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# Iron oxidation state of impact glasses from the Zhamanshin crater studied by X-ray absorption spectroscopy

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## ABSTRACT

X-ray absorption spectroscopy (XAS) has been used to analyze the change in iron oxidation state and coordination numbers of impact glasses from the Zhamanshin crater. Series of samples, specifically aerodynamically shaped small glassy bodies – irghizites, and silica-rich and silica-poor glasses – zhamanshinites, have been investigated. The Fe<sup>2.2+</sup> oxidation state was determined for the irghizites and the highest value Fe<sup>2.8+</sup> for the silica-poor zhamanshinite. These values agree with the trend of oxidation processes upon high speed interaction of meteorite parent body and target rocks. The obtained data can be used for future establishment of the formation parameters (P, T,  $f_{O_2}$  conditions) of these impact glasses as well as relationships between the rocks.

**Keywords:** X-ray absorption spectroscopy; XANES spectroscopy; EXAFS spectroscopy; pre-edge feature; iron oxidation state and coordination numbers; impact glasses.

## 1. Introduction

Tektites and impactites are natural silicate glass bodies that are scattered over extended strewn fields of many thousands squared kilometers. There are four main strewn fields of tektites and impactites: Central European, Australasian, North American, and Ivory Coast. They differ from other naturally produced glasses – e.g. volcanic and fulgurites - by their shapes, petrography and chemistry. It has been accepted that tektites formed from molten high speed ejecta during early stages of impact crater formation (Artemieva, 2008). On the other hand, it has been suggested that large aerial bursts of extraterrestrial bodies like comets and asteroids can explain the formation of the layered tektite strewn field in Indochina in the absence of an associated impact crater (Wasson, 2003). Impact cratering processes invoke the formation of shock-reworked rocks (impactites) too. One specific feature of the crater-forming processes is formation of impact glasses due to melting of target rocks after release of shock pressure. As an example, glasses from the Zhamanshin impact crater (Kazakhstan) are present as aerodynamically shaped small glassy bodies - irghizites and silica-rich and silica-poor glasses – zhamanshinites. It has been noted that Si-rich zhamanshinites are structurally and chemically very similar to layered tektites in Indochina.

Different types of tektites and impact glasses can be formed by different conditions and from different compositions of target rocks. It is known that glass structure (i.e. cation coordination number) is influenced by conditions- pressure (P), temperature (T) - existing during the glass formation process (Mysen and Neuville, 1995; Yarger et al., 1995; Paris et al., 1994; Stebbins and McMillan, 1989). Particularly, pressure tends to increase the mean coordination number of the cations present. The relationship between the local atomic structure and aluminum and titanium in natural glasses and their origin has been widely investigated (Giuli, 2000; Farges, 1996a, 1996b, 1996c). On the other hand, iron is also a common chemical element in the composition of natural glasses which can be potentially useful as a probe to obtain information on formation conditions (P – T –  $fO_2$ ) of tektites and impactites. It was discussed, for example, by Moretti and Ottonello (2003) that the Fe redox state of a melt depends on P, T,  $fO_2$  and melt composition. Influence of P – T –  $fO_2$  parameters on the  $Fe^{3+}/Fe^{2+}$  value of impact melts is discussed by Lukanin and Kadik (2007); they also proposed the decompression mechanism of ferric iron reduction in tektite melts during their formation in the impact process. The goal of the present study is to determine Fe oxidation states and coordination numbers of the impact glasses from the Zhamanshin crater (irghizites, silica-rich and silica-poor zhamanshinites) for accumulating information about Fe role in tektites and impactites and future establishment of the formation parameters (P, T,  $fO_2$  conditions), as well as relationships between the rocks.

X-ray absorption spectroscopy (XAS) (Bunker, 2011; van Bokhoven and Lamberti, 2016) is a powerful modern tool which allows obtaining information on local atomic and electronic structure around the absorbing type of atoms in materials in condensed state even without long-range order in an atomic distribution. X-ray absorption near-edge structure (XANES) spectroscopy gives information on local electronic structure (in particular, oxidation state of atoms) and local 3D atomic structure (interatomic distances and bond angles), while Extended X-ray absorption near-edge structure (EXAFS) spectroscopy allows determining coordination numbers and interatomic distances (Bunker, 2011.; van Bokhoven and Lamberti, 2016). Examples of application of XANES and EXAFS spectroscopies for geological materials studies can be found, for example, in Berry et al., 2017; Berry et al., 2007; Kravtsova et al. 2016. XANES spectra analysis was also successfully used to estimate Fe oxidation state in minerals (Mino et al. 2014; Borfecchia et al, 2012). Fe oxidation states and coordination numbers of impact glasses were analyzed based on XANES and EXAFS spectroscopies much less often (Giuli et al., 2014; Giuli et al., 2002; Wang et al., 2013). And in the literature there are no systematic studies of Fe role in impactites covering all their variety. So, in this study we examined XANES and EXAFS spectra to obtain information about Fe state in different impact glasses from the Zhamanshin crater.

## 2. Experimental

In the study we used the impact glasses from the Zhamanshin crater, specifically two aerodynamically shaped small glassy bodies – irghizites, one silica-rich and three silica-poor glasses – zhamanshinites.

Zhamanshin is a meteorite crater in Kazakhstan, it's coordinates is 48°24'N 60°58'E. It is 14 kilometres in diameter and the age is estimated to be  $900,000 \pm 100,000$  years. All samples under investigation belong to the Meteorite collection of Russian Academy of Sciences.

