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Study of the phenomenon of magnetism in iron-containing silicates glasses

Abstract: Studying the structure and properties of iron-containing glass-crystalline materials is an urgent task, as it makes it possible to develop glass compositions with high magnetic properties. The study subject is the structure and magnetic properties of iron-containing silicate glasses of the SiO_2-FeO-CaO system in the presence of MgO and Al_2O_3. The study object is the structure and magnetic properties of iron-containing silicate glasses. The study aims to obtain silicate glasses with a high content of iron oxides and study their properties. DTA and X-ray phase analysis methods were used to study the materials. The glass's relative quality factor and magnetic permeability were determined using a Q-meter. The phase diagrams of systems containing oxides SiO_2, FeO, Fe_2O_3, CaO, MgO, Al_2O_3, B_2O_3, and K_2O and selected compositions corresponding to the minimum eutectic temperatures were analysed. The studies were based on the works of N. Wojcik, A. Paladino, A., S. Zhang, M., Yu. Ebisawa, M. Plemyannikov and other scientists. The glass was synthesised at a temperature of 1300°C for 2 hours. The resulting glass was crystallised by slowly increasing the temperature to 550-800ºC for 3 hours. The results of differential thermal analysis confirmed the tendency of specific glass compositions to crystallise. According to the X-ray phase analysis results, the formation of magnetite, a ferromagnet, was confirmed. Studies of the relative quality factor and glasses' magnetic permeability confirmed the magnetic properties of the samples that were obtained. These results are consistent with theoretical studies of system state diagrams and the results of instrumental analysis methods.

Keywords: state diagram, glass, iron oxides, crystallisation, magnetism; magnetite.

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Вивчення явища магнетизму у залізовмісних силикатних стеклах

Анотація: Вивчення структури та властивостей залізовмісних склокристалічних матеріалів є актуальним завданням, так як дозволяє розробити склади стекол з високими магнітними властивостями. Предметом дослідження є структура і магнітні властивості залізовмісних силікатних стекол системи системи SiO_2-FeO-CaO в присутності MgO, Al_2O_3. Об'єктом дослідження є структура та магнітні властивості залізовмісних силікатних стекол. Метою роботи є отримання силікатних стекол з підвищеним вмістом оксидів заліза та вивчення їх властивостей. Для дослідження матеріалів використано методи ДТА, РФА та визначено відносну добротність та магнітну проникність стекол за допомогою Q-метра. Були проаналізовані діаграми стану систем, що містять оксиди SiO_2, FeO, Fe_2O_3, CaO, MgO, Al_2O_3, B_2O_3, K_2O та обрані склади, що відповідають мінімальнім евтектичним температурам. Дослідження опиралися на праці N. Wojcik, A. Paladino, A., S. Zhang, M., Yu. Ebisawa, M. Племяннікова та інших вчених. Синтез скла проводили при температурі 1300°C протягом 2 годин. Отримане скло кристалізазували шляхом повільного підвищення температур в інтервалі 550-800ºC протягом 2 годин. Результати диференціального термічного аналізу підтвердили схильність визначених складів стекол до кристалізації. За результатами рентгенофазового аналізу підтверджено утворення магнетиту, що є феримагнетиком. Дослідження відносної добротністі та магнітної проникністі стекол підтвердили наявність магнітних властивостей отриманих зразків. Ці результати узгоджуються з теоретичними дослідженнями діаграм стану систем та результатами інструментальних методів аналізу.

Ключові слова: діаграми стану, скло, оксиди феруму, кристалізація, магнетизм, магнетит.

Abbreviations:

DTA is differential thermal analysis;

EPR is electron paramagnetic resonance;

SEM is scanning electron microscopy;

XRD is X-ray fluorescence analysis;

$XRPA$ is X-ray phase analysis.

Introduction

Studying the structure and properties of iron-containing glass-crystalline materials is an urgent task, as it makes it possible to develop glass compositions with high magnetic properties. Also, this research will allow us to expand knowledge about the effect of FeO on the properties of silicate systems SiO_2-FeO-CaO, SiO_2-FeO-CaO-MgO, SiO_2-FeO-CaO-MgO-Al_2O_3.

