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Rapid cooling of planetesimal core-mantle reaction zones from Mn-Cr isotopes in pallasites

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Abstract

Pallasite meteorites, which consist of olivine-metal mixtures and accessory phosphates crystallised from silico-phosphate melts, are thought to represent core-mantle reaction zones of early differentiating planetesimals. Pallasite meteorites can be linked to five distinct planetesimals, indicating that they are default products of differentiation. However, their formation modes (deep, shallow, and impact environments) and age are still elusive. We have investigated the trace element and Mn-Cr isotopic signatures of Main-Group pallasite olivine, finding enhanced Mn, P and $^{53}Cr/^{52}Cr$ near crystal rims which indicates early ingrowth of radiogenic $^{53}Cr^*$ in silico-phosphate melts. Mn-Cr isotopic data corroborate previous Hf-W isotopic data, indicating an early metal-silicate separation event but additionally that rapid cooling generated silico-phosphate eutectic melts with high Mn/Cr within ~2.5 to 4 Myr of Solar System formation. These melts formed before most known samples of planetesimal crusts (eucrite and angrite meteorites) and are among the earliest evolved planetary silicates. Additionally, Mn-rich phosphates in other, non-Main-Group pallasite meteorites suggest that core-mantle reaction zones are generic, datable features of differentiation.

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Introduction

The accumulated oxygen isotopic evidence for five distinct parent bodies of olivine and metal-rich pallasite meteorites (summarised in Boesenberg *et al.*, 2012) suggests that pallasitic material is the default end-product of planetesimal differentiation. Pallasites may represent samples of quiescent core-mantle boundaries (Boesenberg *et al.*, 2012), or violently formed mixtures of core and mantle materials (Yang *et al.*, 2010; Tarduno *et al.*, 2012) and are often discussed

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in terms of trapping of olivine in a liquid Fe-Ni metal groundmass progressively becoming saturated in chromite, troilite, schreibersite, and phosphate minerals during cooling (Boesenberg *et al.*, 2012). Despite this diverse mineralogy, the short-lived isotope chronologies of pallasites remain poorly understood due to depletion in crustal components (plagioclase and pyroxene; Nyquist *et al.*, 2009). The ^{53}Mn-^{53}Cr decay scheme ($t_{1/2}$ 3.7 Myr) would seem ideal for such meteorites due to the fractionation of parent and daughter between olivine (generally with high Mn/Cr) and chromite (low Mn/Cr); however, pallasite chronologies have stalled since pioneering Thermal Ionisation Mass Spectrometry (TIMS) studies (Birck and Allègre, 1988; Lugmair and Shukolyukov, 1998), with conflicting Secondary Ion Mass Spectrometry (SIMS) results having been later retracted (Huss *et al.*, 2011; Telus *et al.*, 2012). In this study, we re-investigate pallasite olivine trace elements and Mn-Cr isotopes using improved Laser-Ablation Inductively-Coupled-Plasma Mass-Spectrometry (LA-ICP-MS; Spandler and O'Neill, 2010) and SIMS (McKibbin *et al.*, 2013a) in two texturally different Main-Group pallasites: Brenham, with rounded olivines sharing equilibrated triple-junctions across a continuous network; and Brahin, with fragmental olivine of variable grain size (McKibbin *et al.*, 2013b).

