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How does inherited zircon survive in partially molten mantle: Insights on modes of magma transport in the mantle from nanoscale melt-crystal interaction experiments



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ABSTRACT

Inherited zircons found in mantle-derived rocks are increasingly relevant for characterizing the history and petrogenetic processes of these rocks. However, the occurrence of inherited zircons in mafic igneous rocks poses an enigma because, as shown by numerous experiments combined with modelling studies, zircon, e.g., 150 µm size, is expected to dissolve extremely swiftly, e.g., c. 1 day, in the presence of mafic melts. Slow dissolution kinetics may explain zircon survival in fast ascending magmas that scavenged zircons near their final emplacement, but it cannot explain the persistence of inherited zircons that dwelled in the magma-bearing mantle for more extended periods. To understand how these zircons can survive over long times in contact with mafic melts, we performed a series of experiments, including some novel nano-scale experiments, to explore the behaviour of zircons in contact with different volumes of melt. Our results demonstrate that if melt occurs in narrow, spatially restricted domains around zircon grains, they get rapidly saturated with Zr. From this point onwards, the melt acts as a protective layer preventing further dissolution. These results carry important implications for the mode of melt transfer in mantle segments from which inherited zircons have been found: zircon crystals in partially molten mantle systems can only survive long in mush-like zones in which the melt migrates through porous flow, the wetting behaviour of basaltic melt on zircon would appear to further facilitate such survival. Zircon crystals would be rapidly consumed in high melt fraction regions of channelised (e.g., dike) flow. Such regions are therefore constrained to be short-lived (weeks to months) and pulsed in nature. Thus, the presence of zircons in mantle-derived rocks helps to place constraints on modes and timescales of melt transfer in the respective mantle segments.

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1. Introduction

Mantle-derived rocks often contain older-than-host zircons of either mantle or crustal origin that have survived in the mantle magma sources and the rocks derived from them. These include ultramafics (e.g., Bea et al., 2001; Fershtater et al., 2017; Xiong et al., 2011; Zheng et al., 2006); Mid-Atlantic MORB volcanic and plutonic rocks (Bea et al., 2020; Bortnikov et al., 2008; Pilot et al., 1998; Schwartz et al., 2010; Skolotnev et al., 2010); chromitites (e.g., Proenza et al., 2018; Pujol-Solà et al., 2020); carbonatites (e.g., Liu et al., 2008; Montero et al., 2016); kimberlites (e.g., Griffin et al., 2000; Valley et al., 1998); as well as in diverse ophiolitic and juvenile subduction-related igneous rocks (e.g., Rojas-Agramonte et al., 2016; Torró et al., 2018).

The discovery of inherited zircon in mantle-derived rocks is at odds with previous experimental works (Boehnke et al., 2013; Borisov and Aranovich, 2019; Borisova et al., 2020; Dickinson and Hess, 1982; Gervasoni et al., 2016; Shao et al., 2019) because these showed consistently that zircon suspended in mafic and ultramafic melts would dissolve in less than a few weeks (*Fig. 1A*), a conclusion that is fully supported by our own experiments that are reported here. Accordingly, a mechanism must exist to delay or prevent zircon dissolution in mafic systems. In principle, zircon xenocrysts entrained in magmas can survive either if zircon grains are physically prevented from reacting with the melt, i.e., included in early crystallized phases or xenoliths (*Fig. 1B*), or if they occur in environments that are locally saturated in zircon (*Fig. 1B*) (Bea and Montero, 2013 and references therein).



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Fig. 1. A) Numerical modelling of zircon dissolution suspended in a MORB melt, basaltic glass BG-1 used in experiments of present work. We performed the calculation for a zircon sphere of 150 µm radius in contact with a large reservoir of melt, 20 cm³, at different temperatures. Numerical modelling conditions are indicated in Supplementary material. B) Scheme of zircon stability in mafic-ultramafic molten systems in different scenarios: (i) surrounded by large melt volume; (ii) Shielded by magmatic or pre-magmatic phases, and (iii) in contact of low melt volume.

Both options seem plausible. However, considering that plastic flow and deformation involve recrystallization of major mantle minerals, even of zircon primocrysts (Zinger et al., 2010), it would be unrealistic to consider that zircon inclusions would be permanently shielded from the melt over the entire evolution of the magmatic system. This idea finds support from the fact that the few zircon xenocrysts observed in mafic rocks appear in intercrystalline positions and not as inclusions (Pujol-Solà et al., 2020; Schwartz et al., 2010). Therefore, one would expect zircons included early in primocrysts to have "seen" melt at some point in their history; if so, then a mechanism needs to be found to explain their survival.

To address this question, we have explored the behaviour of zircons in the presence of a restricted volume of melt through novel experiments and compared the results with those from increasing volumes of melts in contact with zircon. A situation with restricted volumes of melt may arise, for example, when melt transport occurs by porous flow (e.g., in a mush zone). In that case, a given zircon crystal would be in contact with a spatially restricted, and hence limited volume, of melt at any given point of time.

