PLANETARY SCIENCE RESEARCH
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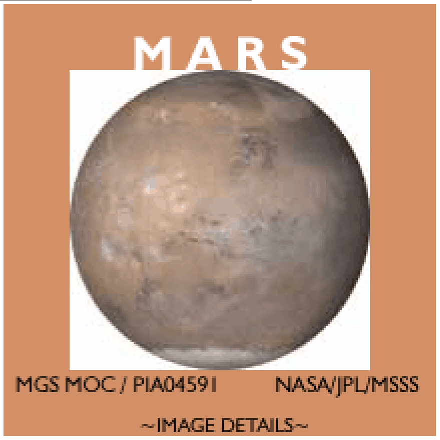

Headline Article

June 23, 2014

The Importance of When

--- Isotopic analyses at the microscopic scale indicate an ancient age for an impact mixture from Mars and appear to confirm a young age for a group of basaltic lava flows.

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Unraveling a planet's geologic evolution requires knowing what happened and when it happened. Two recent studies of the ages of Martian meteorites illuminate the early and late stages of Martian crust formation. One, by Munir Humayun (Florida State University) and colleagues in Australia and France studied Martian meteorite NWA 7533, an impact melt breccia composed of alkali-rich basalt similar to those at the Gusev landing site analyzed by instruments onboard the Spirit rover, and impact-modified products of such basalts. Humayun and colleagues determined the age of rock fragments in the meteorite by using secondary-ion mass spectrometry (SIMS) to date the mineral zircon. They report an age of 4.428±0.025 billion years, indicating formation of a substantial amount of the Martian crust by that time, a mere 100 million years after the planet formed.

Focusing on determining the other end of the age spectrum for magma production on Mars, Desmond Moser (University of Western Ontario) and colleagues at the University of Wyoming, the Royal Ontario Museum, and the University of California, Los Angeles made detailed electron beam observations and SIMS analyses of Martian meteorite Northwest Africa 5298. Their results show that the rock formed as a lava flow 187±33 million years ago, consistent with the young ages of basaltic Martian meteorites compositionally similar to it. Moser and colleagues suggest that the relatively young age resolves an important debate about the formation age of the basaltic meteorites from Mars. Both studies show the great value of using detailed *in situ* measurements to understand how and when rocks formed.

References:

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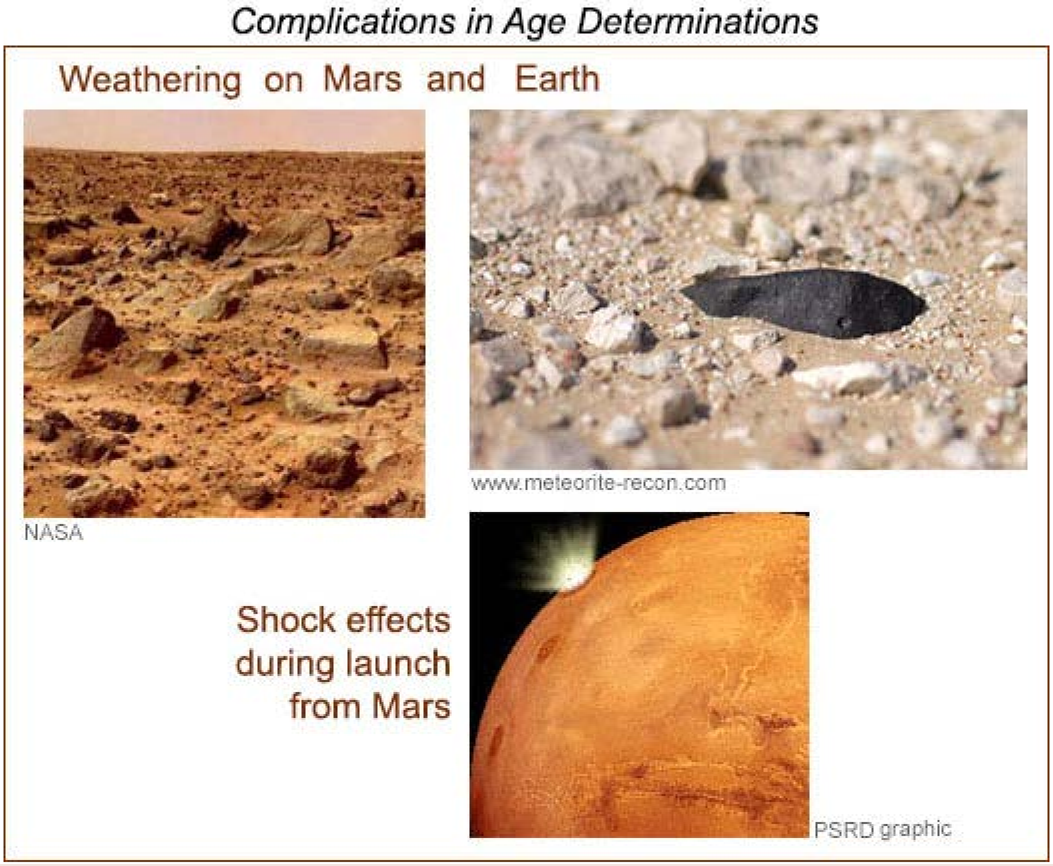
- Moser, D.E., Chamberlain, K.R., Tait, K.T., Schmitt, A.K., Darling, J.R., Barker, I.R., and Hyde, B.C. (2013) Solving the Martian meteorite age conundrum using micro-baddeleyite and launch-generated zircon. *Nature,* v. 499, p. 454-458. doi:10.1038/nature12341.
- **PSRDpresents:** The Importance of When --**Short Slide Summary** (with accompanying notes).

The Tricky Business of Determining When

A central goal of planetary science is to understand the fascinating series of events that led to the planets as they are today, including the timing of these astrophysical, cosmochemical, geological, and (at least in one case) biological events. The oldest dated materials in our Solar System, the calcium-aluminum-rich inclusions (**CAIs**) in **chondrite** meteorites, formed 4.567 billion years ago. Chondrites contain pre-solar grains that must have formed before CAIs, but these tiny grains have not been dated. Cosmochemists have determined the relative ages of assorted early Solar System products such as chondrules and meteorites formed when asteroids melted. However, overlapping geological processes complicate dating of events on planets. Heating events, such as a large impact, can partially reset the isotopic chronometers in a rock that initially formed from magma. Water-driven alteration (weathering on the surface or hydrothermal reactions at depth) can similarly upset the isotopic applecart. On Mars, the oldest igneous rocks making up the crust were reworked, mixed, and sometimes melted by the rain of projectiles that affected all the planets early in their histories. As magma intruded into and flowed onto the surface of the growing crust, it delivered water from the interior to the surface, altering some of the igneous rocks making up the crust. Further complicating the dating process for Martian meteorites is that the rocks were blasted off Mars by impacts, surely affecting the isotopic record in them. Indeed, the launch events converted the feldspar crystals in all samples composing the shergottite group of Martian meteorites into a non-crystalline state called maskelynite.