Methods

Samples were cast in epoxy and polished for microbeam analysis. Trace element analyses of olivine were carried out following a previously developed LA-ICP-MS profiling method (Spandler and O'Neill, 2010). Sampling was undertaken with a 193 nm wavelength ArF Excimer laser coupled to a custom-built two-volume vortex ablation cell at RSES-ANU. An aperture was used to shape the laser beam into a rectangle, enabling high spatial resolution sampling parallel to crystal margins. Sampling of meteoritic olivine was bracketed by NIST610 and 612 glass standards, with the gas being analysed using an Agilent 7500s ICP-MS. Data were reduced according to established methods (Longerich *et al.*, 1996; Spandler and O'Neill, 2010) with NIST 612 as the primary external standard (Pearce *et al.*, 1997). For SIMS Mn-Cr analysis, we modified previous Sensitive High-mass Resolution Ion Micro Probe Reverse Geometry (SHRIMP-RG) methodology (McKibbin *et al.*, 2013a,c). Suitable areas of olivine ~30 µm in diameter were sputtered by a high current unfiltered primary ion beam (~16.9-56.6 nA mixed O^- and O_2^-; McKibbin *et al.*, 2015). Ion beams of interest were directed sequentially to a single electron multiplier by cycling the magnet. For measurements in Brahin chromite (~4.0-5.5 nA filtered O_2^- primary ion beam), sequential collection of $^{52}Cr^+$, $^{53}Cr^+$, and $^{55}Mn^+$ was achieved with a single Faraday Cup. Data were reduced according to the 'first ratio estimator' method (Ogliore *et al.*, 2011) which we earlier referred to as 'ratio of total counts' (McKibbin *et al.*, 2013c). Instrumental mass fractionation was corrected by sample-standard bracketing with San Carlos olivine or in-house reference spinels. To convert $^{55}Mn^+/^{52}Cr^+$ to $^{55}Mn/^{52}Cr$, we used a Relative Sensitivity Factor (RSF) of 0.58 (±0.02) as a multiplier on olivine data. No multiplier was applied to chromite.

Results

LA-ICP-MS trace element profiles (Fig. 1 and Supplementary Information) indicate low Cr and high P and Mn concentrations near edges of pallasite olivine grains. Grain-to-grain and intra-grain variations in P and Cr are more pronounced in

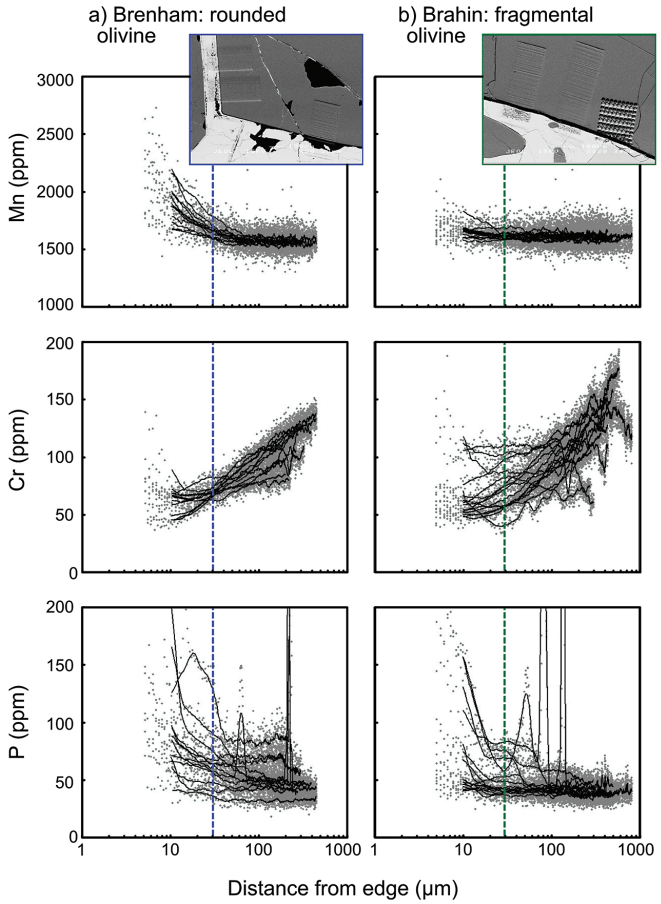

Figure 1 LA-ICP-MS trace element concentrations traverses in olivine for **(a)** Brenham (left panels) and **(b)** Brahin (right panels). Traverses were conducted from core to rim; concentrations are in parts per million by weight (ppm) and horizontal axes represent distances from olivine rims (μm). Data are represented by grey points and traverse averages are traced by solid lines. Vertical dashed lines (blue and green for Brenham and Brahin respectively) represent limit of SIMS spatial resolution (~30 μm) when analysing near olivine margins. LA-ICP-MS tracks from this study and ablation pits from previous work (McKibbin *et al.*, 2013c).