The samples were sectioned and their fractions were ground in agate mortar under ethanol till powder with grain size  $< \sim 3 \mu\text{m}$  for XAS measurements. Also polished sections from samples were inspected under the optical and scanning electron microscope (Jeol JSM 6610-LV at the NHM, Vienna) for compositional homogeneity and lack of mineral inclusions. Chemical compositions were obtained by means of a Jeol JXA 8530-F electron microprobe at the NHM, Vienna and with the electron beam defocused to a diameter of  $20 \mu\text{m}$ .

Fourier-transform infrared (FTIR) spectra of the investigated impact glasses were measured on a Vertex 70 (Bruker) Fourier spectrometer at room temperature in the air. For the measurements an attenuated total reflectance (ATR) accessory with a diamond crystal was utilized. The obtained spectra are shown in the Fig. S1 of SI.

The Fe *K*-edge XANES and EXAFS spectra of the impact glasses as well as reference samples (FeO,  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>,  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>, Fe<sub>3</sub>O<sub>4</sub> oxides) have been recorded at the Structural Materials Science beamline (Chernyshov et al., 2009) using the equipment of Kurchatov Synchrotron Radiation Source (Moscow, Russia). The storage ring with an electron beam energy of 2.5 GeV and a current of 80–100 mA was used as the source of radiation. For the monochromatization of the X-ray beam Si(111) channel-cut monochromator was used, which provided an energy resolution  $\Delta E/E \sim 2 \cdot 10^{-4}$ . Dumping of higher energy harmonics was achieved by distortion of the monochromator geometry. Energy calibration was performed by measuring XAS spectra of Fe foil. All experimental data were collected in transmission mode. For such measurements we used tablets pressed with the addition of boron nitride which is transparent in the X-ray range. Intensities of incident and transmitted X-ray beam were measured by ionization chambers filled with N<sub>2</sub>. In our measurements of XAS spectra three ionization chambers were used providing simultaneous measurements of XAS spectra for sample and reference. In such way the energy calibration of the monochromator could be checked and corrected. Energy step in the XANES region (from 40 eV before the edge to 80 eV above the edge) was 0.6 eV. In the EXAFS region (from 80 eV to 800 eV above the edge) the constant step equal to 0.05 Å<sup>-1</sup> in photoelectron wave number was employed. At every energy point in the XANES region signal was integrated for 1 second, in the EXAFS region integration time was set to 1 second at the beginning of the region and increased to 4 seconds at the end of the spectra. For all the samples at least 3 experimental spectra were collected for further averaging. All spectra were processed (a background was subtracted, and the normalization was performed) using the Demeter Athena software (Ravel and Newville, 2005).

Fitting of the EXAFS spectra was done using Artemis program code (Ravel and Newville, 2005).

