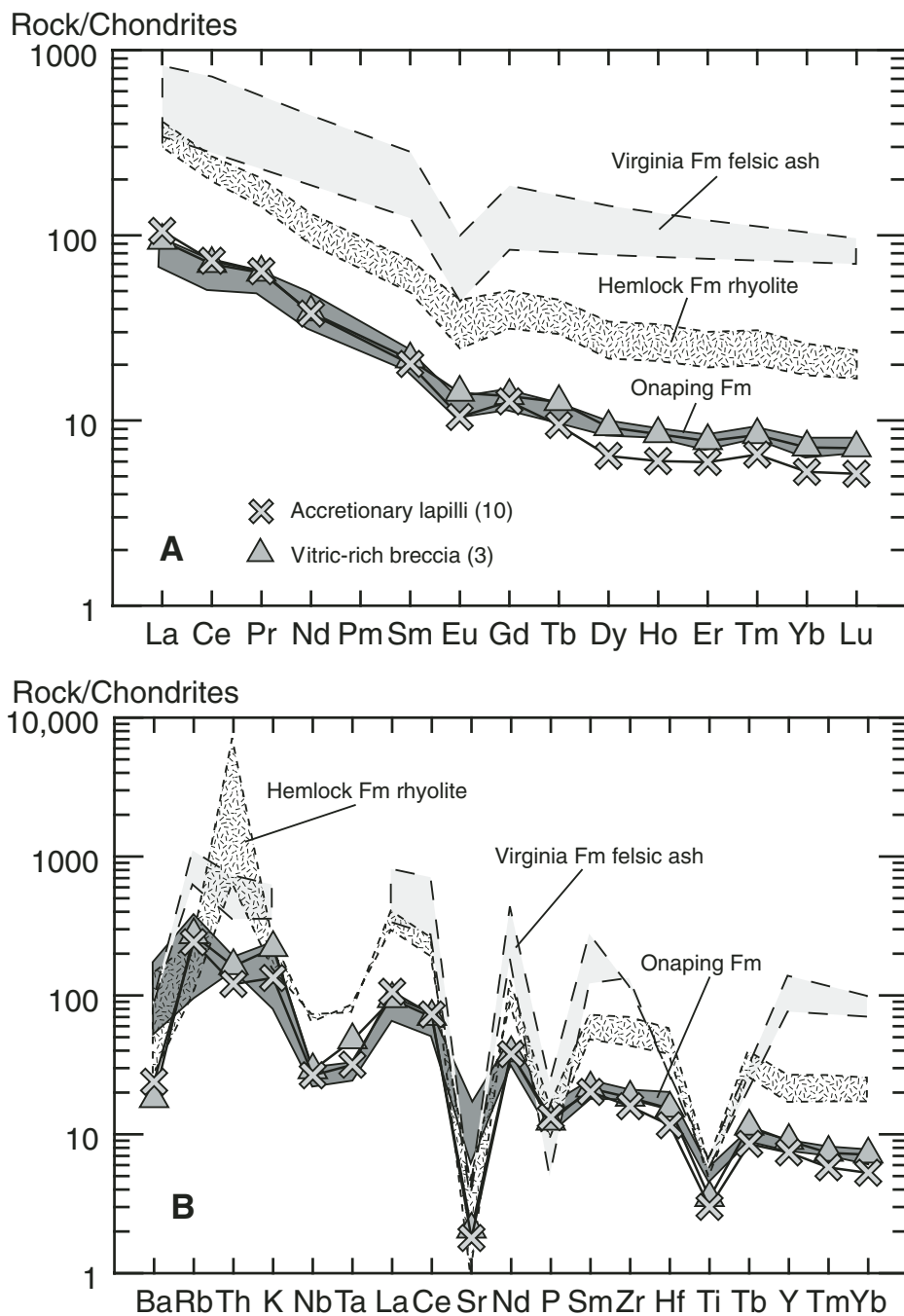


Figure 16. Chondrite-normalized rare earth element (REE) (A) and extended trace-element (B) patterns for accretionary lapilli (sample 10) and vitric-rich breccia (sample 3) samples from the Sudbury impact layer compared to felsic ash samples from the Virginia Formation, Minnesota (Hemming et al., 1995), rhyolite from the Hemlock Formation, Michigan (Schneider et al., 2002), and units of the Onaping Formation, Sudbury, Ontario (Ames et al., 2002). Note: (1) the overall similarity in trace-element composition between the impact-layer samples and units of the Onaping Formation, except for the greater depletion of Ba and Sr in the impact-layer samples, and (2) the dissimilarity between the impact-layer samples and the felsic volcanic rocks. Normalizing values are as in Figure 14.

produce lithological and chemical variations with radial distance from the crater, where deep basement components would dominate near the crater and upper-crustal sedimentary components would increasingly dominate farther from the crater center.