rounded than in fragmental olivine, whereas Mn is very consistent. Mn and Cr variations range over a factor of ~2-3 in olivine $^{55}Mn/^{52}Cr$ which ordinarily would be considered marginal for SIMS Mn-Cr dating. Nevertheless, this is well correlated with $^{53}Cr/^{52}Cr$ in the rounded olivines of Brenham (Fig. 2, blue data), while fragmental olivine exhibits more restricted $^{55}Mn/^{52}Cr$ and $^{53}Cr/^{52}Cr$ (Fig. 2, green data). Chromite has normal $^{53}Cr/^{52}Cr$ (Fig. 2 and Supplementary Information). $^{55}Mn/^{52}Cr$ and $^{53}Cr/^{52}Cr$ regression yields excessively high initial $^{53}Mn/^{55}Mn$ values, assuming *in situ* ^{53}Mn decay and that the system was closed to disturbance (Brenham: initial $^{53}Mn/^{55}Mn$ of 4.0 (±1.2) × 10^{-5} with sub-terrestrial initial $^{53}Cr/^{52}Cr$ at 0.11285 (±0.00024) (2-sigma; MSWD = 0.63); Brahin: initial $^{53}Mn/^{55}Mn$ of 8.4 (±4.5) × 10^{-6} with initial $^{53}Cr/^{52}Cr$ of 0.11340 (±0.00009) (2-sigma, MSWD = 0.49)).

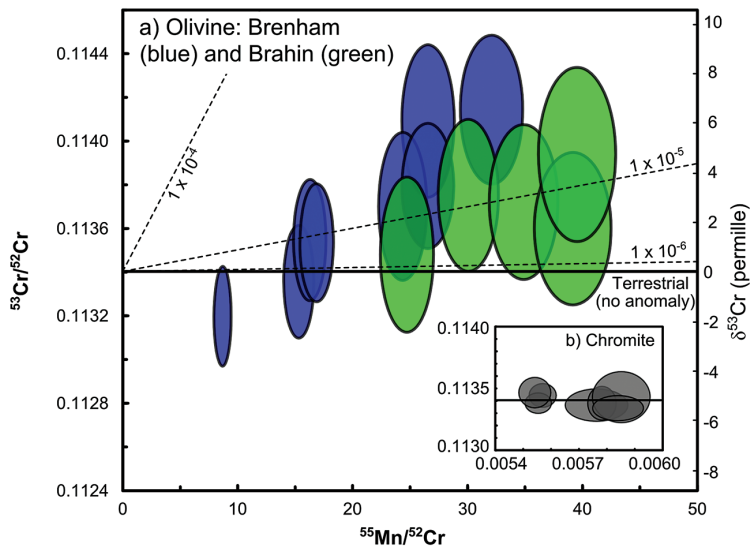

Figure 2 Mn-Cr isotopic composition of olivine and chromite from Brenham and Brahin pallasite meteorites. **(a)** SIMS Mn-Cr systematics for olivine from Brenham (blue ellipses) and Brahin (green ellipses) (errors are 2-sigma), and **(b)** Brahin chromite (data near the origin expanded in subfigure; 2-sigma errors in grey). The terrestrial Cr-isotopic composition (no anomaly) and increasing initial $^{53}Mn/^{55}Mn$ correlations are illustrated by an unbroken horizontal and inclined dashed lines respectively.

Discussion and Conclusions

The cooling history of pallasites is well recorded by late crystallising, P_2O_5-rich accessory minerals, as well as our trace element and isotope results for olivine rims. Phosphoran olivine (with several wt. % P_2O_5) and Mg- and Si-phosphate