We have studied: (i) zircon grains surrounded by a large (infinite for our purposes) volume of melt, as could be the case in large magma chambers or even dikes, (ii) zircon grains embedded in peridotite powder suffering partial melting (iii) zircon grains in contact with a basaltic melt that is only a few hundred nanometers in extent, simulating melt volumes that exist during grain-scale melt percolation.

2. Materials and methods

2.1. Starting material

2.1.1. Zircon specimens

We have used three types of zircons (*Supplementary figure S1*). Two of them, Zrc-1 and Zrc-5, were gem quality zircon megacrysts up to c. 2.5 cm (Supplementary Figure S1A and S1B). These crystals were purchased in the Munich Gem Show-Mineralientage 2018. The mineral dealer did not know their exact origin, but we studied them at the IBERSIMS laboratory from the University of Granada equipped with a SHRIMP IIe/mc ion microprobe. The megacrysts were mounted in thermoplastic acrylic and polished stepwise using first diamond grinding discs, and then diamond paste abrasives of 3 µm and 0.25 µm. Then they were cut in either 1 mm or 2 mm edge cubes that were randomly oriented. The third kind of zircon consisted of a purified grains concentrate separated from the tonalite SAB51 from Central Iberian Zone. Iberian Massif (Bea et al., 2018). Most grains were 100 to 150 um long and 50 to 70 um wide prisms that were terminated at either extreme by short pyramids. Details of zircon specimens are provided in the **Supplementary** Material and Supplementary Table S1.

2.1.2. Basaltic glass

A synthetic glass of basaltic composition was used for the experiments. The components used for glass synthesis were chosen to obtain a composition similar to the average of basalts from East Pacific Rise (EPR), Mid-Atlantic- Ridge (MAR), and Central Indian Ridge (CIR) as given in Winter (2010). Details of the experimental procedure are indicated in *Supplementary Material*, and the composition of the so-obtained glass (BG-1), analyzed in an Electron Microprobe is reported in *Supplementary Table S2*

2.1.3. Peridotite matrix

The peridotite matrix was prepared with material collected from the Ronda ultramafic massif (e.g., Obata, 1980). The starting lherzolite (RON-1) was obtained by mixing optically clean mineral separates from a coarse granular-spinel lherzolite which were handpicked under a binocular microscope. Mineral proportions of RON-1 were 67.9 wt% olivine, 23.5 wt% orthopyroxene, 7.6 wt% clinopyroxene, and 1.3 wt% spinel (*Supplementary Figure S2* and *Supplementary Table S3*). This approach was taken to avoid serpentinized sections that were present in the natural sample because the addition of such water-rich components alters the melting behaviour of a rock core/powder. Details of selected sample are provided in *Supplementary Material*.

Table 1

Run conditions of experiments in the present study. In all cases pressure was 1 bar. BG1: MORB basaltic glass; RON-1: Starting Iherzolite.

Run number	Experiment Type	capsule	T (°C)	log fO ₂	Duration (h)	Zircon specimen	Zircon cube side (mm)	Starting material	Comments
Exp-6	Type-Ia	Au ₇₅ -Pd ₂₅	1200	-9	24	Zrc-1	1	BG-1	Zircon embed in an effectively infinite volume of melt
Exp-8	Type-Ib	Au ₇₅ -Pd ₂₅	1200	-9	24	Zrc-1	1	BG-1+RON-1	Zircon embed in a peridotitic matrix near to basaltic glass BG-1 layer. Thick layer of RON-1 and BG-1 glass were 2/3 and 1/3 of total thickens of the capsule respectively
Exp-15	Type-II	Au ₇₅ -Pd ₂₅	1200	-9	168	SAB-51	100×300°	RON-1	Zircon embed in a peridotitic matrix (proportion 1:20). Low degree of partial melting conditions
Exp-26A	Type-III	Pt	1300	-8	0.05	Zrc-1	2	BG-1 thin-film	
Exp-26B					0.05	Zrc-5			
Exp-27A					0.16	Zrc-1			
Exp-27B					0.16	Zrc-5			
Exp-28A					0.5	Zrc-1			
Exp-28B					0.5	Zrc-5			
Exp-29A					1	Zrc-1			
Exp-29B					1	Zrc-5			Zircon in contact with
Exp-30A					10	Zrc-1			low melt volume
Exp-30B					10	Zrc-5			
Exp-21A					15	Zrc-1			
Exp-21B					15	Zrc-5			
Exp-31A					24	Zrc-1			
Exp-31B					24	Zrc-5			
Exp-32A					48	Zrc-1			
Exp-32B					48	Zrc-5			

Units in µm of irregularly shaped grains from SAB-51 tonalite.

2.2. Experimental procedure

The run conditions of all experiments are provided in *Table* 1.

2.2.1. Type-I experiments

Two type-I experiments were done to evaluate the dissolution of a zircon grain surrounded by an effectively infinite volume of melt, as could be the case in a large magma chamber or a dike:

Type-Ia: One mm edge zircon cubes were loaded into Au₇₅-Pd₂₅ crucibles embedded within finely ground powder of basaltic glass BG-1, leaving the top of the capsule open. This arrangement and an example of a crystal before the experiment is shown in *Fig. 2A*.