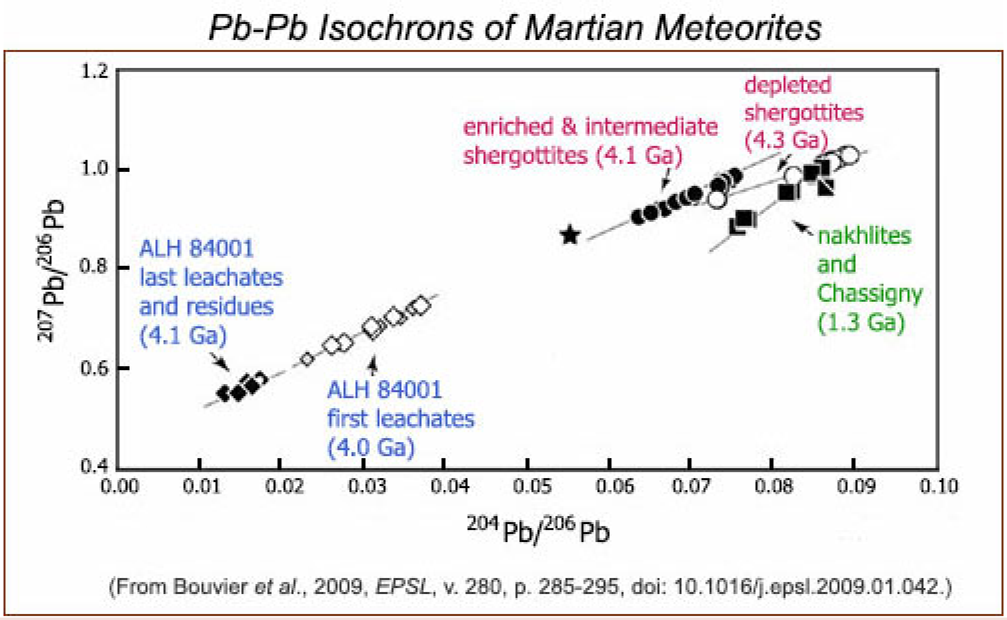


Isotopic dating of Martian meteorites is complicated by weathering on both Mars (left) and Earth (right), and the shock-heating associated with blasting rocks off Mars and during the long history of impact bombardment of the Martian crust.

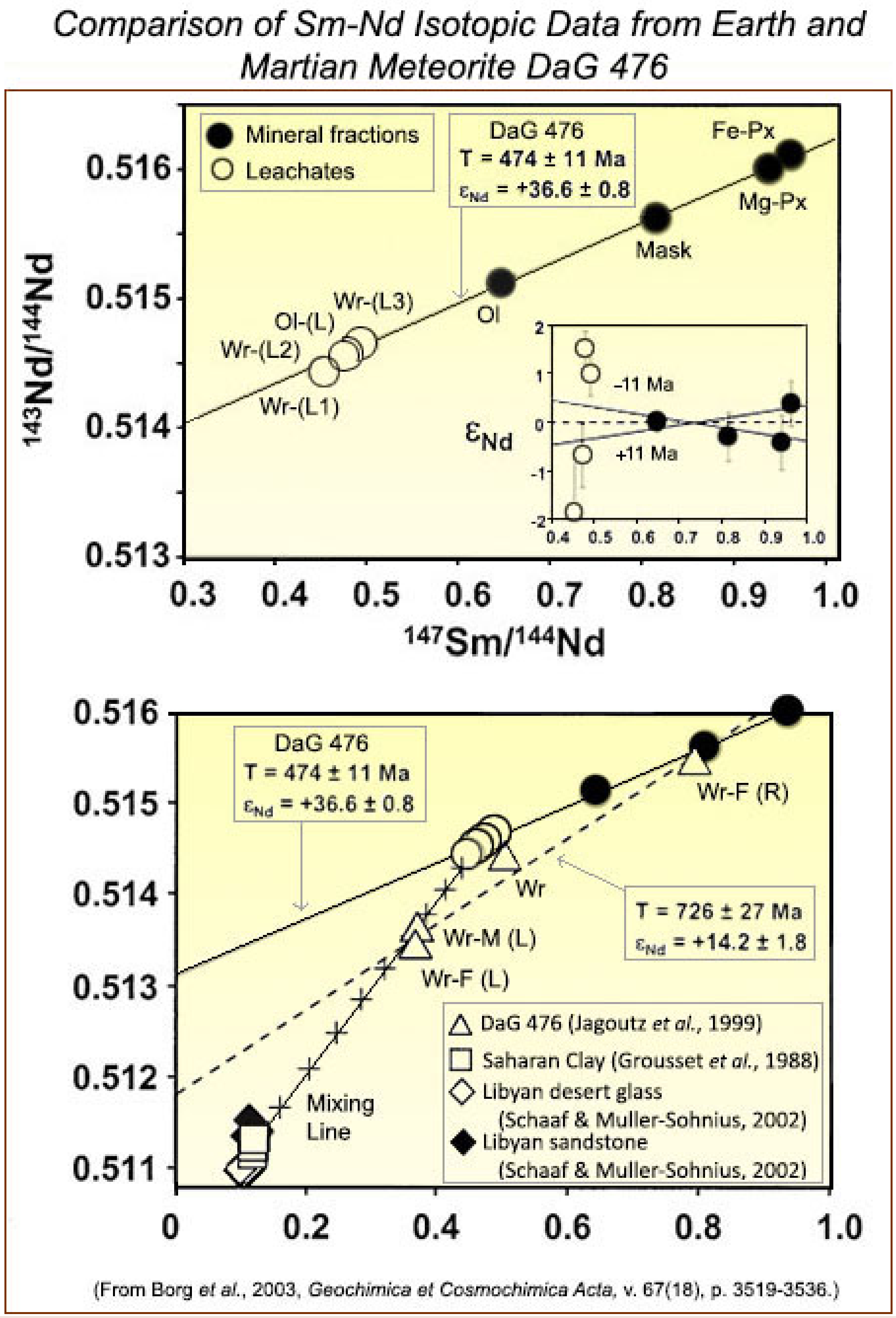
All this whacking and soaking makes it challenging to determine the ages of Martian meteorites. Cosmochemists accept the challenge because they want to know the details of Martian crustal evolution: What happened and when did it happen? Of course, it often leads to disagreements about how the chronological data are interpreted. A particularly vigorous debate has revolved around the ages of the shergottites. Everyone agrees that the rocks are from lava flows or possibly near-surface intrusions, and in spite of agreement about the analytical data, two schools of thought have emerged on when the shergottites formed. One tribe says that the shergottites formed in igneous events between about 180 and 575 million years ago; this group has a large membership, but I'll put Lars Borg (Lawrence Livermore National Laboratory) in charge. The other clan, led by Audrey Bouvier (University of Western Ontario), says that lead isotopic data show that the shergottites formed during two periods early in Martian history, one at 4.3 billion years ago and another at 4.1 billion years ago. The 4.1 billion year age also corresponds to the age of the ALH 84001 meteorite [**Data link** from the Meteoritical Bulletin], which everyone agrees is old, as shown by both lead isotopic data from Audrey Bouvier and lutetium-hafnium isotopic data (see **PSRD** article: **A Younger Age for the Oldest Martian Meteorite**).