minerals crystallised from an unusual P_2O_5-rich silicate melt (Boesenberg *et al.,* 2012) which might ultimately be formed by oxidation of metallic P. High temperature merrillite and peritectic stanfieldite formed at or above ~1175 °C and have generally high rare earth element (REE) concentrations, while eutectic stanfieldite, farringtonite and silico-phosphate crystallised near the ~1120 °C eutectic after *in situ* fractional crystallisation (with generally lower REE concentrations; Ando, 1958; Davis and Olsen, 1991; Hsu, 2003). A parallel trend is recorded by olivine geochemistry: olivine cores exhibit variable Cr and P (Fig. 1) while divalent Mn varies over a shorter distance of several tens of micrometres, consistent with an overgrowth or rapid diffusion of divalent elements (Zhukova *et al.,* 2014a) and more slowly diffusing trivalent Cr (Ito and Ganguly, 2006) and pentavalent P (Spandler and O'Neill, 2010). However, the outermost ~30 µm of olivine crystal margins have very low Cr and steeply increasing P, recording crystallisation of olivine after cooling to the silico-phosphate eutectic (~1120-980 °C; crystallising farringtonite, stanfieldite, olivine, and probably iron and troilite as well). The occurrence of genuinely 'phosphoran' olivine rims in some pallasites, with several wt. % P_2O_5 (Boesenberg *et al.,* 2012) also indicates rapid cooling at this stage and eventual saturation of this metastable mineral.

The inferred initial $^{53}Mn/^{55}Mn$ of 4.0 (±1.2) × 10^{-5} for Brenham and 8.4 (±4.5) × 10^{-6} for Brahin are very high compared to other early Solar System materials (Birck and Allègre, 1988; Lugmair and Shukolyukov, 1998) and cannot represent fossil isochrons because they exceed or are close to the initial $^{53}Mn/^{55}Mn$ for the Solar System (9.1 (±1.7) × 10^{-6}; Nyquist *et al.,* 2009). Small variations (~0.1 per mille) in $^{53}Cr/^{52}Cr$ are likely present in Brenham pallasite olivine, with slightly higher (~2 per mille) excesses in coexisting metal (Hsu, 2005; Qin *et al.,* 2010), due to cosmic ray exposure effects during transport to Earth. However, our $^{53}Cr/^{52}Cr$ in olivine rims are higher and inconsistent with the lower susceptibility of olivine to cosmogenic effects relative to metal (via lower Fe/Cr). Anomalies related to pre-solar, nucleosynthetic isotope components are even smaller and unlikely to survive planetary differentiation (Trinquier *et al.,* 2008; Qin *et al.,* 2010). We therefore conclude that ^{53}Cr excesses are radiogenic.

Although relatively low, $^{55}Mn/^{52}Cr$ (~10-40) implies that radiogenic Cr is unsupported by Mn, the elevated $^{53}Cr/^{52}Cr$ nevertheless implicates a reservoir with high Mn/Cr and indicates fractionation of Mn from Cr after significant decay, *i.e.* at least several million years after establishment of an early reservoir. The unsupported nature of Cr is inconsistent with *in situ* decay of ^{53}Mn followed by ^{55}Mn loss since elemental profiles indicate that, if Mn was mobile, then it migrated into olivine rather than out. Rather, association of $^{53}Cr/^{52}Cr$ with P suggests that ingrowth of $^{53}Cr^*$ occurred in a high Mn/Cr silico-phosphate melt before crystallisation of olivine rims and phosphates.

High Mn/Cr in eutectic phosphates (~60-145 in Springwater and Eagle Station pallasites; Davis and Olsen 1991; Hutcheon and Olsen, 1991), probably due primarily to early chromite saturation which keeps available Cr concentrations low, indicates that the melt from which they crystallised is a good candidate

for the high Mn/Cr reservoir. Ingrowth of $^{53}Cr^*$ in this melt requires several million years, *i.e.* an interval similar to or longer than the half-life of ^{53}Mn. Such a reservoir is modelled in Figure 3 as a function of $^{55}Mn/^{52}Cr$ and the timing of isolation from the parent body or nebula (Myr after Solar System formation) in order to find a model-dependent Mn-Cr date for this event. We assume a Solar System initial of $^{53}Mn/^{55}Mn$ of 9.1 × 10^{-6} (Nyquist *et al.,* 2009) and that ^{53}Mn decays completely, implying that the reservoir persists for ~10-20 Myr. The difficulties of a lower Solar System initial, such as in Trinquier *et al.* (2008), or incomplete decay of ^{53}Mn, are discussed below.

