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Bonding of xenon to oxygen in magmas at depth

Clémence Leroy^a, Chrystèle Sanloup^{b,*}, Hélène Bureau^a, Burkhard C. Schmidt^c, Zuzana Konôpková^d, Caroline Raepsaet^e

^a Sorbonne Universités, UPMC Univ Paris 06, CNRS, Institut de Minéralogie, de Physique des matériaux et de Cosmochimie, 75005 Paris, France

^b Sorbonne Universités, UPMC Univ Paris 06, CNRS, Institut des Sciences de la Terre de Paris, 75005 Paris, France

^c Goettingen University, Geowissenschaftliches Zentrum, 37077 Goettingen, Germany

^d DESY Photon Science, Notkestr. 85, 22607 Hamburg, Germany

^e NIMBE, CEA, CNRS, Université Paris-Saclay, CEA Saclay, 91191 Gif-sur-Yvette, France

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ABSTRACT

The field of noble gases chemistry has witnessed amazing advances in the last decade with over 100 compounds reported including Xe oxides and Xe-Fe alloys stable at the pressure-temperature conditions of planetary interiors. The chemistry of Xe with planetary materials is nonetheless still mostly ignored, while Xe isotopes are used to trace a variety of key planetary processes from atmosphere formation to underground nuclear tests. It is indeed difficult to incorporate the possibility of Xe reactivity at depth in isotopic geochemical models without a precise knowledge of its chemical environment. The structure of Xe doped hydrous silica-rich melts is investigated by in situ high energy synchrotron X-ray diffraction using resistive heating diamond anvil cells. Obtained pair distribution functions reveal the oxidation of Xe between 0.2 GPa and 4 GPa at high T up to 1000 K. In addition to the usual interatomic distances, a contribution at 2.05 ± 0.05 Å is observed. This contribution is not observed in the undoped melt, and is interpreted as the Xe-O bond, with a coordination number of about 12 consistent with Xe insertion in rings of the melt structure. Xe solubility measurements by electron microprobe and particle induced X-rays emission analysis confirm that Xe and Ar have similar solubility values in wt% in silicate melts. These values are nonetheless an order of magnitude higher than those theoretically calculated for Xe. The formation of Xe–O bonds explains the enhanced solubility of Xe in deep continental crust magmas, revealing a mechanism that could store Xe and fractionate its isotopes. Xenon is indeed atypical among noble gases, the atmosphere being notably depleted in elemental Xe, and very strongly depleted in Xe light isotopes. These observations are known as the 'missing' Xe paradox, and could be solved by the present findings.

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1. Introduction

Noble gases are key tracers of Earth dynamics, and as such, Xe isotopes are important markers of planetary and atmosphere formation (Staudacher and Allègre, 1982; Pepin, 2006); they may also be used to track underground nuclear tests (Hourdin and Issartel, 2000). In particular, the I–Pu–Xe system is used to date the formation of the atmosphere (Ozima and Podosek, 1999; Avice and Marty, 2014) due to the very short half-lifes of ¹²⁹I (17 My) and ²⁴⁴Pu (82 My). Degassing of the early Earth could thus have massively contributed to the formation of the terrestrial atmosphere within the first 100 Myr (Staudacher and Allègre, 1982). However, recent Monte Carlo simulations pointed out that given the uncer-

* Corresponding author. *E-mail address:* chrystele.sanloup@upmc.fr (C. Sanloup). tainties on Xe isotopic compositions, such a catastrophic early degassing event could not be evidenced unambiguously, and that the potential chemistry of Xe at depth must be solved first (Boehnke et al., 2015).

Xe abundances and isotopic ratios have been measured in a large variety of samples (atmosphere, fluids, rocks) and from different geological contexts, from Archean rocks (Pujol et al., 2011) to Martian meteorites (Gilmour et al., 1998). From these measurements, it was pointed out as early as 1970 that Xe is missing from the atmosphere of the Earth and Mars (Anders and Owen, 1977), a depletion relative to chondritic patterns of up to 90% (Ozima and Podosek, 1999) and known as the 'missing Xe' problem. Atmospheric Xe is besides strongly depleted in light isotopes (Krummenacher et al., 1962), a depletion that could be even larger if the primordial Earth composition – from which the present-day atmosphere is derived by mass-fractionation processes – is the U–Xe component instead of a solar wind or Q-planetary compo-