This order-of-magnitude difference of opinion stems fundamentally from the difficulty of interpreting data from rocks that have been shocked and affected by reactions with water, both on Mars and on Earth. (Except for a few that were observed to fall, Martian meteorites languished in dirt or Antarctic ice for hundreds to tens of thousands of years before being collected.) Team Bouvier attaches special importance to using whole rock lead isotopic data, which suggest ages for the shergottites and ALH 84001 in the 4.1–4.3 billion year range. Team Borg attaches more importance to the use of mineral separates and rubidium-strontium and samarium-neodymium isotopic data. They argue that the whole-rock data reflect an early differentiation event in Martian history, not the eruption of lava flows. Bouvier counters that the young ages reflect partial resetting of isotopic clocks by the shock events that lifted the rocks off Mars, which cosmic ray exposure data indicate happened between 4 and 20 million years ago. She also points out that the isochrons derived by mineral separations are often complicated, reflecting the combination of shock heating and aqueous alteration.

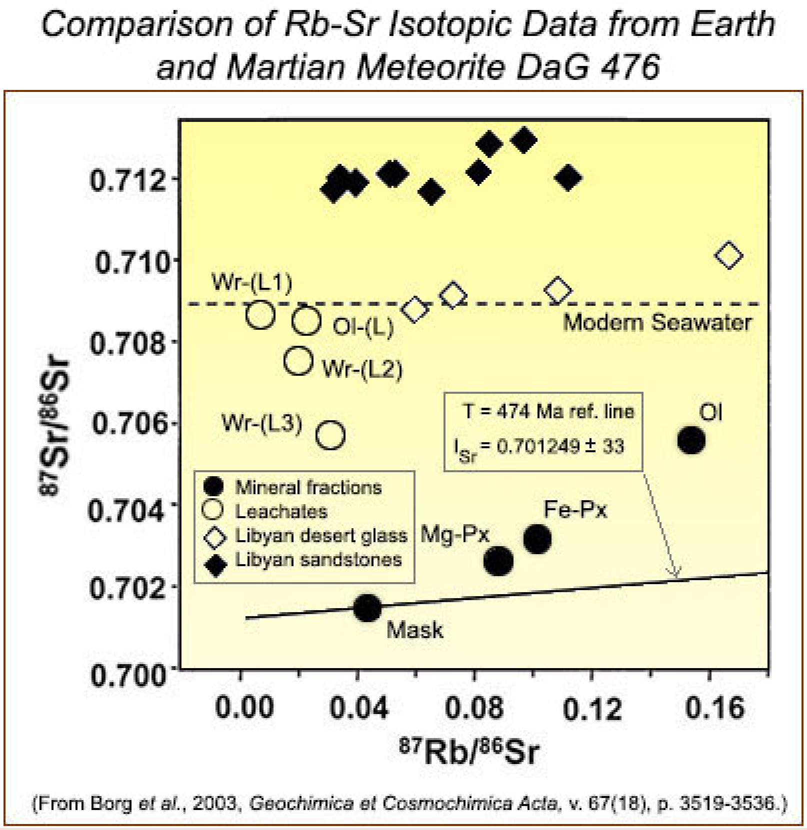
The curious thing about this impasse is that the data are not in dispute. For rubidium-strontium and samarium-neodymium isotopic data, Bouvier also sees good whole-rock isochrons showing young ages. The assorted isotopic datasets are just interpreted differently. Desmond Moser and his colleagues set out to get to the bottom of the young versus old shergottite debate.



This is one form of a lead-lead diagram. Well-defined ages should plot along straight lines; ages are given by the ratio on the y-axis. The data are for leached mineral separates and whole rocks. Four distinct groups are visible, two at 4.1 billion years (ALH 84001 and enriched shergottites), one at 4.3 billion years (depleted shergottites), and the fourth (nakhlites) at 1.3 billion years. Note that all investigators agree that the nakhlites are 1.3 billion years old and that ALH 84001 is 4.1 billion years old. This blissful agreement does not extend to the shergottites.



Two plots of samarium (Sm) – neodymium (Nd) isotopic data for shergottite DaG 476. The upper diagram shows that mineral separates (black circles) and whole rock samples that were treated to remove easily-leached materials (open circles) fall on a well-defined isochron indicating an age of 474 million years. Leaching (removal with acids) is necessary because of contamination with briny water that soaked the rock periodically as it sat in desert dirt in Africa. The bottom diagram shows that samples of desert material fall at one end of a mixing line with the DaG 476 samples. Leached samples from DaG 476 (open circles) fall in between desert material and the unaltered DaG 476 minerals (black circles).



Rb-Sr isotopic data for DaG 476, including separated minerals, leached whole rock samples, and desert rocks. If well behaved, the DaG 476 samples should all along the line at the bottom, but like the Sahara in Africa, the data are all over the map, so there is no age information. Among other things, this shows that age dating is not straightforward!

If the young ages for shergottites are correct, then it is curious that we have only one old rock in our collections of Martian meteorites, ALH 84001, considering that most of the surface is older than a few billion years. A recent find, NWA 7034 [**Data link** from the Meteoritical Bulletin], however, suggests that we might have a sample older than 2 billion years. Nicknamed "Black Beauty," NWA 7034 is a breccia composed of a collection of igneous rock and mineral fragments in a fine-grained, igneous-looking matrix. Munir Humayun and his colleagues studied a separate specimen, NWA 7533 [**Data link** from the Meteoritical Bulletin], that is paired with NWA 7034. We'll refer to both as Black Beauty.