Type-Ib: Identical 1 mm edge zircon cubes were embedded in a peridotitic matrix surrounded by basaltic glass BG-1. To make a reaction couple, we first packed some powder of the peridotite RON-1 in the lower part of an Au_{75} -Pd₂₅ capsule surrounding the zircon cube and then filled the remaining part of the capsule with the basaltic glass powder, leaving the top of the capsule open. The experimental setup and an example of a crystal before the experiment are shown in *Fig. 2B*.

The experiments with these setups were performed in a vertical tube furnace at atmospheric pressure. The temperature was monitored with a B-type thermocouple placed within 2 cm of the sample container. The samples were annealed for 24 h at 1200 $^{\circ}$ C, which represents the calculated liquidus temperature at 1 atm of BG-1 basaltic glass (**Supplementary Material**). The oxygen fugacity was set by a flowing gas mix of CO-CO₂ to 10^{-9} Pa (~ QFM+0.5 buffer) (**Table 1**). The recovered samples were mounted in thermoplastic acrylic, and polished stepwise using diamond grinding discs to finer diamond paste of 3 µm and 0.25 µm abrasives up to central perpendicular section of the cylindrical axis. They were examined qualitatively under reflected light microscope and scanning electron microscope. The objective of these experiments were to study the behaviour of zircons in such systems (and to ensure that such behaviour is consistent with that predicted from model calculations, see below), and not to study the details of melt percolation and reaction.

2.2.2. Type-II experiments

Type-II experiments were carried out embedding SAB-51 zircon grains in the peridotite RON-1 powder in a proportion of 1:20 (*Fig.* 3) and loading the mixture into an Au75-Pd25 crucible with the top of the capsule open (*Fig.* 3A). The idea was to simulate a low degree of partial melting to keep at the minimum the melt volume in contact with each zircon grain. The sample was annealed in the vertical tube furnace, following the same procedure as Type-I experiments, at 1200 °C and 1 atm for seven days (*Table* 1). The selected temperature was near solidus conditions (*Supplementary Material*) to ensure the generation of low melt fraction.



Fig. 2. Schematic and results of Type-I experiments, where zircon was placed in contact with a large volume of melt. Scheme of A) Type-Ia and B) Type-Ib experimental configuration and zircon crystals before and after annealing.

2.2.3. Type-III experiments

Type-III experiments were carried out using 2 mm edge zircon cubes in contact with a minimal melt volume. This was created by depositing a film of the basaltic glass BG-1 on the polished zircon cube face by pulsed laser deposition (PLD method - see Dohmen et al., 2002 for details of the method) (*Fig. 4A*). The thickness of the film was on the order of a micrometer (900-1000 nm), the idea being to create the lowest possible melt volume surrounding the zircon, but still enough to be studied after annealing. Thin-film couples were prepared on Zrc-1 and Zrc-5 cubes. We also deposited a film of basaltic glass on a graphite substrate that was used as a blank during EPMA measurements. The zircon and graphite substrates were heated to c. 400 °C in a vacuum at 6×10^{-3} bar before the deposition to remove any adsorbed water and other contaminants on the surface. Samples were annealed in a vertical tube furnace, following the same procedure as Type-I experiments but increasing the temperature to 1300 °C in Pt crucibles to avoid the possible effect of the melt-derived phases on the zircon dissolution process (i.e., the temperatures were raised above the liquidus of the melt composition). Note that this would facilitate dissolution of zircon even more compared to Type-I and Type-II experiments (i.e., the survivability of zircons is subjected to a more stringent test).

Care was taken to ensure that the melt layer was not in contact with the crucible material (Pt), thus avoiding the effect of iron diffusion between this and the melt (**Fig. 4A**). We did different runs with durations from 3 min to 48 h (**Figs. 5 and 6**), keeping a flow gas mix of CO-CO₂ to set the oxygen fugacity at 10^{-8} Pa (\sim QFM+0.5 buffer) (**Table 1**). Each experiment was duplicated using two different crystals isolated from each other in the same crucible. These were labelled as cases A and case B and served to check the reproducibility of results using independent samples (**Table 1**). After the experiments, samples were stored in a clean desiccator before being analyzed with the EPMA in the field emission (FE) mode.

2.3. Analytical methods

2.3.1. Scanning electron microscope

All experimental samples were carbon coated and have been studied under a high-resolution field emission scanning electron microscope (HR-FESEM) of type LEO/ZEISS 1530 Gemini using a backscatter detector (BSD) at the Ruhr-Universität Bochum and at the Deutsches Bergbaummuseum Bochum, Germany.