The study focuses on the structure and magnetic properties of iron-containing silicate glasses in the SiO2-FeO-CaO system in the presence of MgO and Al_2O_3.

The object of research is the structure and magnetic properties of iron-containing silicate glasses of the SiO_2-FeO-CaO system.

The study aims to obtain silicate glasses with a high content of iron oxides and study their properties.

To achieve this goal, you must complete the following tasks:

- select glass compositions that will have the lowest melting points and increased crystallisation capacity, according to the diagrams of the state of the systems;
- investigate the crystallisation capacity of the obtained glasses and use differential thermal analysis;
- study the phase composition of the obtained samples using X-ray phase analysis;
- study the magnetic properties of glass-crystal materials, namely the magnetic permeability of the material.

The materials were studied using the DTA and XRD methods, and the glasses' relative Q-factor and magnetic permeability were determined using a Q-meter.

Overview of information sources

Glasses containing ferum oxides are constantly of interest to researchers. Since the presence of these oxides significantly affects the properties of the resulting glass and glass-crystalline materials. Features of the primary raw material for producing industrial silicate glasses is quartz sand. For this material, the content of ferum oxides is strictly regulated since even a tiny amount (fractions of a per cent) of iron oxides leads to undesirable staining of the glass, and with a high iron content (up to 10 per cent or more), the glass becomes opaque and black. As an exception, such compositions can produce brown or green glass containers or facing tiles or black glazes (*Plemiannikov & Zhdaniuk, 2023*). When the problem of staining transparent glass arises, it is necessary to consider that ferum can be in two valence states: Fe(II) and Fe(III). Thus, the Fe^{2+} ion has a strong absorption band in the near-infrared range, while Fe^{3+} absorbs mainly in the ultraviolet range. The presence of FeO turns the glass blue and Fe_2O_3 yellow. The combined presence of Fe(II) oxide and Fe (III) oxide causes gradations of glass shades that fall in the green region of the spectrum (*Vercamer, 2016*). The state of thermodynamic equilibrium of $FeO \leftrightarrow Fe_2O_3$ depends primarily on the redox potential of the cooking process, the cooking temperature, the concentration of oxides, and the chemical composition of the glass itself, which must be taken into account when developing charge compositions (*Plemiannikov & Zhdaniuk, 2023*).

When studying the properties of glasses with a high content of Froome oxides, it is necessary to take into account that these compounds can perform a twofold role – a grid-forming agent (Fe^{+3}) and a modifier (Fe^{+2}) (*Vercamer, 2016*; *Plemiannikov & Zhdanyuk, 2021*). Therefore, the shift in the equilibrium of $Fe_2O_3 \leftrightarrow FeO$ in silicate melts with a high ferum compound content significantly affects the glass structure's properties. The study of the equilibrium state between FeO and Fe_2O_3 in the melt, depending on its composition and temperature, showed that as the temperature increases, the equilibrium shifts towards forming Fe(II) oxide. At 1320-1410°C, Fe(II) oxide in the melt contains 50% more than at 1230-1320°C. with an increase in the content of silica and alumina in the melt, the reaction equilibrium shifts towards the formation of Fe(II) oxide, and with an increase in the content of MgO – towards Fe (III) oxide (*Фальковська, 1989*).

Depending on the valence state of iron, ferum oxides play a twofold role in glass: a grid-forming agent (Fe^{+3}) and a modifier (Fe^{+3}) (*Vercamer, 2016*; *Calas & Petiau, 1983*; *Plemiannikov & Zhdanyuk, 2021*). The Fe^{3+} ion is located in the glass in tetrahedral oxygen coordination and can replace silicium in the structure of anions and ensures the structural integrity of the vitreous body. In addition, an increase in the concentration of Fe_2O_3 leads to forming Fe–O–Si bonds, indicating the glass mesh's depolymerisation. The Fe^{2+} ion in glass has octahedral oxygen coordination and acts as a typical glass modifier (*Ebisawa ma ін., 1991*; *Tasheva et al., 2023*; *Alderman et al., 2017*; *Peys et al., 2018*).