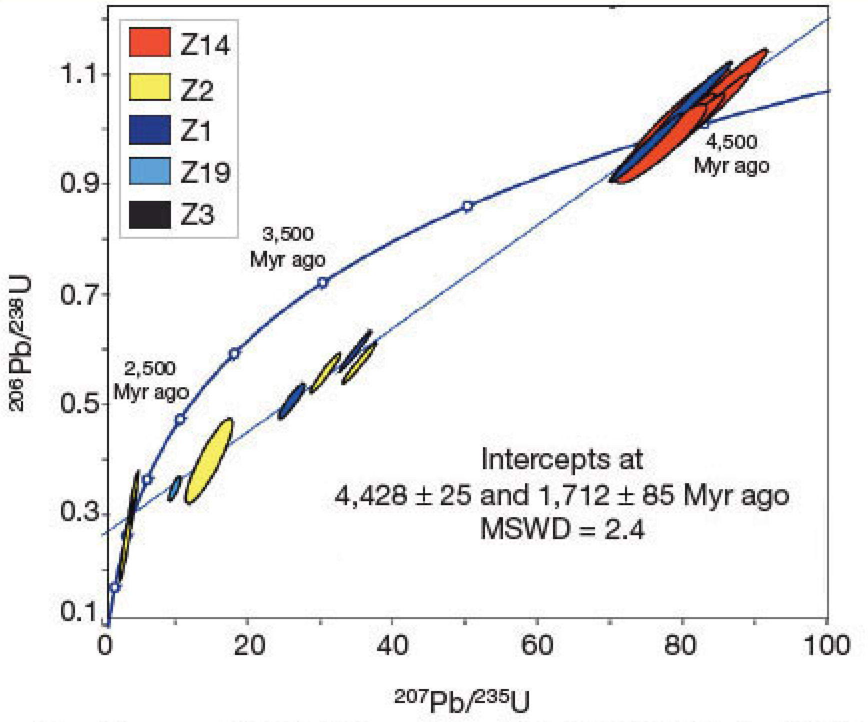
An Ancient Impact Melt from the Martian Highlands

The Black Beauty meteorite was studied by a well-organized consortium led by Carl Agee (University of New Mexico), as summarized in the **PSRD** article: **New Martian Meteorite is Similar to Typical Martian Crust**. Agee and his consortium mates identified the rock as a breccia (mixture of busted up rocks), but ascribed its origin to volcanic processes because it resembled volcanic breccias found on Earth. In contrast, in their thorough study of NWA 7533, Munir Humayun and coauthors suggest that it is an impact melt breccia, a drastically different origin. Either is consistent with the nature of the Martian surface, which is shaped by volcanoes and impact craters.

Munir Humayun and his coworkers made a comprehensive study of their samples of Black Beauty, but I am going to emphasize only the age data here. Carl Agee's group had determined an age of 2.1 billion years for the bulk sample using the rubidium-strontium method. Humayun and coworkers instead used the uranium-lead method to date individual crystals of zircon (zirconium silicate) in pieces of igneous rocks inside Black Beauty. The rocks were fragments of rock bodies that formed by **fractional crystallization** of alkali-rich magmas in the early Martian crust, so knowing their ages helps unravel the timing of crust-forming magmatism on Mars.

The team used a secondary ion microprobe called SHRIMP (sensitive high-resolution ion microprobe) at Curtin University in Perth, Australia to measure the concentrations of lead and uranium isotopes. The SHRIMP has a minimum spot size of 7 micrometers, so they could measure tiny zircon grains. Even with the small spot size, in some cases the beam overlapped the surrounding minerals, making data for those analyses unsuitable for age determination. Nevertheless, one or more points were analyzed for five zircon grains. The data are plotted in the diagram below (called a concordia plot). The entire array of data intercepts the curved line at two points, at 4.43 billion years and 1.71 billion years. Humayun and company interpret the older age as the mean igneous age of the rock fragments in Black Beauty. They interpret the younger age as a disturbance of some kind (most likely an impact event), which they suggest is consistent with the age of 2.1 billion years determined by the rubidium-strontium method.

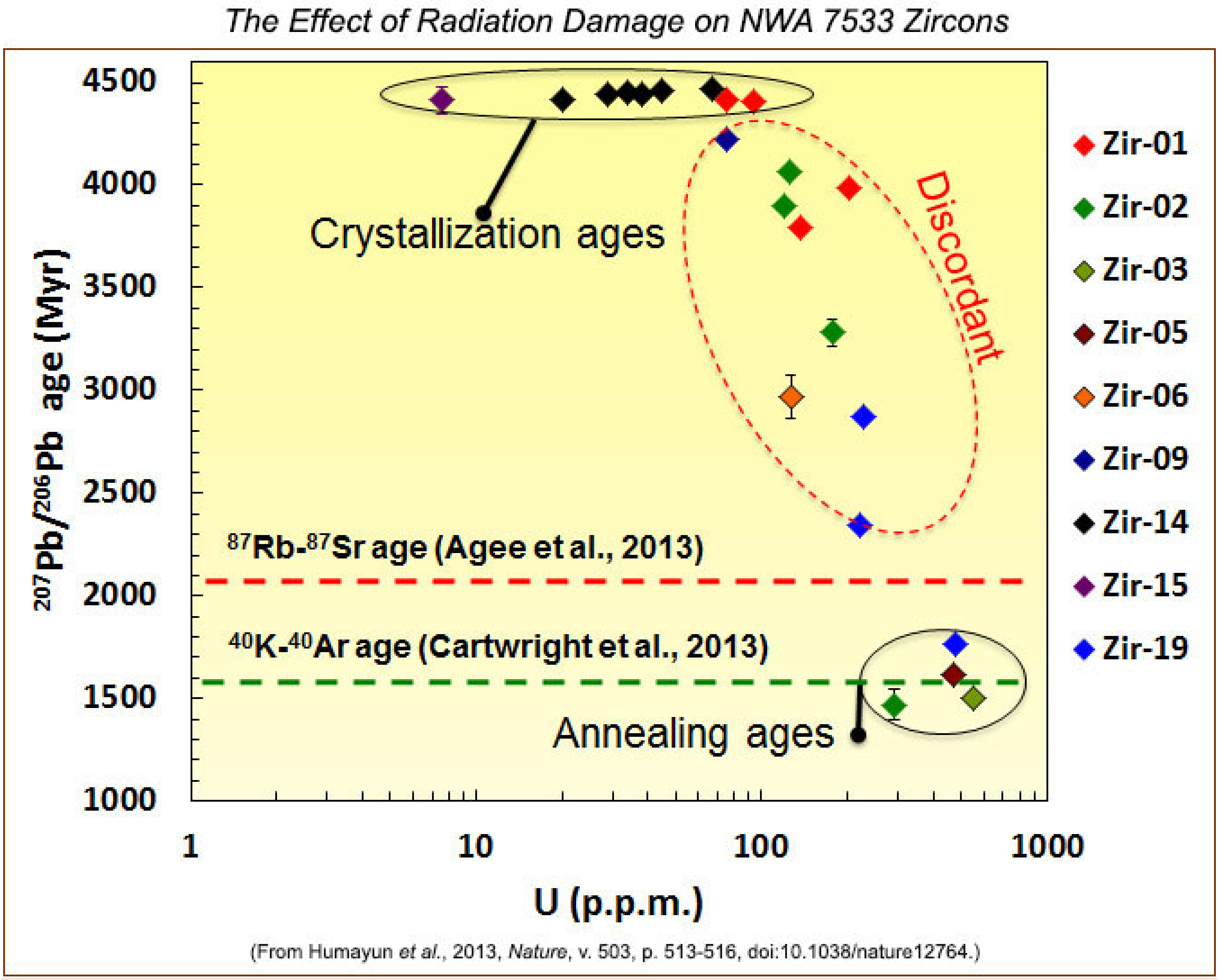
U-Pb Isotopic Data from Zircon Grains from Martian Meteorite NWA 7533