2.3.2. Electron microprobe analyses

Quantitative chemical measurements and X-ray element distribution maps were measured using a field-emission (FE) electron microprobe CAMECA "SX FiveFE" at Ruhr-Universität Bochum. The microprobe was equipped with five wavelength-dispersive (WD) spectrometers and an energy dispersive SD detector. As melt droplets are very thin (<1 µm, see below), we used an acceleration voltage of only 5 kV to achieve an excitation volume that does not extend into the zircon substrate, so that signals may be obtained from the near surface region that are much shallower than the depths from which signals are obtained in conventional electron microprobe analysis (\sim 3 µm). The probe current of the fully focused beam was 150 nA. These beam conditions were carefully checked to avoid potential time-dependent X-ray intensity changes. We measured the Zr L α line on 4 spectrometers simultaneously with t = 90 s on peak and t = 60 s on the background. In addition, Si K α was determined. For quantification of both elements, natural zircon was used as reference material. Further elements contained in the glass (Fe, Ti, Ca, K, P, Al, Mg, Na O) were pre-defined as matrix. The matrix correction procedure X-Phi (Merlet, 1994) was used for quantification of the raw data. X-ray element distribution maps were also acquired at 5 kV acceleration voltage and 150 nA probe current with Zr L α on 4 WD spectrometers and Mg K α . Additional elements Na. Al. Si and Ca were mapped using the energy-dispersive system. All element maps have a resolution of 1024×768 pixels, a step width



Fig. 3. Type-II experiments, designed to produce a low amount of melt in contact with zircons. A) Schematic diagram of a run capsule. Note how zircons of 150-300 µm size were disseminated in the RON-1 peridotitic matrix. B) Cathodoluminescence images of zircons from Central Iberian Zone, Iberian Massif, tonalite SAB-51. Experimental results show a harzburgitic matrix where zircon presents dissolution textures, which is a function of melt volume in contact C) and D). E) Low degree of dissolution of zircon. F) Inset of previous crystal where melt pocket is trapped between olivine and zircon. Zircon has dissolution of few microns from external edge.

of 0.1 μ m and a dwell time of 35 ms per pixel. The XR images were processed with DWImager software (García-Casco, 2007). The processed images show the pixel-sized analyses of melt and zircon (colour-coded, expressed in Zr (L α) counts), masking out the phase of interest in each case, overlaid onto the corresponding BSE image (grayscale base-layer) showing the basic textural features of the scanned areas. We used DWImager to extract compositional profiles in the studied areas.

3. Results

3.1. Type-I experiments

Type-I experiments reproduce the conditions in which zircon grains are dispersed in a large volume of basaltic melt. In the two subtypes of experiments, the zircon crystals were heavily dissolved, although with significant differences. After the experiments the sizes of the zircon grains were measured and compared to initial sizes to estimate extents of dissolution. Sectioning effects may lead to shorter dimensions (e.g., if the central portion of a grain is not exposed) or longer dimensions (e.g., if a diagonal section is obtained) that may lead to an over- or under-estimate of the extent of dissolution, respectively. This adds some uncertainty to the results. To exclude the possibility that such artefacts might affect our conclusions, we also carried out a few experiments with precisely measured cubes of zircons that were then retrieved in 3D and the sizes were measured after the experiments. Pressure is not expected to play a major role in controlling the solubility of zircon in mafic melt (Boehnke et al., 2013; Shao et al., 2019; Watson and Harrison, 1983), and this expectation is supported by some preliminary experiments that we carried out in a multianvil apparatus.

Type-la experiments simulate zircon grains surrounded by an infinite volume of melt. The zircon-melt interface does not contain magmatic phases other than tiny ($<30 \ \mu$ m) acicular plagioclase that crystallized during quenching. After 24 hours at 1200 °C, zircon crystals, placed in the middle of the capsule surrounded by basaltic glass, present dissolution textures, such as rounded edges (*Fig. 2A*). The 2D shape measurements after annealing suggest a volume reduction of 35-60% with respect to the starting crystal.

Type-Ib experiments were focused on the behaviour of zircon in a basaltic melt-peridotite interaction process. Melt rock interaction produces a gradational reaction zone ranging from duniteharzburgite, in the region near the melt rock surface, to the original lherzolite, similar to those found in previous experiments under similar conditions (e.g., Tursack and Liang, 2012; Wang et al., 2013a). Regardless of the melt rock interaction process, i.e., the kind of lithologies found along the length of the capsule, zircon was in contact with significant amounts of melt although it was partially shielded by mineral phases of the peridotite. Zircon was placed in the dunite region, composed of sub-spherical olivine (97 vol.%) with a grain size of 25–150 µm and Cr-rich spinel (<1 vol.%,



Fig. 4. A) Scheme of Type-III experimental configuration. Note the thin film of basaltic layer of about 1 μ m thickness deposited on the zircon substrate using Pulsed Laser Deposition (PLD). The zircon and basaltic melt experimental couples were located in a platinum crucible and the experiments were conducted in a gas mixing furnace at 1300 °C and oxygen fugacity conditions of QFM+1.5. B) Backscattered images of plan view of samples before and after annealing. Note in the inset the globules of melt generated on top of the crystal. C) White light interference microscope study of Type-III experiments. We discriminated the zircon substrate (blue) and melt globules (red) to show the effective volume of melt globules. Moreover, we masked out the melt globules showing the height value (rainbow scale). The results indicate an average height of basaltic melt globules of about 0.72 μ m for the whole area as it is illustrated in the representative profile I-II. (For interpretation of the colours in the figure(s), the reader is referred to the web version of this article.)