nent (Marty et al., 2017). First high precision measurements of non-radiogenic Xe isotopes (Caffee et al., 1999) and their correlation with ¹²⁹Xe/¹³⁰Xe ratio showed that the atmosphere was degassed very early after minor iodine decay in the mantle, and then underwent mass fractionation. Three types of scenarios have been proposed to solve the missing Xe problem. (1) Xe may have escaped from the atmosphere, possibly after ionization (Avice and Marty, 2014), (2) Xe may be hidden at depth in the core (Lee and Steinle-Neumann, 2006; Zhu et al., 2014), (3) Xe may have simply not been accreted on the planet (Dauphas, 2003). The fact that Earth and Mars atmospheres are similarly depleted in light Xe isotopes (Swindle et al., 1986) nevertheless points out to a phenomenon that does not depend on the mass of the planet such as hydrodynamic escape or trapping in the deep reservoirs unique to the Earth, i.e. lower mantle and core. Besides, core formation (Kleine et al., 2002) was likely completed by the time Xe was lost around 110 My (Ozima and Podosek, 1999). Discovery of Archean rocks with Xe isotopic composition falling between primitive chondrites and present Earth atmosphere (Pujol et al., 2011) indicates a storage and fractionation of Xe throughout the Archean that could even continue nowadays through subduction process. Geochemical tracing of Xe recycling in the mantle (Trieloff and Kunz, 2005; Holland and Ballentine, 2006) points indeed to an obvious link between Xe and water intake and release in subduction zones, resulting in the storage of volatiles in the subcontinental lithosphere (Broadley et al., 2016). While natural Xe concentrations are \sim 0.1 ppt in granites (Allègre et al., 1986) and \sim 0.05 ppt in basalts (Ozima et al., 2002), they reach 0.2 ppb in deep sea siliceous fossils (Matsuda and Matsubara, 1989) that may later enter subduction zones.

To understand Xe atypical behaviour, the mainstream approach has been to study the relative stability of noble gases in melts relative to the major silicate minerals by measuring partition coefficients. The results (Heber et al., 2007) span 4 orders of magnitude for Ar, and 7 for Xe, mostly due to different interpretations of where the noble gas was incorporated, i.e. exclusively in bubblefree crystal zones or including sub-surface and small bubbles as reflecting gas release upon quenching from high P-T conditions. This highlights the extreme difficulty to probe the behaviour of volatile elements by analyzing samples recovered at ambient conditions, either natural or synthetic. Besides, despite its generally inert nature. Xe has been shown to react with crystalline planetary oxides (Sanloup et al., 2005, 2011, 2013; Seoung et al., 2014; Crépisson et al., 2018) under the high pressure (P)-temperature (T) conditions relevant of planetary interiors. Xe oxides have been synthesized at ambient P and cryogenic T: XeO₃, XeO₄, Xe peroxides (XeO_6^{4-} ion), XeO_2 (Brock and Schrobilgen, 2011), and lately Xe perovskites (Britvin et al., 2016). On the high P side, Xe oxides have been predicted to be stable above 75 GPa (Zhu et al., 2013; Hermann and Schwerdtfeger, 2014), and observed above 77 GPa (Dewaele et al., 2016). Magmas being the most efficient transfer agents, the next natural step was to determine Xe reactivity in silicate melts at depth.

The first and major objective here is to solve the retention mechanisms of Xe in deep silicate melts by means of *in situ* high P-T X-ray diffraction experiments. While X-ray absorption spectroscopy (XAS) is a classical method to probe the local environment of elements in crystals, and used to evidence the Xe–O bond in high *P* Xe oxides (Dewaele et al., 2016), it is difficult to use it for materials without appropriate standards and with unknown structure, especially disordered materials such as magmas. Instead, X-ray diffraction data are not model dependent. They are however not chemically-selective and all ion–ion contributions must be solved. This implies to work with simplified chemical compositions, such as haplogranitic magmas taken here as analogues of continental crust melts, with only 5 elements (Si, Al, Na, K, and O),

Table 1

Chemical composition in wt% obtained from EMPA except * from PIXE and ** from ERDA. Analyses are based on average of 10 data points, standard deviations are shown in brackets.