The general compositional similarity of the impact layer to that of the Dowling Member of the Onaping Formation at Sudbury is compatible with the prediction for a significant sedimentary component in the more distal ejecta. Although the Sudbury impact is inferred to have occurred in a shallow marine basin at ca. 1850 Ma, no sedimentary deposits of that age remain in the Sudbury area. However, it appears likely that at least some of the sedimentary equivalents of the Marquette Range Supergroup in Michigan extended into the Sudbury area prior to the impact event (Mungall et al., 2004). Carbonaceous clastic metasediments below iron formations in northern Michigan, particularly in the Iron River–Crystal Falls district, are characterized by an absence of detrital feldspar and low abundances of Ca, Na, Sr, and Ba (James et al., 1968; Schulz, 2007, personal commun.). This feature is characteristic of sediments derived from intensely weathered terranes (Nesbitt and Young, 1989), in which those components are selectively leached from weathering profiles during the breakdown of plagioclase (Nesbitt et al., 1980). In addition, overall trace-element patterns for the Michigan sedimentary rocks are similar to those of the Sudbury impact layer and the Onaping Formation (Fig. 17). Thus, sedimentary deposits likely derived from an intensely weathered source area like those present in Michigan also may have been present in the Sudbury area at the time of impact. This is further supported by the apparent decrease in CaO and Na₂O content upward in the Dowling Member (Fig. 15), which may reflect an increasing contribution of sedimentary material derived from a weathered terrane in the plume collapse deposits at Sudbury. These sedimentary units would have been a dominant component of the material ejected from and deposited distally to the crater center.

GEOLOGICAL SIGNIFICANCE OF THE SUDBURY IMPACT LAYER

The large ejecta field currently known in the Lake Superior region, as well as extensions of it likely to be found through continuing studies, has significance for expanded understanding of the Sudbury impact event itself and also for the sedimentology and tectonics of the Lake Superior iron ranges, one of the world's largest preserved accumulations of banded iron formation. The variety of lithologic assemblages and thicknesses

displayed by the Sudbury impact layer in Michigan suggests that multiple processes of transport and deposition are recorded and that their interplay with diverse depositional settings accounts for observed variations between sites. Although our studies to date have been limited to field and optical petrographic examination, supplemented by chemical analyses of a few key rock types, we present here a preliminary synthesis of our data based on our current level of knowledge of these deposits.

We propose that four different types of deposits may be present. Some localities probably record more than one type within their internal stratigraphy:

(1) Air-fall material from the ejecta plume was deposited with little or no disruption of underlying strata. The Puritan, Roland Lake, and L'Anse localities may be examples of this type of deposit. These relatively thin and glass-rich deposits appear to have little or no locally derived material within them and thus are the most representative examples of the material that made up the ejecta plume.

(2) Ballistic deposits formed from a high-velocity ejecta curtain and the consequent ground surge as the ejecta curtain swept across Earth's surface. These deposits are rich in clasts of chert as large as 1 m, derived from immediately underlying stratigraphic units, as well as abundant rounded grains of quartz and chert sand and lesser feldspar sand grains. The deposits contain variable amounts of glass particles and accretionary lapilli. The McClure locality and lower part of the Connors Creek deposit are likely examples of this type of deposit.

(3) Some deposits formed as the result of large tsunamis generated by the impact. If the impact at Sudbury occurred in a marine basin, large tsunami waves within the area of our study would seem inevitable. Bedded ejecta-bearing rocks reported by Pufahl et al. (2007) were interpreted to be, in large part, deposited by successive tsunami waves. In our observations, the upper part of the section at the Connors Creek locality appears to be the best candidate for this type of deposit, where planar-cross-bedded sands rich in ejecta may record high-energy reworking of underlying ground surge deposits. However, unequivocal evidence of such waves in the sedimentary record at other localities is lacking.