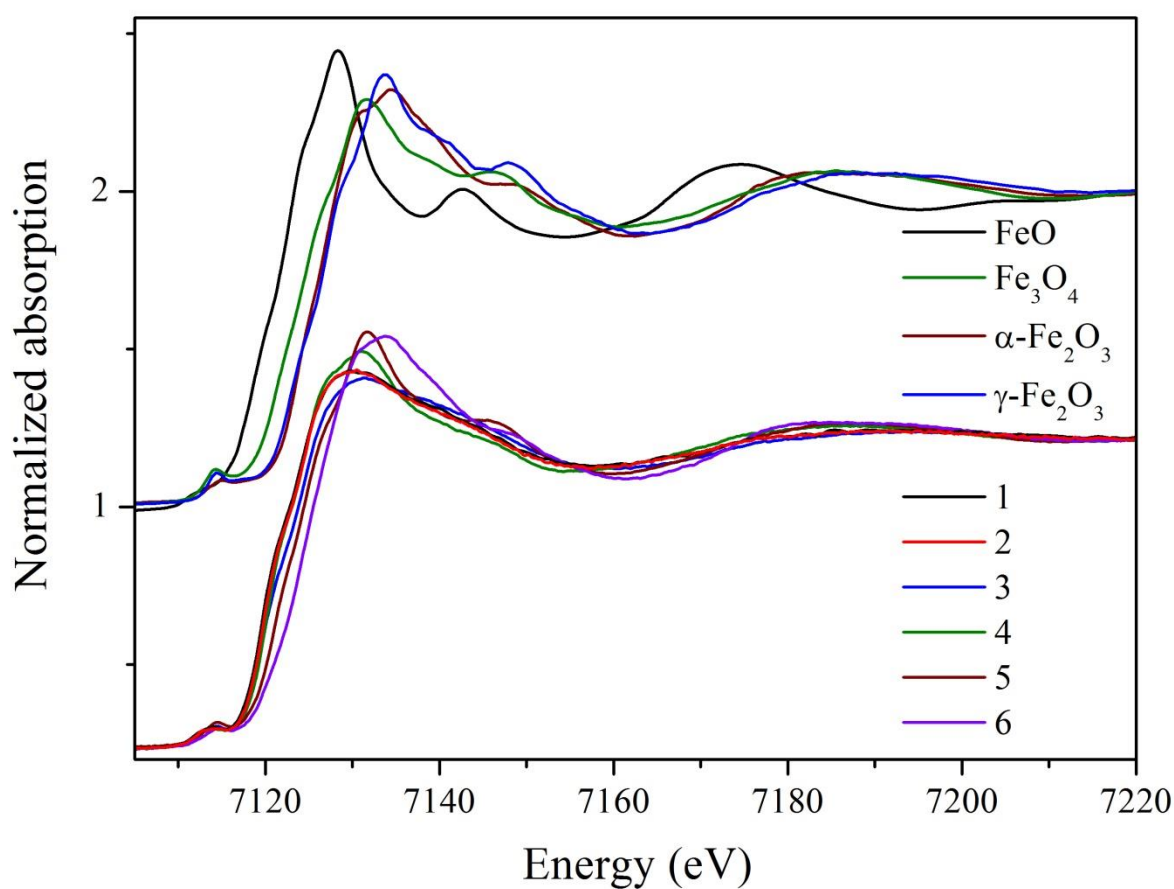
### 3. Results and discussion

At the first stage of investigation, the samples of impact glasses were studied using the optical and scanning electron microscopes for compositional homogeneity and lack of mineral inclusions. Two irghizite samples consist of rather homogeneous dark brown glass and have numerous bubbles. One silica-rich zhamanshinite consists of homogeneous yellow glass. Three silica-poor zhamanshinite samples are present as fluidal dark brown-yellow (samples 4, 5) and red-brown (sample 6) matters consisted of plagioclase tiny laths embedded in glassy matrices, the last contains skeletal aggregates of magnetite. No relict mineral inclusions were observed in polished sections. Averaged compositions of the irghizites and zhamanshinites are present in Table 1. The presented data are averages of at least ten individual electron microprobe analyses.

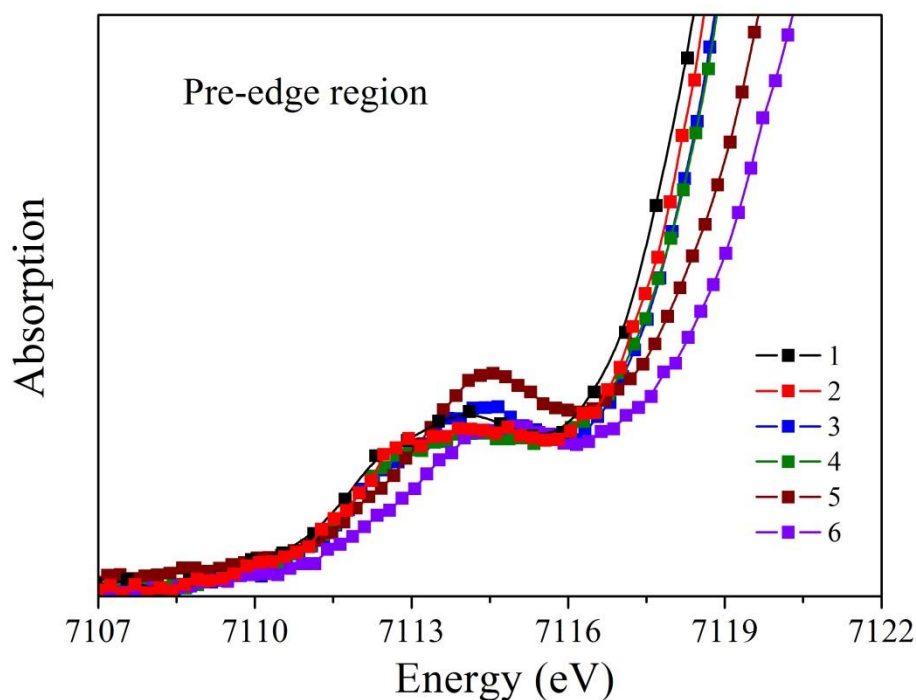
**Table 1** Major element chemical compositions (oxide wt%) of the impact glasses studied. All Fe given as FeO; standard deviations in the last decimal place are shown in parentheses.

Sample number	Sample description	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	FeO	MgO	TiO <sub>2</sub>	Mn O	Na <sub>2</sub> O	K <sub>2</sub> O	Total
1	Irghizite	74.30(36)	9.67(5)	2.44(5)	5.50(12)	2.94(4)	0.84(2)	0.07(3)	1.09(3)	1.95(5)	98.80
2	Irghizite	74.72(30)	9.63(12)	2.41(4)	5.34(8)	2.86(11)	0.86(2)	0.10(1)	1.08(3)	1.98(4)	98.99
3	SiO <sub>2</sub> -rich zhamanshinite	61.30(59)	16.82(23)	5.71(13)	5.24(9)	2.77(5)	0.68(2)	0.07(3)	5.21(15)	1.30(2)	99.09
4	SiO <sub>2</sub> -poor zhamanshinite	56.47(48)	22.41(39)	1.87(13)	7.16(81)	2.58(10)	0.81(3)	0.10(4)	5.38(37)	2.03(5)	98.81
5	SiO <sub>2</sub> -poor zhamanshinite	55.69(32)	20.70(44)	5.66(16)	8.22(54)	2.64(11)	1.19(16)	0.17(3)	4.09(8)	1.28(10)	99.64
6	SiO <sub>2</sub> -poor zhamanshinite	55.99(44)	22.73(49)	4.13(27)	8.54(99)	2.07(18)	1.15(5)	0.20(6)	3.18(7)	1.62(7)	99.62

Then, iron oxidation state in the samples of the impact glasses was investigated. Iron in tektites occurs predominantly as  $\text{Fe}^{2+}$ , while Fe in impact glasses can span a much wider range of Fe oxidation states, from purely divalent to purely trivalent, comprising mixtures of various coordination numbers (Giuli et al., 2002; Lukanin and Kadik, 2007). With this purpose the Fe *K*-edge XANES spectra were measured for the studied impact glasses and for the reference samples. The Fe *K*-XANES spectra of the samples under investigation are shown in the Fig. 1. Fig. 2 demonstrates the enlarged pre-edge region of the Fe *K*-XANES spectra presented in Fig. 1.