(From Humayun *et al.*, 2013, *Nature*, v. 503, p. 513-516, doi:10.1038/nature12764.)

Plot involving two uranium isotopes, U-235 and U-238, which decay to two lead isotopes, Pb-207 and Pb-206. The Pb-207/U-235 ratio is plotted against the Pb-206/U-238 ratio. Both ratios increase with age, but at different rates, accounting for the curvature of the concordia. The intercepts are usually interpreted as containing age information. In this case, the most straightforward interpretation is that the igneous rocks formed at 4428 million years (4.43 billion years), but that some isotope-disturbing event occurred at the younger intercept at 1712 million years (1.71 billion years). The younger intercept might date an impact event that assembled the Black Beauty impact melt breccia.

Humayun and his team examined the zircons closely. They found that the higher the U content of a zircon, the younger the apparent age. In fact, those with most uranium are metamict—they have lost most of their crystalline structure due to radioactive decay of uranium and thorium in the original zircon crystals. Zircons heated the most by the young event were annealed and their memory of their earlier formation (4.43 billion years ago) was completely erased. Others are partially reset (the sloping group labeled "discordant" in the diagram below), while others were not affected and record their formation in magmas.



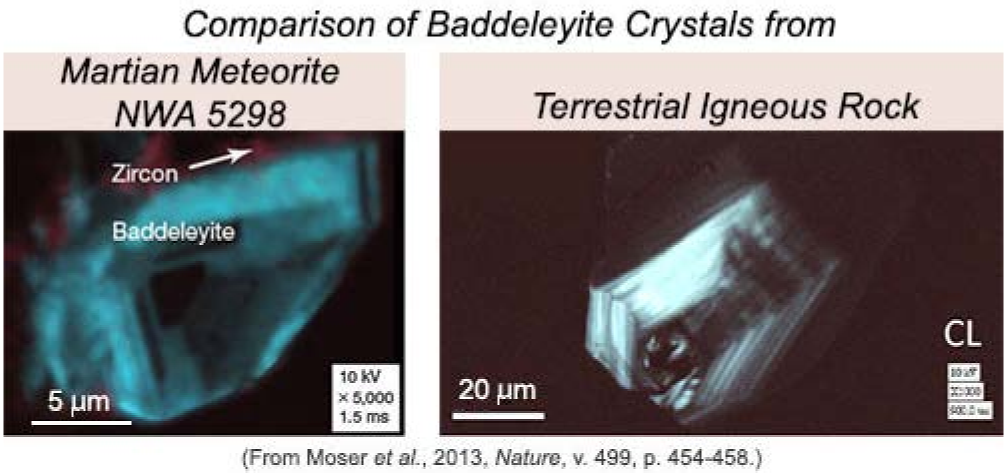
Lead-lead ages plotted against uranium concentrations in zircons. Those with lowest uranium experienced no resetting of the ancient age (4.4 billion years), whereas the samples with young ages were probably completely reset. The group labeled "discordant" contains samples that fall along the line in the age diagram shown above, and demonstrate partial resetting.

By anyone's standards, 4.43 billion years is an old age. In fact, Lars Borg and colleagues suggested on the basis of two samarium isotopes with drastically different half lives and their neodymium decay products that the Martian mantle and primitive crust formed 4.49 billion years ago. This age of early differentiation was updated by Vinciane Debaille and colleagues, who used the same isotopic systems to show that the mantle rocks from which depleted shergottites formed crystallized 4.535 billion years ago, only about 30 million years after formation of the first solids in the solar system. On the other hand, enriched shergottites indicate that their mantle source region formed 4.457 billion years ago, about 80 million years after formation of the mantle source rocks for the depleted shergottites. Munir Humanyan suggests that the 4.457 billion year age might represent formation of the earliest crust on Mars, indicating that the rocks in the Black Beauty breccia formed only about 30 million years after construction of the crust began. (See **PSRD** article: **The Multifarious Martian Mantle** for a discussion of enriched and depleted shergottites, and their possible formation mechanisms.) The new data from Black Beauty support an early formation of much of the Martian crust. The younger event at 1.7 billion years seems to be a resetting event. Could this be analogous to what we see in the shergottites, an igneous age of over 4 billion years and then resetting by impacts sometime during the past 500 million (0.5 billion) years?

A Young Lead-Lead Age for a Shergottite

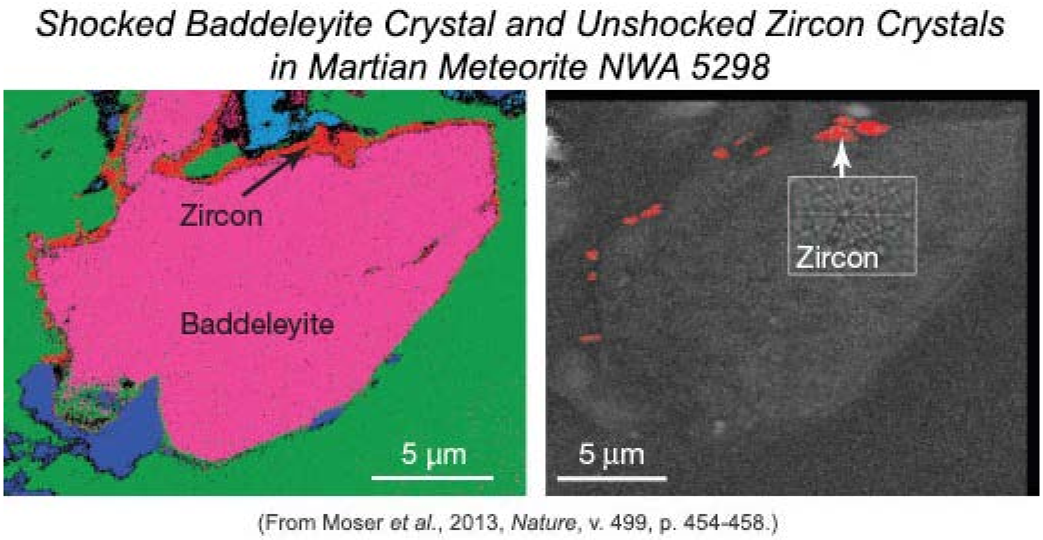
Desmond Moser and colleagues set out to determine the nature of the young event in the shergottites. Are they young igneous rocks or old ones heated by young impacts that annealed them and messed up their ages? To answer this question, Moser and his colleagues used microscopic imaging on Martian meteorite NWA 5298 [**Data link** from the Meteoritical Bulletin] to determine the history of minerals in the rock before determining the age using a secondary ion mass spectrometer (**SIMS**) by the lead-lead and uranium-lead methods. The rock is a typical basaltic shergottite that clearly formed in a lava flow, but is also heavily shocked. The trick was to find minerals rich in uranium but not shocked and heated so much that the uranium-lead isotopic system was altered and its age information degraded.