The course of glass crystallisation is significantly affected by the concentration of ferum ions and their redox state. In some cases, crystallisation may occur spontaneously. Fe^{2+} causes glass crystallisation faster and more significantly than Fe^{3+} (*Alderman et al., 2017*; *Wisniewski et al., 2011*; *Zhdaniuk & Plemiannikov, 2024*). This is due to the role of the modifier Fe^{2+}, which can stimulate crystallisation, while Fe^{3+} is a grid-forming agent and is less mobile. Trivalent ferrum ions can form magnetite, crystallising from the silicate melt at temperatures below 1300°C. Magnetite increases glass heterogeneity and its viscosity (*Chevrel et al., 2013*).

The paper (Wisniewski et al., 2011) confirmed that during the crystallisation of iron-containing glasses, magnetite can give glass magnetic properties due to clusters or crystals with magnetic properties. In addition, the paper (*Wisniewski et al., 2011*) proved that hematite (Fe_2O_3) is the primary crystallised phase, and magnetite (Fe_3O_4) is the result of phase transformation after primary crystal growth. In addition, the presence of MgO in the iron-containing silicate melt contributes to the formation of magnesioferite ($MgFe_2O_4$), which is characterised by high magnetic properties in addition to magnetite. SEM combined with electron backscattering diffraction was the primary method for phase characterisation (EBSD).

Thus, the analysis of literature sources confirms the possibility of shifting the chemical equilibrium of $FeO \leftrightarrow Fe_2O_3$ and determines the possibility of directional synthesis to obtain glass-crystalline materials containing magnetic nanoparticles. Oxide glasses containing paramagnetic inclusions attract attention as materials from which it is possible to obtain magnetically ordered particles of microscopic size, which is one of the innovative directions of modern physics of magnetic phenomena.

Materials and methods

To determine the optimal compositions of iron-containing glass-crystalline materials, state diagrams of the two-component SiO_2–FeO and SiO_2–Fe_2O_3 systems were analysed. System state diagram analysis indicates the presence of low-melting eutectics at temperatures of 1173ºC and 1455ºC, respectively (*Figure 1*).

SiO_2-FeO is selected for cooking iron-containing glasses. To optimise the properties of glasses, state diagrams of systems were analysed, in which various combinations were added to the essential components: CaO, MgO, Al_2O_3. Then, the composition of the glass was determined at a minimum eutectic temperature. Mass percentages were recalculated for the glass compositions selected in this way. The recipe for various systems' glasses is in the appendix (*Table 1*).

The following chemical reagents were used for cooking glass: amorphous silica (SiO_2), Fe(II) oxide (FeO), calcium carbonate ($CaCO_3$), magnesium oxide (MgO), and aluminium oxide (Al_2O_3). Cooking took place in a silite electric oven at 1300°C for 2 hours in neutral conditions. Chamotte crucibles with a capacity of 250 ml were used for cooking. Before the study, the glass was heat-treated to crystallise it. The glass was slowly heated in the temperature range of 550-800°C for 2 hours.

The cooking capacity of glass and the aggressiveness of glass mass concerning chamotte were performed visually according to the method described in the article (*Plemiannikov & Zhdanyuk, 2021*).

Thermal studies of samples of glass-crystalline materials were performed on the derivatograph Q-1500 device (Hungary) of the Paulik-Paulik-Erdey system. The samples were heated to 1000ºC in the air atmosphere at a heating rate of 10ºC/min. The samples' weight was 1500 mg. Al2O3 was taken as the reference substance. The experiments used a platinum crucible. The device's sensitivity on the DTA scale is 250 MV.

The phase composition of vitreous materials was studied using the XRPA method on a DRON 3M diffractometer. The X-ray tube is Cukaα. U = 30 kW. The phases were identified using the ICDD file.

The paper also investigated the magnetic properties, namely the relative Q-factor and magnetic permeability of glasses using Q-meter E 9-4. The equation calculated the relative magnetic permeability of the material:

$$\Delta Q = \frac{Q_0 - Q}{Q_0} \cdot 100\%,$$

where

Q_0 – Q-factor of a circuit with a coil filled with air (μ≈1),

Q – Q-factor of a circuit with a coil filled with a substance (μ≠1).