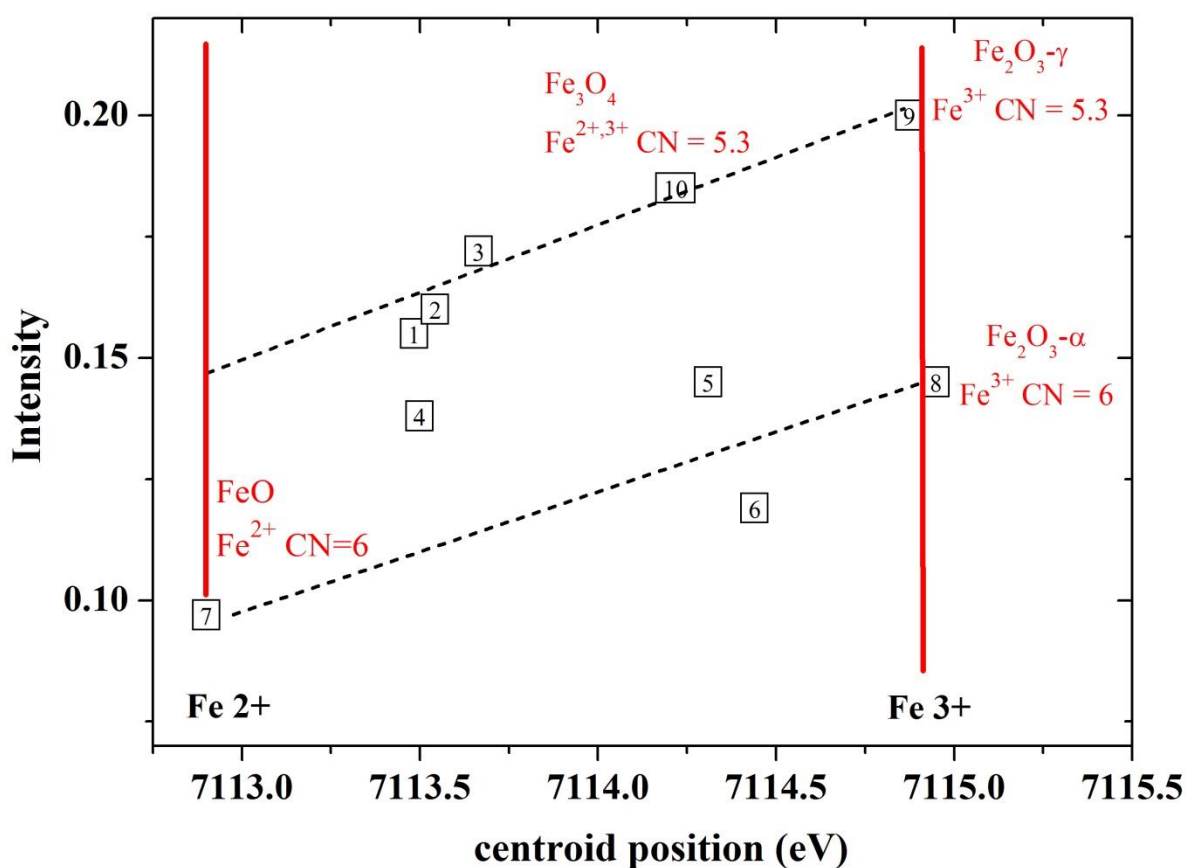
**Fig. 1.** (*Lower panel*) Fe *K*-XANES spectra of the impact glasses (samples marked as 1–6). (*Upper panel*) Fe *K*-XANES spectra of the reference samples – iron oxides  $\text{Fe}^{2+}\text{O}$ ,  $\text{Fe}^{2+,3+}_3\text{O}_4$ ,  $\alpha\text{-Fe}^{3+}_2\text{O}_3$ .



**Fig. 2.** The pre-edge region of the Fe *K*-XANES spectra of the impact glasses (samples marked as 1–6).

The Fe oxidation states of the impact glasses were determined by several methods. First, the so-called chemical shift was estimated where the energy position of the absorption edge of XANES spectra was analyzed. For such analysis we compare the positions of the absorption edge of the Fe *K*-XANES spectra of the impact glasses with those of the reference samples with well-known Fe oxidation state –  $\text{Fe}^{2+}\text{O}$ ,  $\alpha\text{-Fe}^{3+}_2\text{O}_3$ ,  $\gamma\text{-Fe}^{3+}_2\text{O}_3$ ,  $\text{Fe}^{2+,3+}_3\text{O}_4$ . The position of the absorption edge was determined at the points where the normalized absorption coefficient equals 0.4 and 0.6 (these values are empirical; we chose two values for averaging purposes). Similar approach for analysis of energy positions of absorption edges of Fe *K*-XANES spectra to derive information on the Fe oxidation state was recently applied to some tektites and natural glasses by Wang et al. (2013). Second, the Fe oxidation states were determined by analyzing the pre-edge region of the Fe *K*-XANES spectra by the method described by Wilke et al. (2001), Guili et al (2002). The dependence of the intensity of the pre-edge feature of the Fe *K*-XANES of the investigated samples on the pre-edge centroid energy is shown in Fig. 3, while pre-edge fitting results are shown in figures S2-S7 of SI. The averaged values of the Fe oxidation state obtained on the basis of several methods described above can be seen in Table 2.