(4) Submarine debris flows were generated by the strong impact-related earthquake. Because the area of our study was a marine basin at the time of impact, it is likely that intense seismic shaking would have liquefied recently deposited unconsolidated sediments and generated submarine debris flows. The chert breccias of the Hiawatha Graywacke were interpreted

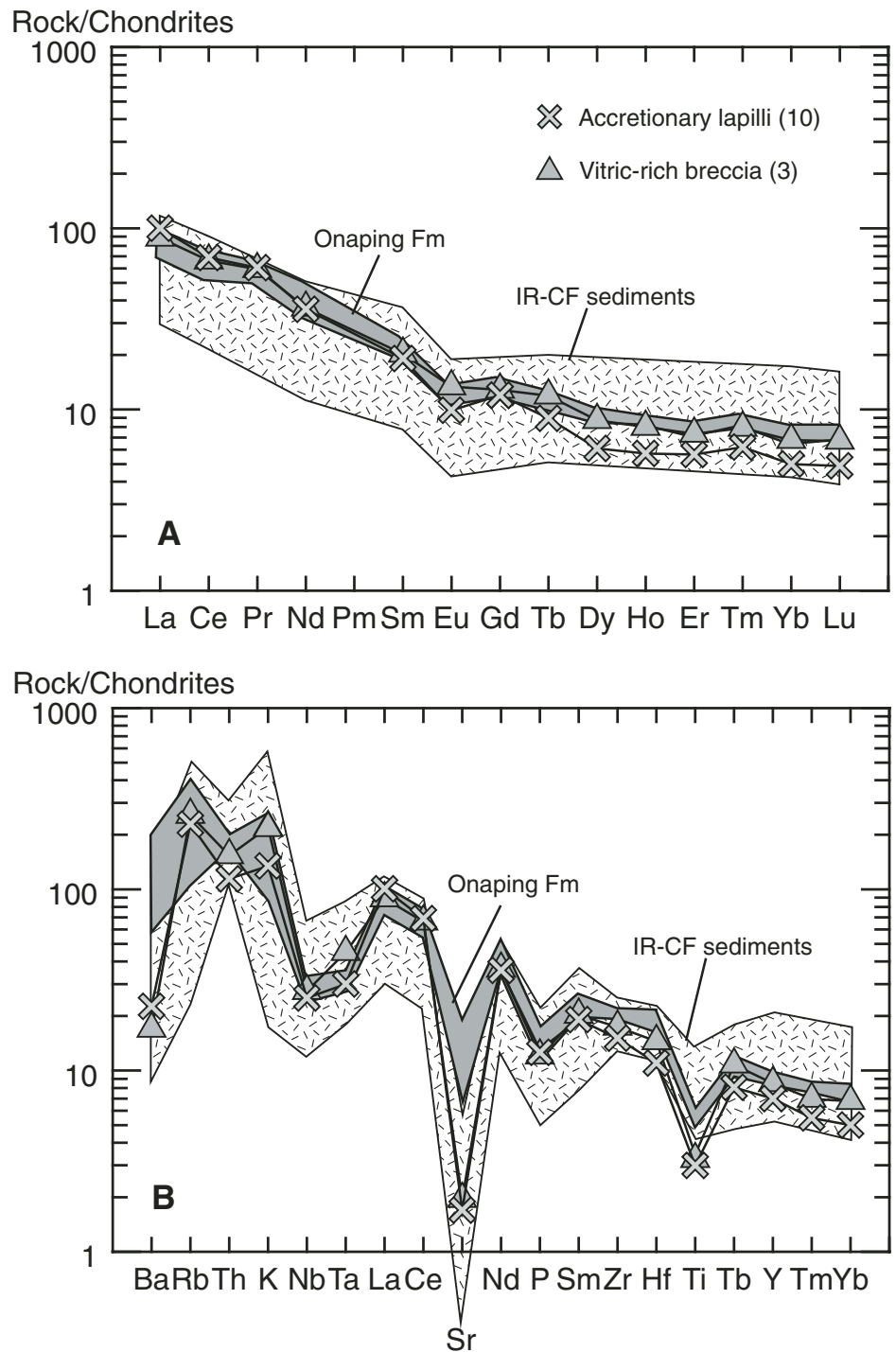


Figure 17. Chondrite-normalized rare earth element (REE) (A) and extended trace-element (B) patterns for accretionary lapilli (sample 10) and vitric-rich breccia (sample 3) samples from the Sudbury impact layer compared to units of the Onaping Formation (Ames et al., 2002) and clastic metasediments from the Iron River–Crystal Falls Basin, Michigan (field labeled IR-CF shales; Schulz, 2007, personal commun.). Note the similarity between the impact-layer samples and the sediments from the Iron River–Crystal Falls Basin, including prominent depletions of Ba and Sr for both. Normalizing values are as in Figure 14.

as seismogenic slump deposits by James *et al.* (1968). Our restudy of the Hiawatha Graywacke has shown that these slump deposits have incorporated large amounts of ejecta and thus were probably active immediately after impact as ejecta material was being deposited. The thick breccia bed at the Wakefield locality is also likely to be a similar submarine debris flow composed largely of the underlying Ironwood Iron Formation with intermixed ejecta glass.