The relative magnetic permeability of the core material was calculated by the formula:

$$\mu = \frac{C_0 - C}{C_0} \cdot 100\%,$$

where

C_0 – capacity of a circuit with a coil filled with air (μ≈1),

C – capacity of a circuit with a coil filled with a substance (μ≠1).

Results

All three compounds formed an opaque black glass during cooking. All windows were air-conditioned. It is worth noting that the lowest-melting glass was formed for Composition No. 1 after recrystallisation, and the colour of glass-crystalline materials was also black.

DTA was used to examine samples of glass-crystalline materials. Exothermic effects on the DTA curves of all samples indicate a high crystallisation capacity of the glasses and the probability of formation of glass-crystalline materials. Moreover, several peaks are present for some compositions, indicating a complex crystallisation polymineral nature (*Figure 2*).

Analysis of the XRD results of sample No. 1 confirmed the presence of a crystal phase – magnetite (Fe3O4). The resulting magnetite has spheres of magnetic properties. Also, a wide halo is visible on the diffraction pattern, indicating a large proportion of the glass phase (*Figure 3*).

For composition No. 2, in addition to small amounts of magnetite, the formation of magnesioferite ($MgFe_2O_4$) is also possible. The presence of MgO in the iron-containing silicate melt contributes to this (*Wisniewski et al., 2011*). For sample 3, only a small number of crystals with magnetic properties can form. We studied this system in our paper (*Plemiannikov & Zhdanyuk, 2021*). It is confirmed that Crystal phases are formed in this system $2(Mg,Fe)O{\cdot}SiO_2$, $FeO{\cdot}SiO_2$, FeO, Al_2O_3, $MgO{\cdot}FeO{\cdot}Al_2O_3$, for which magnetic properties are uncharacteristic. It is also possible to form small amounts of magnesioferite, justified in the work (*D'Ippolito et al., 2015*). To test the effect of Fe^{2+} and Fe^{3+} cations on the vibrational spectra, solid solutions of $MgAl_2O_4$–$MgFe_2O_4$ were examined using Raman spectroscopy. It was found that the Raman scattering modes are affected by the substitution of Mg^{2+} for Fe^{2+} in tetrahedral locations of the $MgAl_2O_4$–$FeAl_2O_4$ system, and the substitution of Fe^{3+} for Al^{3+} in octahedral locations of $MgAl_2O_4$–$MgFe_2O_4$, which confirms the possibility of magnesioferite formation.

An important part of the research was studying the magnetic properties of the samples. The results of experimental tests showed the relative magnetic permeability and changes in the Q-factor of the contour of the obtained glass-crystal materials (*Figure 4*).

According to research results, Composite glasses have ferrimagnetic properties. The most pronounced magnetic properties are characteristic of sample No. 1, explained by the formation of magnetite crystal phases ($FeO{\cdot}Fe_2O_3$). Magnetite is a typical representative of magnetic materials. Due to the ordering of the spin moments of ferum cations due to the exchange interaction between the electrons of the 3D shell of neighbouring cations, it has ferrimagnetic properties. According to the structure, the magnetic spin moments of Fe(III) ions located in the octa – and tetrahedral positions are mutually compensated, and the parallel ordering of the magnetic spin moments of Fe(II) ions located in the octahedral positions forms the magnetic moment of the mineral (*Dudchenko, 2011*). In samples 2 and 3, magnetite and magnesioferite are formed in small amounts. Magnesioferite, like magnetite, has strong properties. It is the extreme member of the isomorphic series: magnetite ($FeO{\cdot}Fe_2O_3$) – magnesioferite ($MgO{\cdot}Fe_2O_3$). Substitution of Fe^{2+} by a magnesium cation in the octahedral position interrupts the exchange of the Fe^{2+} 3D electron with neighbouring Fe3 + cations and has a lower magnetic susceptibility than magnetite. In this paper, we have obtained an iron-containing glass-crystal material with pronounced magnetic properties. Compositions with a high content of magnetite are promising. Research on the properties of iron-containing glasses will continue, as this is one of the innovative areas of modern physics of magnetic phenomena.