10–50 μ m), and it was surrounded by basaltic glass (c. 40–60 vol.%) (*Fig. 2B*). Zircon shows heterogeneous dissolution, being more pronounced where more liquid is in contact with the crystal (*Fig. 2B*). The presence of olivine crystals slows down the zircon dissolution process compared to Type-Ia experiments so that the volume of the initial crystals reduces only to 60–80% after 24 h at 1200 °C. These experimental results suggest that the extent of dissolution of zircon depends on the volume of melt in the system, as will become clearer through the other kinds of experiments described below.

3.2. Type-II experiments

Type-II experiments, conducted at atmospheric pressure, were designed to produce a low amount of melt and study the effects of small basalt volumes on the textures of crustal zircons. Instead of a large crystal, we used natural zircon grains from sample SAB-51. The duration of the experiment was increased to seven days to

ensure that complete dissolution of zircon would occur, based on the results of the Type-1 experiments if dissolution rates remained the same. Then the presence of zircon illustrates the reduction of rates controlled by only the melt volume in contact (Fig. 3A and Table 1).

After annealing, the main peridotitc matrix of the experimental charge was composed of mineralogy that corresponded to a depleted peridotitic matrix rather than the staring lherzolite (i.e. the assemblage evolved towards harzburgitic compositions). It is composed of olivine (70 vol.%), orthopyroxene (25 vol.%), clinopyroxene (4 vol.%), spinel (<1 vol.%) and melt (2 vol.%) (**Fig. 3C and 3D**). The texture of the zircon grains changed depending on the volume of the neighbouring melt phase. If the volume was large, zircons showed pronounced dissolution textures (**Fig. 3C**). On the contrary, if the melt volume was small, the intensity of zircon dissolution decreased significantly (**Fig. 3D and 3E**), forming small melt pockets isolated between the zircon and other matrix phases, mainly olivine (**Fig. 3F**). Once again, these results suggest that the extent

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Fig. 5. X-ray maps of $Zr(L\alpha)$ counts from Type-III experiments including: A) Zircon crystal not covered with a film; B) Thin film coated crystal before annealing; C) Thin film coated graphite disk to be used as a blank. Experimental results showing the Zr content of zircon substrate, globule and profile extracted using DWImager software of: D) to F) 1 hour; G) to I) 10 hours; J) to L) 48 hours experiments.

of dissolution of zircon is controlled by the amount of melt that has access to the surfaces of a zircon crystal.

3.3. Type-III experiments

Type-III experiments were designed to evaluate the effects of very low melt volumes in contact with a zircon crystal. To produce such conditions, we used the PLD technique. This is a novel methodology in the study of the stability of zircon in silicate melts. This arrangement and an example of a crystal before the experiment is shown in **Fig. 4A**

After annealing, the samples were macroscopically unchanged, but microscopic observations showed that the surface was covered with tiny round glass (= ex-melt) globules (diameter about 10-20 μ m, height about 0.72 \pm 0.02 μ m, and the globules made up 20% of the surface area, characterized using a white light interference microscope (see Dohmen et al., 2002 for a description of the equipment and details of the method) (*Fig. 4B and 4C and Supplementary Figure S4*). This effect results from the wetting characteristics of the basaltic melt, which tends to form droplets rather than a uniformly spread-out film over the zircon surface. Moreover, as seen in Fig. 4C, the walls of the droplets make an angle of almost 90° with the surface of zircon (the angle is much higher



Fig. 6. A) Results of electron microprobe analysis (EMPA) in the field emission (FE) mode of (i) zircon cubes, for, Zrc-1 and Zrc-5 specimens, not covered with a film (horizontal grey bar corresponds to standard Zr content of Zircon c. 490000 ppm); (ii) thick film coated crystals before annealing, diamonds, including a blank made with a graphite substrate, cubes (horizontal grey bar corresponds to Zr content of basaltic glass); and (iii) melt globules generated after annealing at different run times. Each experiment with different colours. All experiments were duplicated and these are designed by case A and case B, respectively. Error bars are at 2σ . For all cases the detection limit of each measurement is also shown as a purple plus symbol. Zr weighted means for all measurements of globules for each experiment are also indicated (box on top of Fig. 6A(ii)). We included the zircon saturation conditions at experimental conditions, horizontal grey bars, considering the BG-1 basaltic glass composition used: WH83 (Watson and Harrison, 1983); B13 (Boehnke et al., 2013); G16 (Gervasoni et al., 2016); S19 (Shao et al., 2019); BA19 (Borisov and Aranovich, 2019). We indicated the compositional factor used in each model: M (Boehnke et al., 2013; Watson and Harrison, 1983); G (Gervasoni et al., 2016); B (Borisov and Aranovich, 2019). B) Zr (ppm) vs SiO₂ (wt%) of same cases as in A. Note that vertical grey bars correspond to (i) SiO₂ (wt%) content of zircon and (ii and iii) the initial SiO₂ (wt%) content and analytical error for analysis of basaltic glass.