**Fig. 3.** Correlation of the intensity of the pre-edge feature of the Fe *K*-XANES spectra of the impact glasses and reference samples with the pre-edge centroid energy.

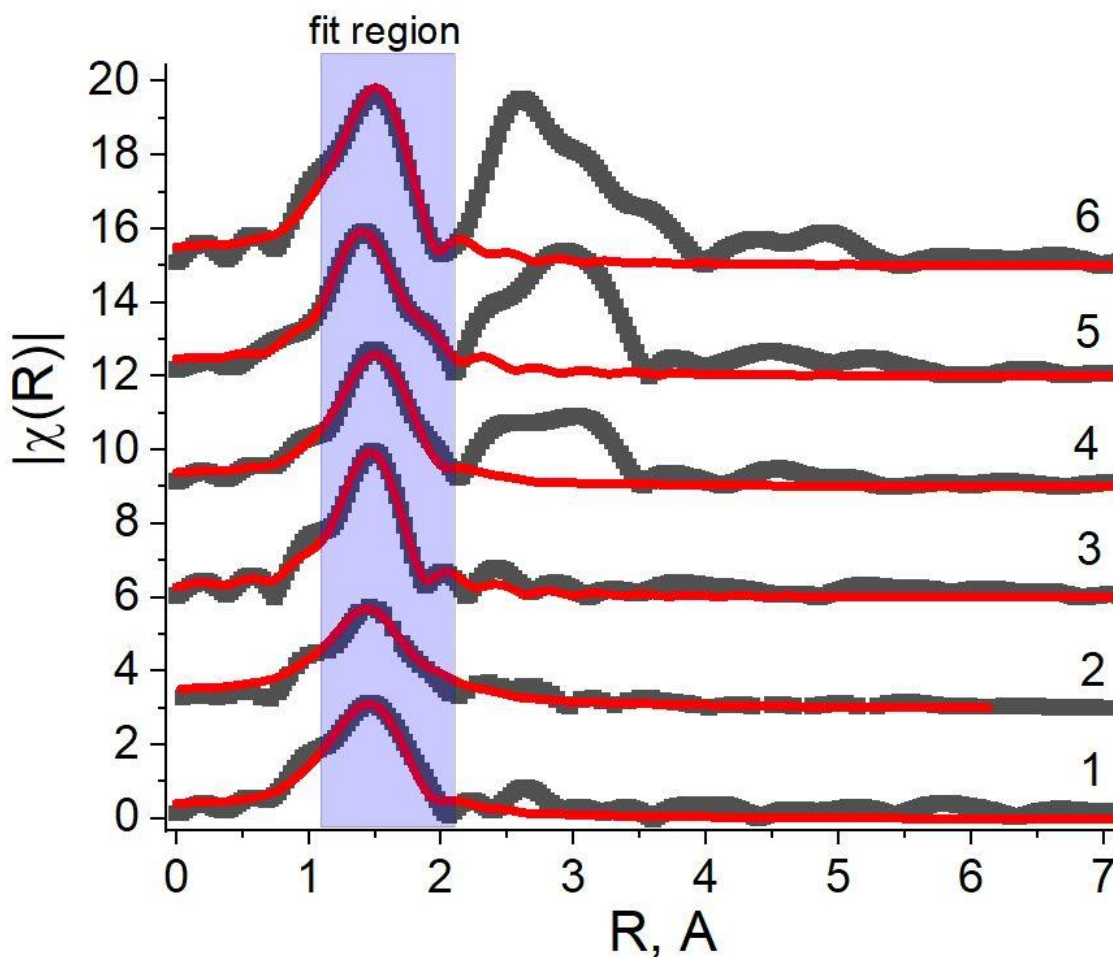
**Table 2** List of samples and values of mean oxidation state of iron in the impact glasses determined by analyzing the pre-edge region (Wilke et al., 2001; Giuli et al., 2002) and the energy position of the absorption edge of the Fe *K*-XANES spectra.

Sample number	Sample description	Fe oxidation state: pre-edge analysis	Fe oxidation state: absorption edge position on 0.4 <i>I</i> analysis	Fe oxidation state: absorption edge position on 0.6 <i>I</i> analysis	Averaged Fe oxidation state	Standard deviation
1	Irghizite	+2.3	+2.2	+2.1	+2.20	± 0.06
2	Irghizite	+2.3	+2.2	+2.2	+2.23	± 0.05
3	Silica-rich zhamanshinite	+2.4	+2.3	+2.3	+2.33	± 0.04
4	Silica-poor zhamanshinite	+2.3	+2.3	+2.2	+2.27	± 0.04

5	Silica-poor zhamanshinite	+2.7	+2.5	+2.5	+2.57	± 0.06
6	Silica-poor zhamanshinite	+2.8	+2.8	+2.8	+2.80	0.00
7	FeO (reference sample)	+2.00	+2.00	+2.00	+2.00	0.00
8	$\alpha$ -Fe O <sub>2 3</sub> (reference sample)	+3.00	+3.00	+3.00	+3.00	0.00
9	$\gamma$ -Fe O <sub>2 3</sub> (reference sample)	+3.00	+3.00	+3.00	+3.00	0.00
10	Fe O <sub>3 4</sub> (reference sample)	+2.67	+2.67	+2.67	+2.67	0.00

The Fe oxidation state in the irghizite impact glasses is about +2.2, while the one in the silica-rich zhamanshinite is about +2.3. The highest values of the Fe oxidation state were found in the impact glasses of silica-poor zhamanshinites (ca. +2.3, +2.6, +2.8).