Discussion

Studying glass-crystalline materials based on iron-containing glasses is an urgent task. Since the content of ferum oxides in industrial silicate glasses is strictly regulated (fractions of a per cent), most studies were carried out on such materials. Introducing more ferum oxides into the silicate system significantly changes its properties. It makes it possible to obtain materials with unique properties, such as glass ceramics containing a magnetic phase in a biocompatible vitreous Matrix. Such materials can be used as thermal mattresses for treating cancer with magnetic induction hyperthermia. Magnetic properties are usually explained by the presence of a magnetic phase (magnetite (Fe_3O_4) or hematite (Fe_2O_3) in the glass mass. In such glass-crystal materials, the $Fe^{3+}(3d^5,6S^{5/2})$ ion EPR absorption at room temperature. For modern medical magnetic glass ceramics, silicate systems are used $CaO–SiO_2$; $CaO–SiO_2–P_2O_5$; $Na_2O–CaO–SiO_2–P_2O_5$; $MgO–CaO–SiO_2–P_2O_5–CaF_2$. The study of $CaO–Na_2O–B_2O_3$, $CaO–SiO_2–P_2O_5–Na_2O$ systems is promising.

The magnetic properties of glasses allow them to be used in electronics. The unique magnetic and mechanical properties of some amorphous alloys make the materials attractive for magnetic heads. Electromagnetic transducers and sensors made from conventional magnetic alloys have been used to measure compression or tension for several years, and the development seemed almost complete. The use of metal glasses in surface acoustic wave delay lines is particularly interesting, as they give a relatively large variation in the delay time with a slight change in the shear field.

Anisotropic microstructures accompanying spatial changes in magnetisation can also create effective magnetic anisotropies. In many cases, the magnetisation is significantly reduced due to exchange fluctuations, and materials usually have a low coercive force and no magnetic anisotropy.

The conducted research allowed us to expand our knowledge about the properties of iron-containing glasses that can be used to recycle iron ore processing waste. What wastes have a high raw material value, containing SiO_2, Na_2O, CaO, MgO, Al_2O_3, and up to 15% iron oxides? Studies of the properties of iron-containing silicate systems make it possible to conduct directional glass synthesis and dispose of waste using glass technology. For example, the production of facing material with high decorative properties (SiO_2–Na_2O–CaO, SiO_2–CaO–MgO, SiO_2–MgO–Al_2O_3 systems) or the production of fibrous materials using glass technology from rocks containing a high content of ferum oxides.

Developing thermal insulation materials based on iron-containing glass by foaming iron-containing glass is relevant and promising. The resulting foam glass has a cellular structure. It is characterised by high thermal insulation properties (0,05-0,07 M/(m·K)) and noise absorption up to 56 dB. Using highly ferruginous foam glass compositions requires optimisation of its production's chemical and physical processes.

Conclusion

Silicate glasses containing an increased amount of iron oxides and glass-crystalline materials based on them are studied in this paper. The DTA results confirmed that the proposed glass compositions have a high crystallisation capacity. Analysis of the XRD results confirmed the presence of a crystalline phase in glass-crystalline materials-magnetite (Fe_3O_4), which determines the magnetic properties of glasses. Studies of relative magnetic permeability have confirmed that the synthesised glasses have ferromagnetic properties. Thus, it is confirmed that high magnetic properties characterise iron-containing glass-crystal materials and can be used in high-tech devices.

Conflict of interest

The authors declare that there is no conflict of interest.

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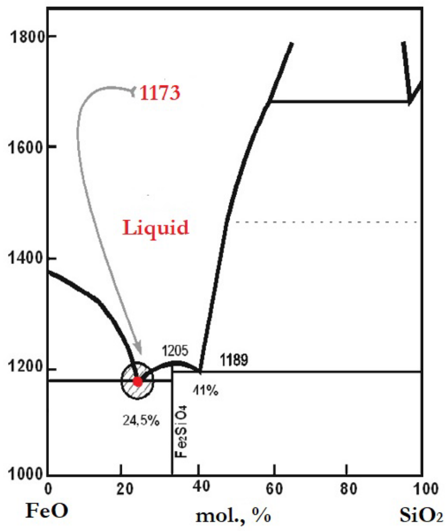
Appendix