The $^{55}Mn/^{52}Cr$ of phosphates (~60-145; Davis and Olsen, 1991; Hutcheon and Olsen, 1991) and olivine rims (~25-40; this study) can be used to bracket the parental silico-phosphate melt (vertical lines in Fig. 3). Mn and Cr are generally considered to be fairly neutrally partitioned between olivine and melt (*e.g.,* Mallmann and O'Neill, 2013) although by comparison with other pallasites, Mn may have been more incompatible in olivine from the Brenham and Brahin

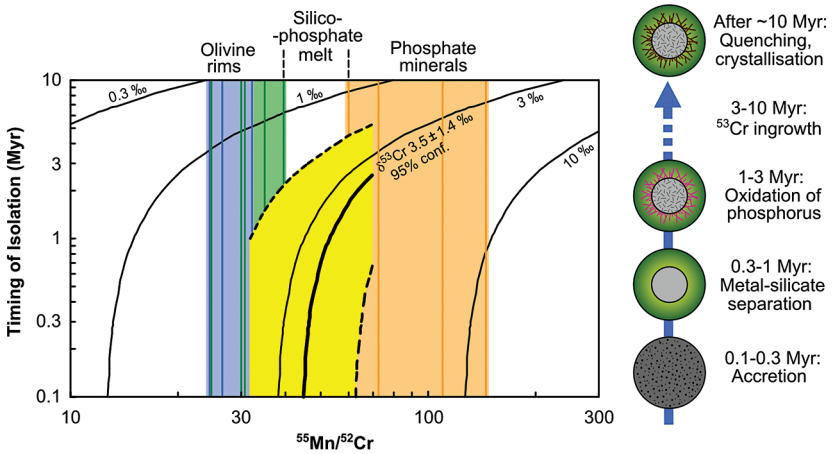

Figure 3 Model of the Mn-Cr evolution of a planetary (pallasite) reservoir as a function of $^{55}Mn/^{52}Cr$ and time of isolation from the Solar System. Hyperbolic lines indicate various $^{53}Cr/^{52}Cr$ compositions (in per mille). SIMS analyses for Brenham and Brahin olivine which yielded radiogenic Cr (in the $^{55}Mn/^{52}Cr$ range 20-40, blue and green respectively) and literature compositions for phosphate minerals (orange) are presented as vertical lines. A Solar System initial $^{53}Mn/^{55}Mn$ of 9.1 × 10^{-6} (Nyquist *et al.,* 2009), along with a conservatively estimated ^{53}Cr isotope enrichment in pallasite olivine rims of 3.5 ± 1.4 per mille and a bulk $^{55}Mn/^{52}Cr$ for late interstitial melts of 40-60 (bounded by olivine and high Mn/Cr phosphates) constrains the isolation of evolved silico-phosphate melt to before ~2.5 to 4 Myr (yellow field) followed by decay of ^{53}Mn to extinction (white field bound by olivine rims and phosphate minerals). The evolution of a planetesimal isolated from the nebula sufficiently early (*i.e.* with cooling and core-mantle redox-reactions occurring in the yellow region, before 2.5-4 Myr) is given on the right. Chronological constraints are from Mn-Cr (this study) and Hf-W data (Kruijer *et al.,* 2014).

environment (Boesenberg *et al.,* 2012). However, Mn is compatible in phosphates, while Cr is probably incompatible (Davis and Olsen, 1991; Hutcheon and Olsen, 1991). Therefore, olivine and the various phosphates constrain their parental melt to $^{55}Mn/^{52}Cr$ ~40-60. This melt may have occasionally been quenched to 'phosphoran olivine' under favourable conditions (Boesenberg *et al.,* 2012) in the terminal pallasite cooling event (Fowler-Gerace and Tait, 2015).