As the intensity of a pre-edge feature of Fe *K*-edge XANES spectra of geological materials depends on the iron coordination (for example, Giuli et al., 2005), the comparison of the positions of investigated impact glasses with those of reference samples in Fig. 3 allow not only determining the Fe oxidation state, but also estimation of the coordination numbers (CNs) of iron in the impact glasses. Such kind of estimation of Fe CNs for tektites and impact glasses was recently used by Giuli et al. (2002, 2005). According to the Fig. 3, CNs of samples marked as 1, 2, 3 are close to 5, CNs of samples 4, 5 are close to 5.5, and CN of sample 6 is close to 6. These results are further supported with EXAFS analysis performed for the same samples. EXAFS fit was performed using two different distances along with their multiplicity as described in Giuli et.al. (2002). The comparison between the module of the Fourier transforms of the experimental and theoretical EXAFS signals of the impact glasses studied is shown in Fig. 4. Mean coordination number (<CN>) and mean interatomic distances in the first coordination sphere of iron (<Fe-O>) in the impact glasses are presented in Table 3.



**Fig. 4.** Comparison between the module of the Fourier transforms of the experimental, theoretical EXAFS signals of the impact glasses studied. The curves were shifted vertically for clarity.

**Table 3** Essential parameters of the EXAFS refinement.

Sample number	$\langle \text{CN} \rangle$	$\langle \text{Fe-O} \rangle$ , Å	$\langle \sigma^2 \rangle$ , Å <sup>2</sup>	$S_0^2$	R
1	4.8	1.94	0.010	0.8	0.0060
2	4.5	1.96	0.009	0.8	0.0063
3	4.5	1.94	0.009	0.8	0.0033
4	4.8	2.0	0.008	0.8	0.0013
5	5.6	1.99	0.006	0.8	0.0018
6	5.8	1.96	0.007	0.8	0.0059

$\langle \sigma^2 \rangle$  is the EXAFS Debye-Waller factor,  $S_0^2$  is the EXAFS amplitude and R is the disagreement index.

It has been discussed, for example, by Lukanin and Kadik (2007) that the  $\text{Fe}^{3+}/\text{Fe}^{2+}$  value of impact melts is influenced by  $P - T - f_{\text{O}_2}$  parameters. So, we hope that the obtained in the present study values of Fe oxidation states and CNs in the impact glasses from the Zhamanshin crater are useful from the point of view of accumulating information about Fe role in the impact glasses and further establishment of their formation parameters ( $P, T, f_{\text{O}_2}$  conditions).

#### 4. Conclusions

The Fe  $K$ -edge XANES and EXAFS spectra of six impact glasses from the Zhamanshin crater have been collected to estimate the Fe oxidation states and CNs. The lowest value of the Fe oxidation state ( $\sim +2.2$ ) was found for the irghizites and the highest value ( $\sim +2.8$ ) for the silica-poor zhamanshinite. The oxidation states of the samples describe the aqueous alteration and thermal and shock metamorphism of the parent bodies of the impact. The data obtained can be useful for future establishment of the formation parameters ( $P, T, f_{\text{O}_2}$  conditions) of the impact glasses and relationships between the rocks.

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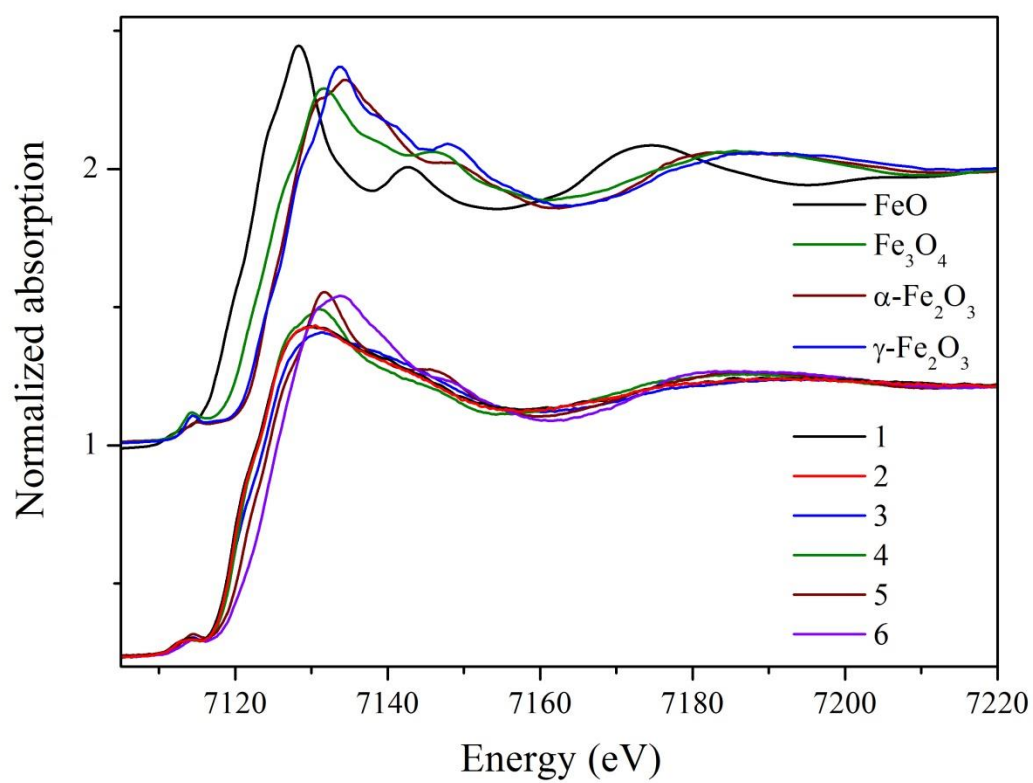
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**Figure 1.** (*Lower panel*) Fe *K*-XANES spectra of the impact glasses (samples marked as 1–6). (*Upper panel*) Fe *K*-XANES spectra of the reference samples – iron oxides Fe<sup>2+</sup>O, Fe<sup>2+,3+</sup><sub>3</sub>O<sub>4</sub>, α-Fe<sup>3+</sup><sub>2</sub>O<sub>3</sub>.