The team concentrated on baddeleyite (ZrO_2). As shown below, baddeleyite crystals preserve their igneous chemical zoning, as shown by the patterns in a cathodoluminescence image. The image is formed by scanning a beam of electrons across a sample that emits photons (many in the visible range) when hit with electrons. The luminescence is produced by assorted trace impurities in a mineral, almost always present during crystallization. Their distribution preserves the crystallization history of a mineral crystal. A terrestrial baddeleyite with similar igneous zoning is shown for comparison. Desmond Moser and colleagues argue that preservation of the igneous zoning makes the baddeleyite crystals in NWA 5298 good candidates for age dating. In addition, the baddeleyite crystals are randomly distributed and intergrown with the main igneous minerals, thus precluding a secondary origin, such as by shock. The baddeleyites are igneous minerals with preserved igneous zoning.



Cathodoluminescence image of a baddeleyite crystal in Martian meteorite NWA 5298 (left) and one in a terrestrial rock from South Africa (right). Both have concentric zoning. Preservation of the zoning in the Martian meteorite indicates that it is a good candidate for retaining the age information about its crystallization.

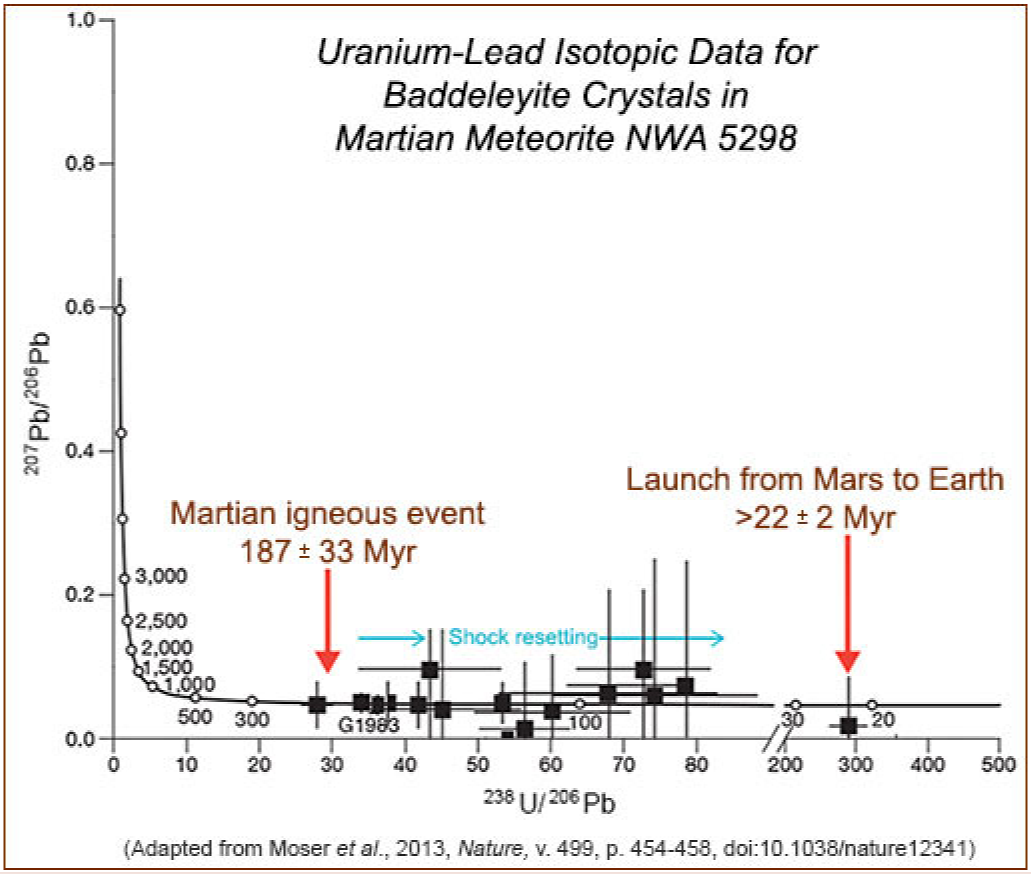
Besides the baddeleyite, little grains of zircon also occur in NWA 5298. They formed on the edges of the baddeleyite crystals. Significantly, the zircon crystals are not shocked. Since all the other minerals in the rock are severely shock damaged, including baddeleyite, this suggests that the zircon crystals formed after the event that damaged the other minerals.



[LEFT] False-color element map showing a baddeleyite grain (pink) embedded in plagioclase (green), now shocked to a non-crystalline state. The small red grains are zircons, shown better on the right. [RIGHT] The same area as on the left with an inset showing a representative electron back-scattered diffraction pattern for zircon. This technique allows determination of crystal structures and orientations. The key observation in this image is that the diffraction pattern is weak, indicating almost everything in the image is shock-damaged. The exception is the strong diffraction strength from zircon grains, which are colored red. Moser and coworkers conclude that the baddeleyite was shocked along with the other minerals, but that zircon formed after the shock event. Note that although baddeleyite is shocked, Moser and colleagues argue that the preservation of original igneous zoning (previous images) indicates that the isotopic clocks in it were not reset by the shock event.