Possible Environmental Effects of the Sudbury Impact

Giant impacts of the magnitude of Sudbury would be expected to produce regional, if not global, alterations of surficial conditions on Earth. We emphasize here one aspect of the Sudbury impact layer and suggest that it points to such changes across the Lake Superior region, and possibly beyond, in a manner not yet well understood. Figure 2 shows the regional stratigraphic position of the Sudbury impact layer, both at the sites described in this study and at sites in Minnesota and Ontario described in recent reports (Addison *et al.*, 2005; Jirsa *et al.*, 2008). At eight of the ten sites described here, and at all sites described in Minnesota and Ontario, the Sudbury impact layer lies at a stratigraphic horizon that marks a substantial change in the character of sediments being deposited. In particular, the impact layer lies on banded iron formation or variably ferruginous and dolomitic chert and is overlain by fine-grained clastic rocks, most commonly black shale. Because the impact layer was likely deposited over a time span of a few days at most, this regional change in sedimentation from ferruginous and cherty chemical sediments to fine-grained clastic sediments seems to have occurred essentially synchronously across the region precisely at the time of impact. Furthermore, the change in sedimentation occurred across a variable set of local conditions ranging from shallow-water, peritidal conditions to water depths in excess of wave base, and from shelf to foreland basin settings, suggesting that the change was not related to a gradual shift in local sedimentary facies, but was rather a regional event superimposed on all sedimentary facies simultaneously at the time of the Sudbury impact.

Exceptions to this observation occur in the Connors Creek locality, where shallow-water chert both overlies and underlies the impact layer. Sections from two nearby drill holes reported by Pufahl *et al.* (2007) also show one section where intertidal sandstone occurs both above and below the ejecta layer and another where the layer is underlain by intertidal sandstone and overlain by chert. The Sudbury layer

at the Puritan locality also does not mark a major lithologic boundary, according to the minimum data available for our study. As described by Schmidt (1980), the impact layer at the Puritan locality lies within a sequence of fine-grained clastic rocks. In spite of these exceptions, we suggest that the large number of instances throughout the Lake Superior region where the impact layer marks a major change in the style and character of sedimentation is more than coincidence and reflects a regionwide change in conditions created by the Sudbury impact.

In particular, the Sudbury impact layer most commonly marks the highest stratigraphic extent of banded iron formation (or equivalent lower-grade ferruginous and dolomitic chert) throughout the region. The major iron formations of Michigan's Iron River–Crystal Falls district, Gogebic iron range, and Marquette iron range, and the less ferruginous but regionally extensive chert-carbonate member of the Michigamme Formation in the Baraga and Dead River Basins are mostly immediately beneath the impact layer, but some (Marquette range) are as much as 500 m below it (Fig. 2). Likewise, the giant iron formations of the Mesabi Range in Minnesota and Gunflint Range in Minnesota and Ontario lie immediately beneath the impact layer (Addison *et al.*, 2005; Jirsa *et al.*, 2008). The Sudbury layer at the Gunflint localities in Ontario may be unique to the region in having been deposited subaerially inasmuch as there is some evidence of a previously unrecognized disconformity between the ejecta layer and the underlying Gunflint formation (Addison *et al.*, 2005; Burton and Fralick, 2007). However, ~100 km along strike to the west, in Minnesota, breccias in the impact layer contain highly contorted clasts of chert, indicating that the chert was still gelatinous at the time of impact, and a substantial hiatus is unlikely there. In the Mesabi, Iron River–Crystal Falls, and the Baraga–Dead River areas, there is no evidence of a disconformity between underlying iron formation and the impact layer (Lucente and Morey, 1983; James *et al.*, 1968; Puffett, 1974) other than the rip-up clasts of chert within the Sudbury impact layer, which require some erosion of underlying strata during emplacement of the impact layer itself. In all of those areas, it appears that iron formation or banded chert-carbonate rocks were being deposited at the moment of impact, but they did not continue into the postimpact sedimentary record.