**Figure 2.** The pre-edge region of the Fe *K*-XANES spectra of the impact glasses (samples marked as 1–6).

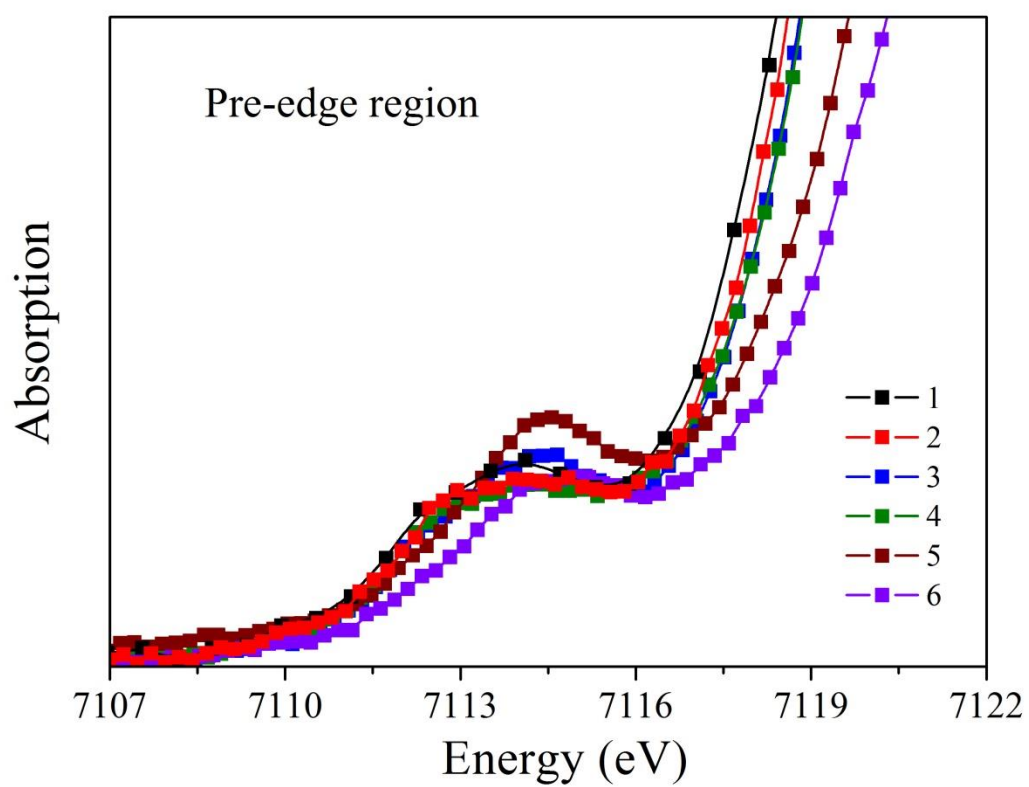
**Figure 3.** Correlation of the intensity of the pre-edge feature of the Fe *K*-XANES spectra of the impact glasses and reference samples with the pre-edge centroid energy.

**Figure 4.** Comparison between the module of the Fourier transforms of the experimental, theoretical EXAFS signals of the impact glasses studied. The curves were shifted vertically for clarity.