Because olivine $^{55}Mn/^{52}Cr$ is not high enough to generate detectable anomalies in-situ, Mn-Cr fractionation must occur after decay, as during crystallisation from such a silico-phosphate melt strongly enriched in Mn and hence eventually in $^{53}Cr^*$. A weighted mean of the $^{53}Cr/^{52}Cr$ for olivine rims (taking points with $^{55}Mn/^{52}Cr$ >20) gives a value of 0.11380 ± 0.00016 (95 % conf., Mean Square Weighted Deviation [MSWD] 2.4), or an enrichment of 3.5 ± 1.4 per mille. These data give a model-dependent Mn-Cr date for isolation at ~1 Myr after Solar System formation, with absolute upper limits of 2.5-4 Myr for a melt with $^{55}Mn/^{52}Cr$ in the range 40-60. This timescale confirms early accretion (0.1-0.3 Myr) and protracted core-mantle differentiation (to ~1.1-1.3 Myr) suggested by Hf-W chronologies for iron meteorite parent bodies (Kruijer *et al.,* 2014). Incomplete decay of ^{53}Mn, or low values of initial $^{53}Mn/^{55}Mn$ for the Solar System (Trinquier *et al.,* 2008), become difficult to accommodate. We therefore favour a higher Solar System initial $^{53}Mn/^{55}Mn$ (Nyquist *et al.,* 2009) and near complete decay of ^{53}Mn (~10 Myr at eutectic temperatures) for $^{55}Mn/^{52}Cr$ in the range 40-60. If isolation occurred earlier than indicated by Hf-W, then such constraints could be relaxed.

The history outlined above closely follows silicate-metal differentiation to form planetesimal cores at ~1.1-1.3 Myr after Solar System formation (Kruijer *et al.,* 2014). Elevated $^{53}Cr/^{52}Cr$ in Brenham olivine therefore indicates heating, core-mantle separation, and cooling-induced core-mantle back-reaction in the Main-Group pallasite parent body within ~2.5 to 4 Myr of Solar System formation. The latter requires cooling of olivine and liquid metal to the silico-phosphate and Fe-FeS eutectics around 1120-980 °C (Boesenberg *et al.,* 2012). Cooling to low-temperature eutectics therefore occurred rapidly. It suggests that even though cooling was initially fast, at perhaps 100-200 K Myr^{-1}, it slowed considerably at the phosphate eutectic (~1120 °C) and stayed near this temperature for at least several million years, allowing ingrowth of $^{53}Cr^*$ before quench-crystallisation. Trace element variations and clear charge-balancing relationships between Cr and slow-diffusing Al in olivine (McKibbin *et al.,* 2013b) indicates suppression of Cr diffusion. That Cr might move slower than divalent cations (Ito and Ganguly, 2006), and that low silica activity can suppress divalent (Zhukova *et al.,* 2014a) and trivalent cation diffusivity (Zhukova *et al.,* 2014b) suggests that in pallasites, trace element variations might not be erased at 1000 °C even over millions of years. This timescale, similar to or longer than the lifetime of ^{53}Mn, accommodates a cooling history comprising 1) rapid accretion, heating and core-mantle separation (Kruijer *et al.,* 2014); 2) rapid cooling to near-solidus temperatures followed by thermal buffering due to removal of crust, formation of regolith (Warren, 2011) and, potentially, crystallisation reactions (Morse, 2011; Namur *et al.,* 2014); and 3) final quenching to preserve disequilibrium assemblages (Boesenberg *et al.,* 2012; Fowler-Gerace and Tait, 2015).

Isolation of late silico-phosphate melt before ~2.5 to 4 Myr after Solar System formation implies that it represents one of the earliest silicate differentiation products, potentially predating almost all basaltic eucrites and angrites (Wadhwa *et al.,* 2009; Schiller *et al.,* 2010; Brennecka and Wadhwa, 2012; McKibbin *et al.,* 2015; Schiller *et al.,* 2015). Early cooling of the Main-group pallasite parent body to near solidus temperatures provides further evidence for early accretion of iron meteorite-type parent bodies, distinct from later forming ureilite- and chondrite-type parent bodies (*e.g.,* Budde *et al.,* 2015). Additionally, this process appears to have been reproducible: the presence of high $^{55}Mn/^{52}Cr$ phosphate in the Eagle Station pallasite (Davis and Olsen, 1991), which originated in a different parent body in a different part of the Solar System (Clayton and Mayeda, 1996) indicates that coexisting silico-phosphate and metal-sulphide-phosphide melts could be a general feature of planetesimal differentiation. Such phosphates and associated olivine rims (potentially phosphoran) should lend themselves to dating using Mn-Cr, enabling broad-scale mapping of the ^{53}Mn extinct nuclide chronometer to other systems and constraining the timing of core-mantle separations in planetesimals.

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Additional Information

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