Oxide Plain glass Xe-dopped glasses Recovered sample P5 SiO2 73.8 (0.6) 69.3 (0.7) 71.3 (0.7) 67.9 (0.9) Al2O3 12.2 (0.3) 11.1 (0.3) 11.7 (0.2) 10.8 (0.3) Naco 4.7 (0.1) 3.6 (0.1) 4.6 (0.1) 3.2 (0.5)	
SiO ₂ 73.8 (0.6) 69.3 (0.7) 71.3 (0.7) 67.9 (0.9) Al_2O_3 12.2 (0.3) 11.1 (0.3) 11.7 (0.2) 10.8 (0.3) N_{2O} 4.7 (0.1) 3.6 (0.1) 4.6 (0.1) 3.2 (0.5)	
$\begin{array}{ccccc} Al_2O_3 & 12.2 (0.3) & 11.1 (0.3) & 11.7 (0.2) & 10.8 (0.3) \\ N_{22}O & 4.7 (0.1) & 3.6 (0.1) & 4.6 (0.1) & 3.2 (0.5) \end{array}$	
$N_{22}O = 47(01) = 36(01) = 46(01) = 32(05)$	
1420 $4.7(0.1)$ $3.0(0.1)$ $4.0(0.1)$ $3.2(0.3)$	
K_2O 4.2 (0.2) 3.7 (0.2) 4.1 (0.2) 3.9 (0.1)	
MgO – 1.5 (0.1) – 1.6 (0.1)	
Xe – 3.1 (0.2) 4.6 (0.1) 1.5 (0.2)	
Xe* – 3.1 (0.4) 4.0 (0.8) 2.9 (0.4)	
H_2O^{**} 5.9 (1.7) 7.2 (2.0) 5.1 (1.4) 7.2 (2.0)	
Total 100.8 99.5 100.8 97.5	

and Si making 70 at% of the cations. X-ray diffraction is a well established technique to probe the structural environment of major cations in silicate melts *in situ*, and the recent study of Lu environment in compressed melts has demonstrated its potential to probe the local environment of minor elements (de Grouchy et al., 2017). The second objective is to measure the solubility of Xe in silicarich melts at high P, current data under high P conditions being limited to the tholeitic composition (Schmidt and Keppler, 2002), and discuss the structural data in the light of Xe solubility measurements and theoretical estimates.

2. Experimental methods

2.1. Glass synthesis

The haplogranite (HPG) glass composition was chosen as a continental crust melt analogue. The starting HPG glass was prepared by mixing reagent grade oxides (Al₂O₃, SiO₂) and carbonates (Na₂CO₃, K₂CO₃) powders. Powder was ground and decarbonated by slow ramp heating from room temperature to 1473 K within 8 h in a platinum crucible, further fused at 1873 K in an atmospheric furnace for one hour, and quenched in water. The obtained glass was crushed into a powder, and remelted twice at 1873 K to ensure homogeneity. Hydration was done by high P-T synthesis at 2 GPa and 1673 K using a piston-cylinder apparatus with a half-inch talc pyrex assembly, a graphite heater, and dried MgO powder packed around the Pt capsule. Two hydration levels were targeted, 5 wt% and 7 wt%. For that purpose, the glass was encapsulated along with the approximate amount of distilled water in a platinum capsule weld shut at both ends. Xenon doping was done subsequently by gas-loading (Boettcher et al., 1989) another platinum capsule partially filled with the recovered hydrous HPG glasses, and brought to 3.5 GPa and 1873 K for one hour, followed by rapid T quenching and decompression at room T. The major part of the capsule material was used for X-ray diffraction experiments, and a few glass pieces were spared for chemical analysis. The HPG-7 wt% H₂O composition was slightly contaminated upon retrieving the hydrated glass from the first platinum capsule by the MgO P-medium packed around the capsule (1.5 wt% MgO, cf. Table 1).

2.2. Recovered sample analysis

Starting glasses and one sample (P5) were analyzed for major elements using a CAMECA SX-FIVE electron microprobe analyzer (EMPA) at Camparis (UPMC, Paris), using a defocused beam of 15 µm diameter and the following operating conditions: 1) 15 keV accelerating voltage, 6 nA beam current for Na, K, Al, Si, and 2) 15 keV and 100 nA for Xe. Calibration for Xe was established by measuring the counts for the neighbouring elements, I (CuI) and Cs (CsCl) following the procedure developed by Montana et al. (1993). Hydrogen (i.e. water) and Xe contents were analyzed with

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