than the limiting value of 60° in any case that is necessary for wetting behaviour). This observation – the formation of droplets with near vertical walls from an initial continuous amorphous film – demonstrates directly that basaltic melt does not wet the surface of zircon. We note here that dihedral angles have been measured and used in the petrological literature as a measure of wettability and interconnectivity of melts/fluids because such direct measurements were not possible before (and remain impossible in samples from high pressure experiments, which are mostly relevant for melt generation in the interior of the Earth). The connection between observations of drops on a surface and dihedral angles have been elucidated in the materials science literature (e.g. Smith, 1948; Cassie and Baxter, 1944; Parikh and Humenik, 1957) and the adaptation to petrological systems explained in early studies such as Waff and Bulau (1979) and Mckenzie (1984). An early attempt to measure the interfacial angle directly for a petrological system was that for basalt on an olivine surface by Cooper and Kohlst-edt (1982). They placed a layer of basalt powder on olivine and studied the angle made by melt droplets. The ability to produce continuous amorphous thin films that are well-bonded to the surface, and to analyze the results using nanoscale observation tools, makes such experiments much more controlled and feasible now.

The globules only appeared in runs of durations more than c. 10 minutes (*Supplementary Figure S4*), indicating that this is the

time required for the melt to reach textural equilibrium with the surface. This observation also ensures that the initial configuration is a continuous melt film on zircon.

To characterize the chemistry of the melt droplets, we used electron microprobe analysis (EMPA) in the field emission (FE) mode. We studied thick film-coated crystals before and after annealing and compared them with (i) an uncoated zircon and (ii) a thin melt layer deposited on a graphite substrate to determine the signal that comes from the film only i.e., this sample was used as a blank. *Fig. 6A*(*i*) shows the detection limit of Zr and the composition of a zircon surface that is not covered with a film. *Figs. 6A*(*ii*) *and 6A*(*iii*) show the compositions of thick film coating zircon crystals before and after annealing, respectively. The analytical results are presented in *Supplementary Table S4*.

Studies of the elemental distribution maps of Zr, obtained using the Zr-L α lines in a Cameca SX Five FE electron microprobe, show the contrasting nature of Zr distribution on the surfaces of the different samples. (Fig. 5). Zircon crystal not covered with a film shows high Zr, as expected (Fig. 5A). Before annealing, basaltic layer of the thin film coated crystal shows very low Zr, similar to the film deposited on the graphite substrate; this indicates that there is a negligible contribution of the Zr-signal from the zircon substrate to the measurements on thin films (Figs. 5B and 5C). The Zr-content of the melt globules is notably lower than that of the zircon substrate (Figs. 5D to 5L), but higher than that of the film of the unannealed sample or in the blank sample (Fig. 6). The Zr concentration in melt globules seems constant over the entire area (Figs. 5D to 5L). Only in the contact regions between melt globule and zircon substrate is it possible to observe a diffusion-like pattern, which in this case is an expected analytical artefact caused by convolution (e.g., Ganguly et al., 1988). This shows that in the inner part of the globules, the region of interest, the measurement of the Zr content does not show a contribution from the zircon substrate (Figs. 5D to 5L).

Fig. 6A(i) shows the detection limit of Zr, and the composition of a zircon surface that is not covered with a film. Figs. 6A(ii) and 6A(iii) show the compositions of thick film coated crystals before (including a "blank" made of a film on a graphite substrate) and after annealing, respectively. The analytical results are presented in Supplementary Table S4. It is demonstrated that before annealing, essentially the basaltic glass is analysed by this technique and the Zr-content is on the order of magnitude of the detection limit, as it should be (Fig. 6A(ii)). These concentrations increase after the anneal (Fig. 6A(iii)). Also shown on this figure are values of zircon concentration in basalt that would be expected according to different experimental datasets on dissolution of zircon. In short duration experiments, where no melt globules were generated, the basaltic layer was so thin that the measurements showed a marked Zr contribution from the substrate (Fig. 6A(iii)). However, in the experiments where globules formed (of durations more than 30 min), the melt globules are thick enough to prevent contributions from the substrate showing through. In these cases, we got a constant Zr concentration of about 16000 ppm for all globules (Fig. 6A(iii)). Two main observations may be derived from these data. First, the measurements done on different globules yielded the same concentrations of Zr. Second, this concentration remains constant with increasing run duration (Fig. 6A(iii)). Remarkably, the constant measured values agree perfectly with those that would be predicted by the Borisov and Aranovich (2019) model, thus providing additional corroboration of the measurement technique. Plots of compositions from different regions (zircon, melt globules) on Zr-SiO₂ diagrams (*Fig.* 6B) confirm that this method provides reliable chemical analyses of such sub-micrometer thick films, even when they sit next to a source with high Zr-content, such as zircon.