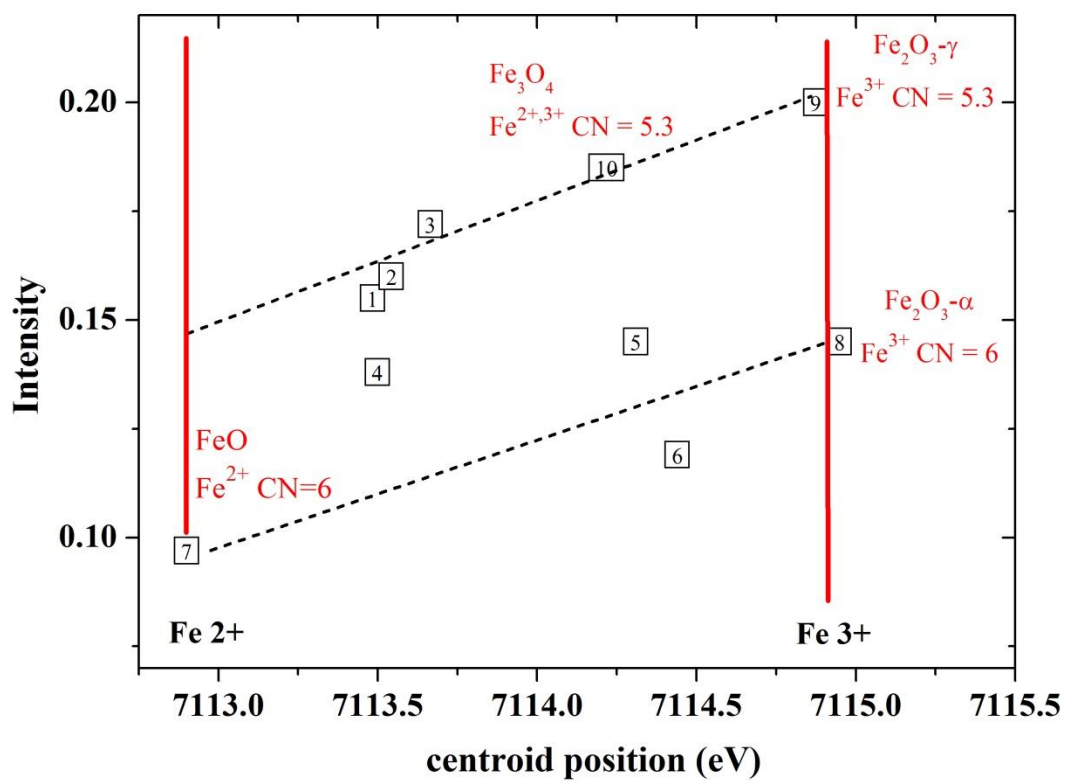


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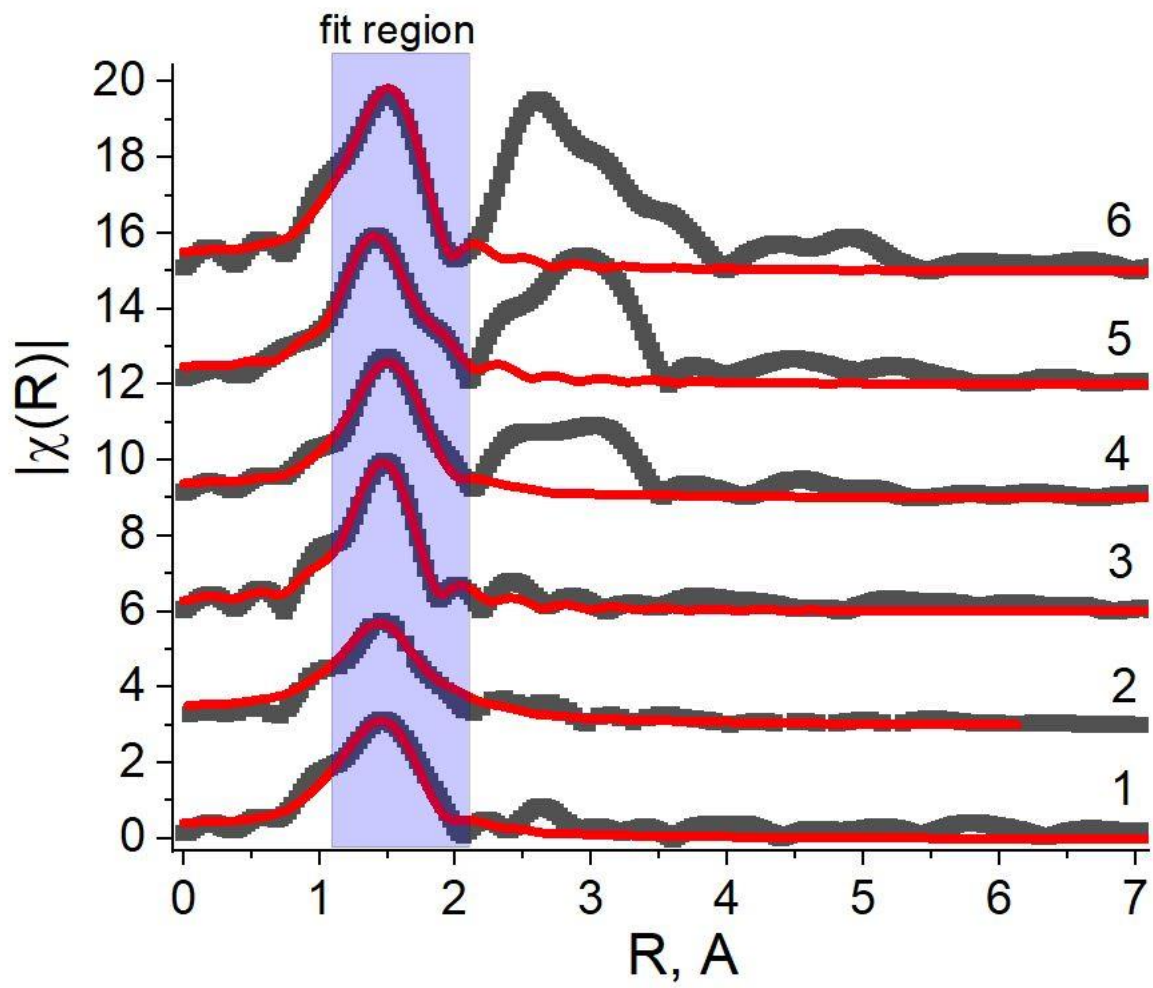




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**Highlights**

- Irghizite samples have the lowest oxidation state of 2.2+.
- Silica-poor zhamanshinite sample has the highest oxidation state of 2.8+.
- Average coordination number for irghizites is ~4.8.
- Average coordination number for silica-rich zhamanshinite is ~ 4.8.
- Average coordination number for silica-poor zhamanshinites is ~ 5.5.

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