The diagram below shows uranium and lead isotopic data from ion microprobe analyses in another form of a concordia diagram from what was shown above from Audrey Bouvier's work and for the Black Beauty data. Ages in millions of years appear along the curved line. In this case, the isotopic data for the baddeleyite (black squares) lie along the flat portion of the concordia, rather than plotting along a line that intercepts the curve to two places, as was the case for the Black Beauty data. The mere fact that the points rest along the bottom of the diagram indicates that they are recording a young age. Desmond Moser and his colleagues suggest that the best age determination comes from averaging the four grains with the lowest $^{238}U/^{206}Pb$ ratios, hence with the most Pb produced by radioactive decay. The average (red arrow) is 187±33 million years, indicating a young age for this shergottite. The points that string out to the right may reflect loss of Pb caused by the shock and associated heating that the rock experienced. The youngest one, hence with the most thoroughly reset age, lies at 22±2 million years. Moser and coworkers take this age as the upper limit for when the meteorite was launched from Mars.

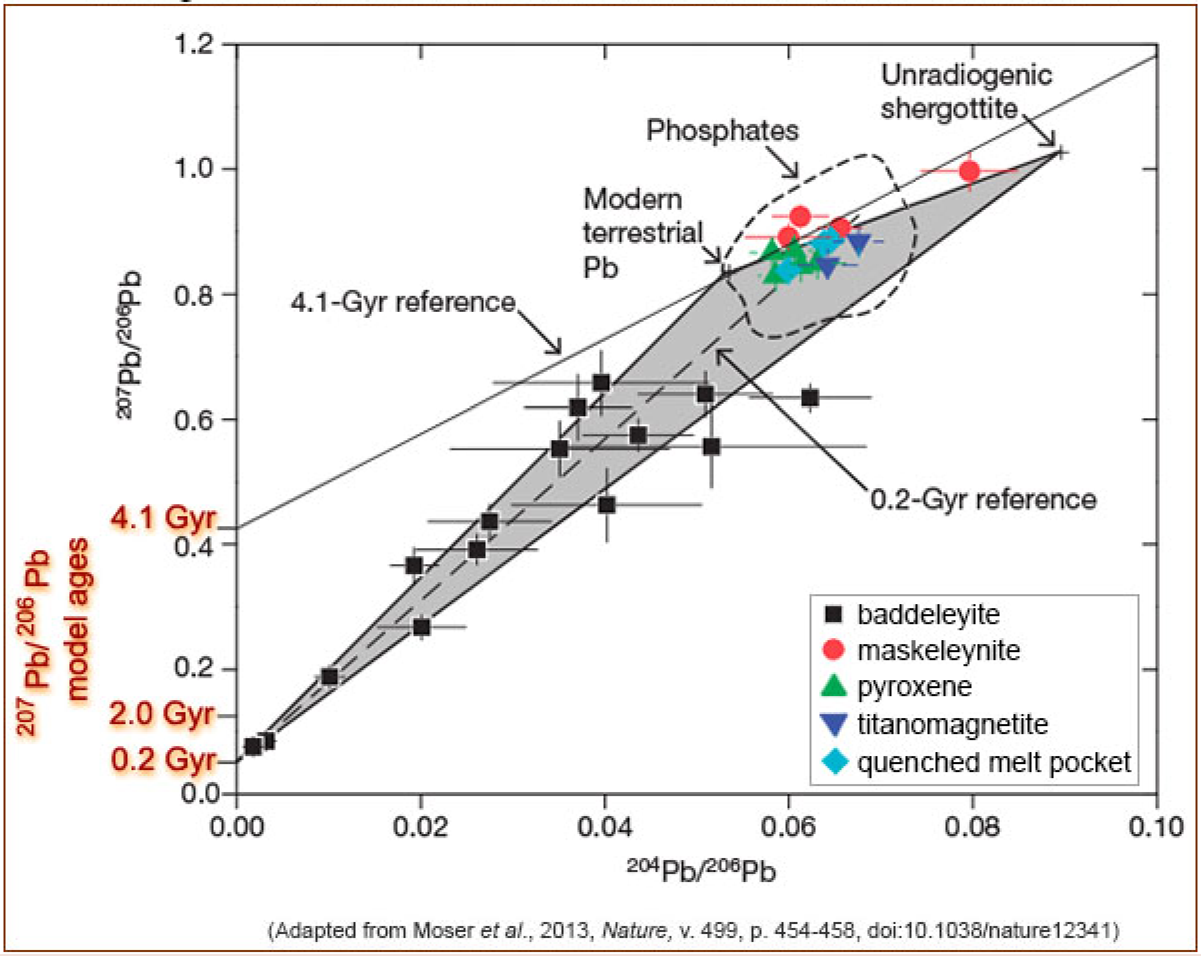
The string of baddeleyite pearls vary in the extent to which their igneous zoning is preserved, as revealed by cathodoluminescence imaging: the more distinct the zoning, the more preservation, and the older the apparent age. The baddeleyites with the most shock damage have the youngest apparent ages. Thus, the samples with dates around 187 million years probably most closely reflect the time when NWA 5298 formed in a lava flow on Mars. Consistent with this age interpretation, Qin Zhou (Institute of Geology and Geophysics, Chinese Academy of Sciences, Beijing) and colleagues analyzed baddeleyite crystals in the shergottite Zagami and found a young age of 182±7 million years. An important additional observation is that the data show that even baddeleyite uranium-lead ages can be altered by shock, in contrast to arguments put forward by some young-Mars enthusiasts.



Uranium-lead concordia diagram showing data for baddelyite crystals in shergottite NWA 5298. Ages are determined by where the data lie on the curve (numbers indicate age in millions of years). The four oldest baddelyite grains have a weighted average of 187±33 million years, consistent with young ages interpreted for other shergottites. The point farthest to the right may represent the upper limit of the time when the meteorite was launched off Mars by an impact event. Desmond Moser and co-authors interpret the points in between the average age at 187 million years and the launch event at 22 million years as indicating partial resetting of the uranium-lead age by shock-heating during the launch impact.

Moser and colleagues also plot their isotope data on a lead-lead diagram, which entails plotting one ratio ($^{207}Pb/^{206}Pb$) against another ($^{204}Pb/^{206}Pb$), as shown earlier for Audrey Bouvier's data. Although Pb may be redistributed by shock heating (hence changing the ratio of a lead isotope to a uranium isotope), ratios of one lead isotope to another do not change significantly. On this diagram, see below, grains that formed at the same time string out along a line, with the age determined by the intercept along the y-axis. Calculations of how the lead ratios vary with time allow cosmochemists to determine the absolute age from the $^{207}Pb/^{206}Pb$ value of the intercept, and those ages are shown in red along the y-axis. Measurements and inferences from measurements of lead in the Earth, a frequent contaminant in samples and even labs, and initial lead in Mars (labeled "Unradiogenic shergottite") in the diagram, fall on or close to the 4.1 billion year (Gyr) reference line. Audrey Bouvier's data also fall on this line as shown in the first isotope plot above. In addition, minerals from NWA 5298 also cluster on the line.