4. Discussion

Type I experiments confirm the fast zircon dissolution in silicate melts calculated by many authors using numerical methods (e.g., Bindeman and Melnik, 2016; Harrison and Watson, 1983; Watson, 1996) (*Fig. 1A*, numerical calculation procedure is indicated in *Supplementary Material*). Type-Ib experiments show a smaller extent of zircon dissolution than Type Ia (*Fig. 2*), likely caused by the partial shielding of zircon by olivine, which reduces the amount of melt in contact with zircon (*Fig. 2B*). This effect might help to explain the occurrence of zircon in some short duration natural processes such as rapid mafic dyke intrusion, i.e., kimberlites, carbonatites, lamproites, lamprophyres. However, this mechanism only slightly enhances the lifetime of zircon grains and cannot explain the occurrence of zircon in systems where mafic melts reside over long durations.

Type-II experiments simulate low degree of partial melting of a peridotite matrix (*Fig. 3*). As in Type-I experiments the volume of melt controls the survival of zircons dispersed in the matrix.

Type-III experiments reveal that when a thin layer of melt is placed in contact with zircon, (i) the wetting behaviour of the melt leads it to form isolated droplets rather than a continuous film, reducing the contact area between zircon and melt, and (ii) the initial rapid dissolution gets retarded as the limited volume of melt layer gets saturated in Zr. The Zr content to saturate small melt reservoirs is in better agreement with predictions of the Borisov and Aranovich (2019) model rather than older models (Fig. 6) because that experimental study used mafic bulk compositions, analogous to our system, to obtain the parameters for compositional and T dependence. Thus, in the present work we demonstrate how small melt volumes of mafic melt can become zircon saturated at high temperatures even at near liquidus conditions and once saturation is reached, how the melt layer actually acts as a protective, rather than a corrosive, layer.

The consistency of experimental results allows us to extend these observations to other different relative volumes of zirconmelt. *Fig. 7A* shows the numerical calculations of how the lifetime of zircon crystals of different sizes depends on the size of the melt volume that surrounds it. At $1200 \,^{\circ}$ C, if a melt reservoir larger than about 1 mm³ is in contact with a spherical zircon grain of 150 µm of radius, it dissolves away within a timescale of a few days. As the size of the melt reservoir decreases, the lifetime of a zircon crystal increases dramatically, so that zircon crystals c. 150 µm are stable for unlimited time in melt reservoirs smaller than c. 0.30 mm³ (*Fig. 7A*).

Fig. 7B uses such calculations to provide a comprehensive map of timescale of survival of zircon grains of different initial sizes in melt reservoirs of different volumes. The figure also shows the final grain sizes of zircons that would be preserved in each case. It is seen that even relatively moderate sized zircon grains that are on the order of 10's – 100's of microns in size would survive indefinitely long if the surrounding melt reservoir is not larger than about one mm³. The method used for these calculations can be used for any other temperature, melt composition, or zircon grain size.

The results from all our experiments of different kinds taken together demonstrate that the melt volume in contact with a zircon grain is the most critical parameter controlling the preservation of zircons in high-temperature mafic systems. Thus, the fact that zircons are being increasingly found in high temperature ultramafic and mafic rocks that come from the mantle, the ocean floor and other tectonic settings indicates that the zircons could not have been in contact with large volumes of melts for periods that are longer than only a few days at most. In agreement, when inherited zircons are preserved in ultramafic-mafic rocks, like gabbros, chromitites or dunites, they show clear dissolution



Fig. 7. A) Numerical calculation to evaluate the dissolution process of a spherical zircon, 150 µm radius, suspended in different volumes of NMORB basaltic melt, BG-1 glass composition, at 1200 °C. Note that in a reservoir larger than about 0.65 mm³ the zircon grain is dissolved in a few days, whereas for a melt volume reservoir smaller than 0.64 mm³ zircon is preserved for the entire duration shown in the plot. B) Timescale of survival of zircon grains of different initial sizes in different melt reservoir volumes of MORB basaltic melt, BG-1 glass, at 1200 °C. This graph shows the time required to saturate the volume of mafic liquid indicating the radius of the remaining zircon. This calculation shows the critical volume where the zircon is completely dissolved.