Iron formations above the impact layer are not entirely absent, but they are rare, and their volume is many orders of magnitude less than the giant pre-impact iron formations for which the Lake Superior region is famous. Postimpact iron formations are restricted to: (1) the Puritan

locality in the Gogebic Range, where ~40 m of sediments immediately overlying the impact layer consist of argillite, including black shale, interlayered with ferruginous strata, mostly massive to faintly bedded ferruginous carbonate, and these are overlain by pyritic black argillite (Schmidt, 1980), and (2) the Bijiki Iron Formation, a locally significant unit in the western Marquette Range (Klasner and Cannon, 1978). The Bijiki consists of cherty silicate (probably carbonate prior to regional metamorphism) iron-formation. It is as much as 50 m thick, and it lies roughly 100 m above the impact layer within a sequence of chloritic, carbonaceous, and pyritic slate.

The initial mass of pre-impact banded iron formations in the Lake Superior region is estimated to be 10^{13} tons (James, 1983). Although deposition of some of these iron formations ceased before impact, apparently as a result of local tectonic uplifts (Negaunee Iron Formation in the Marquette Range for instance), the deposition of vast tonnages of banded iron formation in much of the Lake Superior region appears to have continued precisely until the Sudbury impact event, to be succeeded by fine clastic sediments immediately after the impact. These observed stratigraphic relationships, therefore, suggest that the Sudbury impact was in some manner responsible for termination of the major metallogenic episode of banded iron formation deposition in the Lake Superior region. The character of the Sudbury impact layer documented here indicates that, in addition to direct deposition of ejecta onto the ocean surface, major impact-induced tsunamis and submarine slump deposits were widespread in the area of our study. Any or all of these events may have ended the sedimentary conditions needed for deposition of iron formations across the Lake Superior region essentially instantaneously at the time of the Sudbury impact. The nature of the regional or global changes in environmental conditions that resulted in this dramatic shift in iron deposition is a fertile topic for future research.

SUMMARY

A layer of ejecta-bearing breccia, here called the Sudbury impact layer, was produced by the large impact event at Sudbury, Ontario, 1850 Ma ago. We have identified the ejecta layer at ten localities in northern Michigan. Together with other recent finds of the thin layer in Ontario and Minnesota, an extensive field of ejecta-bearing rocks now encompasses most of the iron ranges of the Lake Superior region at distances of ~500–850 km from Sudbury. Relict planar deformation features in quartz grains have been documented at five of the ten localities, and they

establish the connection between the breccia layer and a hypervelocity impact. In addition to shock metamorphosed quartz grains, other widespread impact-related features include accretionary lapilli and impact glass particles, now devitrified and variably recrystallized. Glass occurs both as angular fragments, commonly highly vesicular, and as millimeter-scale spherules. Independent age constraints show that the layer was deposited in the interval between ca. 1875 Ma and 1830 Ma, a time span that includes the 1850 Ma Sudbury impact.

At all of the sites in Michigan, the impact layer was deposited in a marine setting varying from shallow, partly peritidal, water depths to depths greater than wave base. Most of the layer is a hybrid rock that contains fragments of ejecta intermixed with detritus of more local provenance in varying proportions. Very coarse basal breccia containing large rip-up clasts from underlying rocks attests to the extreme high-energy conditions marking the deposition of this unit at many of the localities. We infer that four types of deposits are represented among the Michigan sites. These include: (1) direct air-fall deposits of mostly fine-grained glass particles and rock and mineral fragments, (2) ballistic deposits of high-velocity ejecta that resulted in ground surges, (3) submarine debris flows triggered by strong impact-induced earthquakes, and (4) deposits formed by large impact-generated tsunami waves. Deposit types 2, 3, and 4 are hybrid rocks composed of both ejecta and intra-clasts of relatively local pre-impact sediments.

The geochemistry of impact-layer samples, particularly the accretionary lapilli and vitric-rich breccias, which likely represent material directly from the primary ejecta plume, shows similar compositional characteristics, although concentrations tend to vary inversely with increasing SiO₂ content. The samples mostly have similar ratios of relatively immobile elements and similar extended chondrite-normalized trace-element patterns characterized by enriched LREE ([La/Yb]_n ~8–20), no to moderately negative Eu anomalies (Eu/Eu* = 0.65–0.94), and prominent negative anomalies for Ba, Sr, Nb, Ta, and Ti. The sample compositions generally overlap those of the NASC and PAAS shale composites, particularly in trace elements, but they have much lower concentrations of Ca, Na, Sr, and Ba. The latter compositional characteristic reflects very low feldspar content in the impact-layer samples, which we interpret as a primary feature related to the source materials at the Sudbury impact site, most likely clastic sediments derived from a highly weathered source area. Such sedimentary rocks, with very low feldspar content and trace-element compositions similar to those of the impact-layer