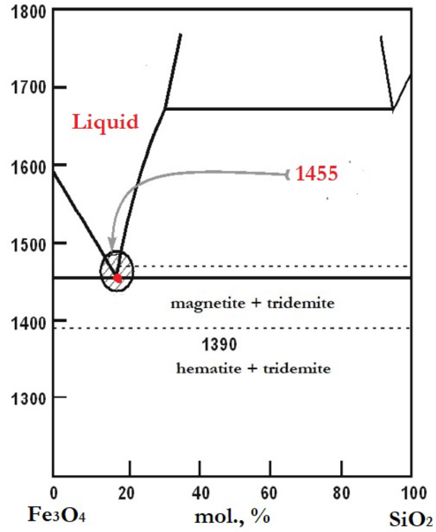


Figure 1. binary systems $FeO–SiO_2$ and $Fe_2O_3–SiO_2$

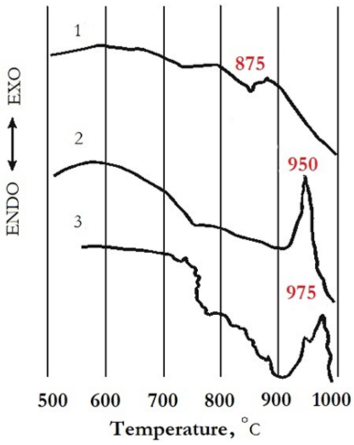


Figure 2. DTA results of glass samples

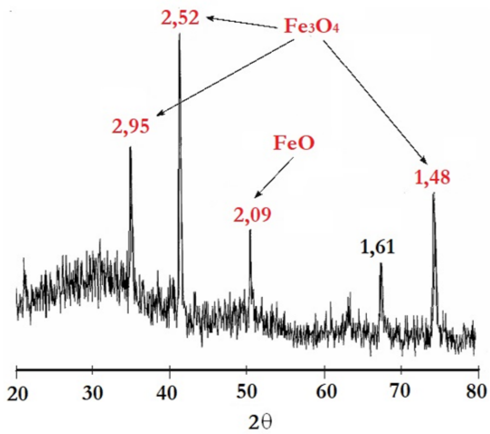


Figure 3. Diffraction pattern of composite glass No. 1

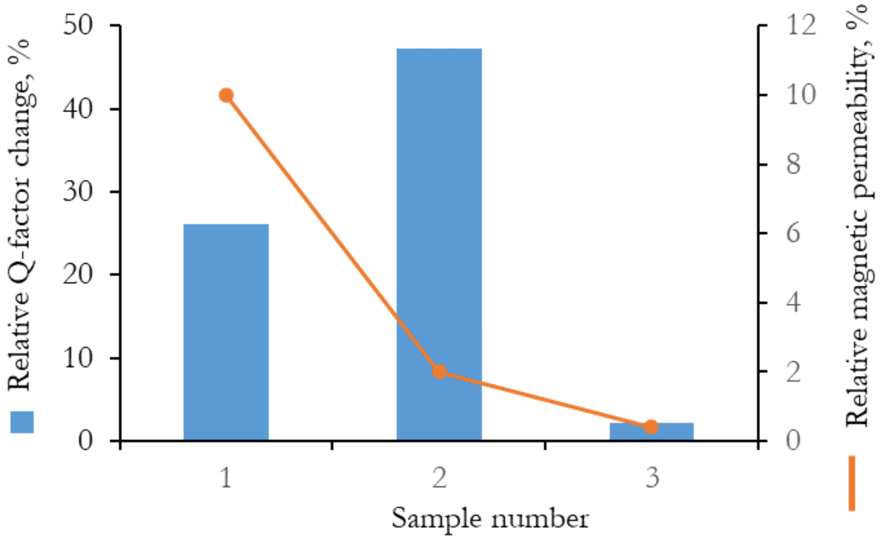


Figure 4. Relative change in contour Q-factor and relative magnetic permeability of glass

Table 1. Compositions of iron-containing glasses

No.	System	T,°C	Glass composition, weight %				
			FeO	SiO_2	CaO	MgO	Al_2O_3
1	SiO_2–FeO–CaO	1200	40	40	20	-	-
2	SiO_2–FeO–CaO–MgO	1150	40	37.5	13	9.5	-
3	SiO2–FeO–CaO–MgO–Al_2O_3	1220	17	53	8	7.5	14.5