textures with corrosion gulfs or partially digested textures (Bea et al., 2001; Bortnikov et al., 2022; Proenza et al., 2018; Pujol-Solà et al., 2020; Rojas-Agramonte et al., 2016; Schwartz et al., 2010), indicating that even when preserved, an initial phase of dissolution took place. Even for systems such as kimberlites that evolve rapidly, the time of transit from the mantle is thought to be on the order of a few days to weeks (e.g., Russell et al., 2012; Wilson and Head, 2007). According to our results, if the zircons were exposed to high-temperature melts for much longer in the source region, they would have dissolved away completely. *Fig. 8* sum-

marizes a mid ocean ridge setting in which inherited zircons have been found and illustrates the possible modes of melt transport in the mantle that are discussed in the literature (e.g., Aharonov et al., 1995; Kelemen et al., 1995; McKenzie, 1985; Spiegelman et al., 2001; Stracke et al., 2006). Based on the results of this study, it is apparent that in regions with channelized melt flow or regions of melt-pooling, zircon grains are unlikely to survive for significant lengths of time (Fig. 8). On the other hand, grain-scale porous flow where individual melt batches are restricted to sub-millimeter, and more likely micro- to nanometer scale grain boundary regions, are ideal for the preservation of zircon in high temperature partially molten systems (Fig. 8). The surface tension properties of the meltzircon system would also help the process because the melt clearly prefers not to wet a zircon surface (see experimental results above, Figs. 4 and 5, and Supplementary Figure S4). Therefore, we argue that the widespread finding of zircons in mafic-ultramafic systems are direct physical evidence of widespread porous flow or existence of largely crystalline mush zones in diverse mantle settings. Additionally, the observations constrain the duration of existence of large volumes of melt pools (e.g., in channels, dikes, melt-rich zones) in mantle settings to below a few days to weeks. Longer residence of zircon grains in such settings would dissolve them. In combination, the observation of zircons in mafic-ultramafic systems and the kinetics of their dissolution behaviour indicate that melt flow/melt-rock interaction in such settings occur by porous flow with only short-lived pulses of melt propagation via dikes, channels and formation of large melt pools (e.g., during compaction processes).

5. Conclusions

The high solubility of zircon in mafic melts makes it unstable in partially molten mafic-ultramafic systems which are generally deeply undersaturated in zircon, so that this mineral dissolves in a matter of weeks. Although some zircons may have survived because of short residence in high temperature settings (e.g. Bea et al., 2020), this explanation does not apply to zircons that have travelled long in the deep mantle - for example, zircons recycled through subduction (e.g., Bea et al., 2001; Proenza et al., 2018; Rojas-Agramonte et al., 2016; Wang et al., 2013b) must have been in contact with melt over a protracted history.

Our experimental results confirm the high solubility of zircon. Dissolution times do increase when a crystal is shielded by other phases, but not substantially. Novel experiments where zircon crystals (several mm in size) were placed in contact with a very limited volume of mafic melt (a film of few 100 nanometers thickness, deposited by pulsed laser deposition (PLD) indicate that initial rapid dissolution of zircon gets gradually retarded because the melt prefers not to wet a zircon surface and the limited melt volume in contact with the zircon gets saturated. Once saturation is reached, the zircon-saturated melt layer around the zircon protects it from further dissolution or reaction with other phases. With this mechanism, zircon can survive indefinitely in the presence of a small amount of mafic melt (locally, in contact with zircon), as is expected in regions of porous flow or mush-like cumulates. The wetting properties of basaltic melt further facilitate such survival. In contrast, if the magma segregates into melt-rich channels, the chances of survival for zircon xenocrysts decrease dramatically. This, in turn, places constraints of a maximum of a few weeks to a month (depending on the size of zircons, extent of shielding by other phases) on the likely lifetimes of such channelized melt flow zones in mantle regions where inherited zircons are found.



Fig. 8. A) Map of the age of the oceanic igneous crust, from Müller et al. (2008), indicating the location of inherited zircon occurrence in mid-ocean ridge mafic-ultramafic rocks (Bea et al., 2020; Pilot et al., 1998; Schwartz et al., 2010). The active mid-ocean ridges are also indicated: MAR: Mid-Atlantic Ridge; SWIR: Southwest Indian Ridge; SEIR: Southeast Indian Ridge; CIR: Central Indian Ridge; PAR: Pacific-Antarctic Ridge; East Pacific Rise: EPR; Juan de Fuca Ridge: JDF; Galápagos Spreading Centre: GSC. B) Scheme of melt generation and transport from the mantle to crust in Mid Ocean Ridge setting, modified from (Brandl et al., 2016). We include schematic descriptions of channelised flow and porous flow melt transport and the behaviour of zircon in contact with the melt in each case. We also included the same scheme for the mush zone and magma chamber in the upper crust.

CRediT authorship contribution statement

Aitor Cambeses: Formal analysis, Funding acquisition, Investigation, Methodology, Project administration, Resources, Software, Validation, Visualization, Writing – original draft, Writing – review & editing. **Sumit Chakraborty:** Conceptualization, Formal analysis, Funding acquisition, Methodology, Project administration, Resources, Software, Supervision, Validation, Visualization, Writing – original draft, Writing – review & editing. **Niels Jöns:** Resources, Visualization, Writing – original draft, Writing – review & editing. **Pilar Montero:** Resources, Validation, Visualization, Writing – original draft, Writing – review & editing. **Fernando Bea:** Conceptualization, Formal analysis, Methodology, Software, Supervision, Validation, Visualization, Writing – original draft, Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

No data was used for the research described in the article.

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Appendix A. Supplementary material

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Further reading

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