Lead-Lead Isotopic Data for Baddeleyite Crystals in NWA 5298 with Shaded Field of Three-component Mixtures of Radiogenic Pb, Terrestrial Common Pb, and Martian Common Pb

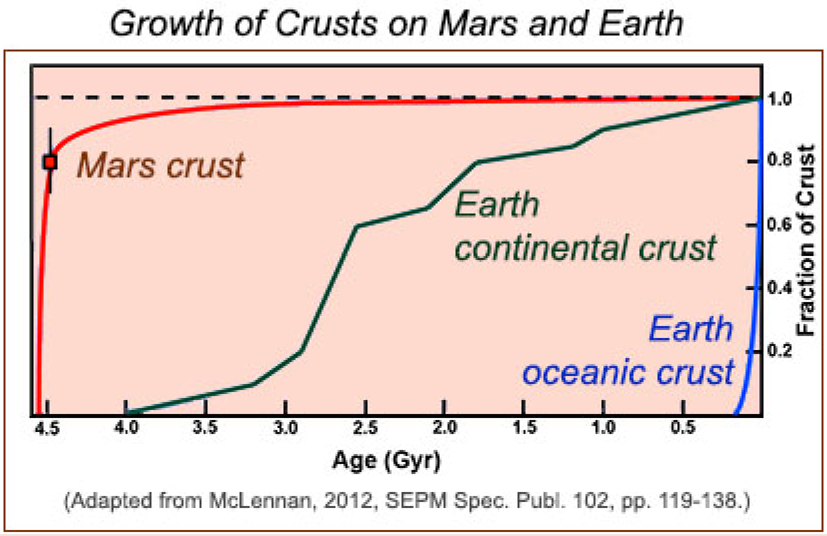


Lead-lead diagram showing baddeleyite analyses occupying a shaded triangle, implying a mixture of initial lead in Mars, terrestrial contamination, and a young event in the history of shergottite NWA 5298. The reference line at 0.2 Gyr (billion years) is consistent with the age determined from the condordia diagram above and drastically different from the 4.1 Gyr reference line.

In their 2008 paper, team Bouvier examined the idea that the isotopic composition of lead in shergottites could have resulted from mixing three components: (1) lead from young rocks (which would plot on the y-axis at about 0.2 billion years, (2) initial lead in Mars (unradiogenic shergottite), and (3) terrestrial contamination. They concluded that a young rock would scatter inside the shaded triangle, with the points representing differing amounts of the three components. As the diagram above shows, the baddeleyites fill up the triangle, though they do lie along a 0.2 billion year reference line. This is consistent with the young age determined from the concordia diagram. In addition, Moser and colleagues note that the data plot far from a 4.1 billion year reference line, hence inconsistent with team Bouvier's ancient age for the shergottites.

Knowing When—What Does it Tell Us?

The rocks residing in the Black Beauty breccia are old, having formed in magmas (or possibly impact melt pools) over 4.4 billion years ago. The ancient ages show that a large volume of crust had piled up within 100 million years of the formation of the planet, a rate of crustal growth much different from the growth of continents on Earth.



Plots of the cumulate growth of the crust of Mars and the continental and oceanic crusts on Earth. Vertical axis is the fraction of the total final crust that has formed by a given time. For Mars it is based on the ages of geologic units and is calibrated by crater counts. Terrestrial continental crust appears to be a slow, methodical process, whereas much of the basaltic crust of Mars was formed early, with crustal formation petering out with time. Thus, the ancient rocks in the Black Beauty meteorite are important to understanding formation of the crust of Mars. Although Earth appears to be languid compared to Mars, production of the basaltic oceanic crust is vigorous, but is continuously recycled by plate tectonics back into the mantle. No such recycling takes place on Mars.

As Carl Agee has noted, Black Beauty is not just one rock. It is like a Martian geologic field area conveniently aggregated into one rock. Chemical analyses by Agee and coworkers and Humayun and his team show that the rock collection nestled in the meteorite is composed dominantly of alkali-rich rocks with geochemical properties that could react with shergottite magmas to form the enriched group of shergottites (see **PSRD** article: **The Multifarious Martian Mantle** for a discussion of enriched and depleted shergottites). The matrix of Black Beauty is a complicated mixture of impact products, possibly developed over billions of years. The young intercept age of about 1.7 billion years might represent the assembly time of the whole rock. Thus, Black Beauty contains fragments of the origin and early evolution of the Martian crust. The ancient rocks are set in a matrix of impact-modified materials that may help us understand how impacts and water-driven processes have modified the Martian crust. It is, indeed, a field area in a rock, and one that tells a sweeping story of Martian geological evolution.

Black Beauty is old, but are the shergottites old? Desmond Moser and his colleagues present evidence that shergottite NWA 5298 retains a record of its igneous origin 187±33 million years ago. The age record is complicated by the impact event that blasted the meteorite off Mars, which appears to have taken place less than 22 million years ago. However, as Moser and colleagues argue, the lead-lead isotopic data is resistant to resetting, so perhaps the launch event did not erase the record of a young igneous origin about 200 million years ago. The overall data from baddeleyite crystals are inconsistent with the whole rock ages reported by Audrey Bouvier and colleagues. It seems likely that the shergottites formed as lava flows on Mars from a couple to a few hundred million years ago, not over 4 billion years ago as interpreted by team Bouvier. However, the lead-lead data is an excellent tracer of the chemical processes that produced the chemically-modified region of the mantle that eventually melted to produce the shergottite magmas.

The two studies described in this article and the predecessor studies on which they build provide important data points in understanding the timing of Martian magmatic history and mantle chemical and dynamical evolution. The mantle source regions seem to have formed early, some by 4.535 Gyr, with the crust forming no more than 80 million years later, by 4.457 Gyr (the age of the oldest clasts in Black Beauty). This indicates rapid growth of the crust at first, as suggested by models for growth of the Martian crust. Because magma carries most of the volatiles such as water from the interior to the surface, most of the water-driven processes on Mars, such as creation of valley networks and thick deposits of clay minerals, must have taken place early in the planet's history.

The regions of the mantle that produced the shergottite magmas appear to have languished inside Mars for over 4 billion years, producing little if any magma, until springing to life about 500 million years ago, leading to shergottite lava flows decorating the surface. This lazy period for regions of the Martian interior implies that the mantle does not constantly overturn in the dynamic fashion that the mantle of Earth does.

The story may not be complete, but what we know so far illustrates the importance of knowing when.

Additional Resources

Links open in a new window.

- **PSRDpresents:** The Importance of When --**Short Slide Summary** (with accompanying notes).

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