samples, are present below the impact layer in northern Michigan and were also likely present in the Sudbury area at the time of impact (Mungall et al., 2004). This is supported by the presence of abundant carbonaceous mudstone fragments in the upper portion of the Onaping Formation at Sudbury (Bunch et al., 1999), the compositional overlap between the impact-layer samples and the Onaping Formation, and the apparent upward decrease in Ca and Na content in the Onaping Formation. These results are compatible with crater impact modeling studies (Grieve and Cintala, 1992; Pierazzo et al., 1997) that predict the upper-crustal portion of the shock-melted target rock volume at Sudbury would be ejected away from the crater area and would dominate in more distal ejecta deposits, such as those in Michigan. Overall, the chemistry of the Sudbury impact layer is more similar to compositions of the Onaping Formation within the Sudbury Basin than it is to felsic volcanic rocks above and below the impact layer in the Lake Superior region, reinforcing the interpretation that this layer is composed in significant part of ejecta produced by the Sudbury impact event.

The widespread occurrence of this unique and instantaneously deposited layer provides a new tool to establish exact temporal correlations between the numerous geographically separated iron ranges of the Lake Superior region. Our work, along with other recent studies, shows that the Sudbury impact layer marks the uppermost stratigraphic extent of the major banded iron formations of the Lake Superior region. In many of the iron ranges, it appears that banded iron formation was being deposited at the moment of impact, since the Sudbury impact layer lies directly on iron formation, which was at least partly un lithified when the impact layer was deposited. The impact layer is commonly overlain by black shale or other fine-grained clastic rocks. These relationships point to a fundamental change in sedimentation across the region, from ferruginous chemical sediments to clastic deposits, coincident with the impact. This change likely reflects some fundamental change in environmental conditions produced by the giant Sudbury impact event.

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intersections of the Sudbury layer at the Roland Lake and L'Anse localities. Staff of the Geological Survey Division, Michigan Department of Natural Resources, provided assistance and access to a large collection of drill core. Field work and drill core examination were assisted by Laurel Woodruff (U.S. Geological Survey) and John Klasner.

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APPENDIX: ANALYTICAL METHODS

Samples were analyzed at Activation Laboratories, Ltd., Ancaster, Ontario, Canada. Samples were pulverized with mild steel to minimize contamination and prepared for inductively coupled plasma (ICP) analysis using a lithium metaborate/tetraborate fusion dissolution procedure to ensure total digestion of any refractory minerals (e.g., chromite, zircon, sphene, monazite). The major elements and loss on ignition (LOI) (detection limit = 0.01 wt%, except TiO₂ and MnO = 0.001 wt%) plus Ba (1 ppm), Sr (2 ppm), V (5 ppm), and Y (1 ppm) were analyzed using ICP-optical emission spectroscopy (ICP-OES); Cu (1 ppm), Ni (1 ppm), Pb (5 ppm), S (0.001 wt%), and Zn (1 ppm) were analyzed using total digestion-ICP (TD-ICP); the rare earth elements (REE; La, Nd 0.05 ppm; Ce 0.1 ppm; Pr, Dy 0.02 ppm; Sm, Tb, Ho, Er, Yb 0.01 ppm; Eu, Tm 0.005 ppm; Lu 0.002 ppm) plus Cs (0.1 ppm), Ga (1 ppm), Ge (0.5 ppm), Hf (0.1 ppm), Nb (0.2 ppm), Rb (2 ppm), Ta (0.1 ppm), Tl (0.05 ppm), Th (0.05 ppm), U (0.05 ppm), and Zr (1 ppm) were analyzed using ICP mass spectrometry (ICP-MS); and Au (1 ppb), As (1 ppm), Br (0.5 ppm), Co (0.1 ppm), Cr (0.5 ppm), Sb (0.1 ppm), and Sc (0.01 ppm) were analyzed using instrumental neutron activation analysis (INAA). Accuracy and precision were monitored through repeat analyses of several international standards and are within analytical uncertainty compared with recommended values (major elements $\leq 5\%$; trace elements generally $\leq 10\%$ except near detection limit). In addition, FeO (0.01 wt%) was determined by titration, H₂O (0.1 wt%) was determined by gravimetric methods, and CO₂ (0.01 wt%) was determined by coulometry methods.

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