



Investigation of the Properties of Metallurgical Slags and Dust of Electro Filters to Obtain Protective Anticorrosive Coatings

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Abstract. Corrosion-related irreparable metal failures can cause massive losses in various sectors, including agricultural engineering and construction. Steel structures are typically coated to withstand corrosion pressures during the service life specified in technical standards to avoid corrosion damage. This article presents research results on metallurgical cast iron slags and a mixture of slags with electro-filter dust to form silicate anticorrosive coatings. X-ray phase analysis, electron, and optical microscopy were used to analyze the surface nucleation of crystals in glasses using cast iron slags, a combination of steelmaking slag, and electro-filter dust with the addition of Cr_2O_3 . It is shown that the main phases crystallizing from the surface of the samples are diopside ($\text{CaO}\cdot\text{MgO}\cdot 2\text{SiO}_2$). When 1.5 - 2.0 wt.% Cr_2O_3 was added, the results showed that diopside phase glasses could be made with a cast iron slag level of up to 72 wt.%. Studies have shown the important role of Fe^{2+} , and Mg^{2+} , especially in samples containing cast iron slag, based on the most fusible compounds obtained. The optimal model of glass formation and crystallization was established as a result of the analysis of these compositions, and the microhardness of slag glass-crystalline materials was investigated. It was found that the hardness of the obtained glass-crystal materials increases in the presence of chromium oxide. The research revealed the possibility of synthesizing glass-crystal materials from cast-iron slags and dust of electro-filters.

Keywords: Glass-crystalline materials; Protective coatings; Slag; Three-layer panels

1. Introduction

The use of three-layer (sandwich) panels with adequate, effective insulation and protective coatings is a promising trend for the development of the construction industry, allowing for an increase in the volume of objects in industry and agriculture (Su et al., 2022). The primary material for three-layer panels is steel. Steel structures in agricultural construction are subject to severe corrosion damage due to high concentrations of animal waste, high humidity, and ammonia-phosphate fertilizers used in agriculture. To strengthen the corrosion resistance of steel and the attractiveness of the metal surface, the quality of the relevant protective coatings must be improved (Saraswati et al., 2018).

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The reduction of porosity, the structure of heterogeneity, and the composition are significant factors for enhancing protective coatings' physical and mechanical qualities. These reserves have been effectively utilized in the case of glass-ceramic coatings by using industrial waste. Compared to other coating materials, glass-crystal coatings feature chemical inertness, high-temperature resistance, and superior mechanical qualities such as scratch and impact resistance.

Glass and glass-ceramic coatings, in general, offer good adherence to a defect-free surface as well as fire resistance in addition to imparting the essential functional qualities such as heat, abrasion, and corrosion resistance to fulfil the specific requirements of the end-use (Dorofeeva & Semin, 2014). Thus, glass and glass-ceramic coatings are not only a new generation of coatings but also versatile engineering materials that extend the life of various types of metal substrates. They have a potential and promising market, and most likely, they can significantly replace industrial painting methods (Majumdar & Jana, 2001). The use of glass-crystal materials (sitals) in coatings proved particularly effective since it was able to strengthen heat resistance and protective characteristics at high temperatures practically without deterioration of the most significant technological features (wetting ability, covering capacity, and spreading), as well as to maintain a suitably low temperature at the start of softening, i.e., protective properties at low temperatures (Solntsev, 2007). Construction opportunities are significantly great. This is due to the availability of raw materials and smelting slag while retaining the valuable technological features of ceramics. Because of their high wear and chemical resistance, slag metals may effectively be used to safeguard building structures and equipment in the chemical, mining, and other sectors (Lazareva et al., 2009).

One of the work directions on slag disposal is their usage as the major base of mineral raw materials used to produce glasses and slag-glass. (Sycheva & Poljakova, 2016). Reuse of steelmaking slags will lead to improved quality characteristics of protective coatings, as well as to environmentally safe (Huang et al., 2012) and more efficient management of these wastes and preservation of the environment (Rincon & Romero, 1996; Rincon, 2016; Oluwasola et al., 2014; Zhao et al., 2016; Niyazbekova & Gladkikh, 2017; Maharaj et al., 2017; Sofyan et al., 2010). The use of blast furnace and metallurgical slags as the main crystal phase indicates a significant increase in the mechanical properties of composite materials. (Ponsot et al., 2014; Ashadi et al., 2015; Jexembayeva et al., 2020).

Metallurgical slags are the valuable raw material for obtaining protective silicate coatings for structures of livestock complexes under the influence of dangerous chemical and biological factors. Therefore, the task of using inexpensive secondary raw materials - slags of local production, the dust of electro-filters for the manufacturing process in slagositals, which will solve the environmental safety concerns of inhabited regions - was allotted to work (Sarkisov, 2001; Efimov et al., 2010). This research investigates the characteristics of metallurgical iron slags and a mixture of slags and electro-filter dust used to manufacture glass-crystal materials and their appropriateness as protective coatings for building structures.

This research also included experiments to manufacture glass-crystal materials based on metallurgical slags and electro-filter dust. The key components in producing synthesis glasses were cast iron slag, a mixture of steelmaking slag and electro filter dust, quartz sand, and tuffs. Chromium oxide was added to the glassy matrix, which can display isomorphism and increase crystallization stimulation (Yatsenko et al., 2012). Micro and nanocrystals can be formed during the heat treatment of glasses, contributing to the system's strength.

2. Methods

2.1. Materials

JSC "ArcelorMittal Temirtau" supplied the slags and all chemicals utilized in this investigation (Karaganda, Kazakhstan). The chemical composition of the starting materials was the basis for planning the composition of the mixtures. The mineralogical composition of slags and mixtures is represented mainly by silicate, iron-containing minerals, which create the prerequisites for the synthesis of glass-crystalline materials. As seen in Table 1, the primary elements are Ca, Fe, and Al, and the chemical composition of the slags is also shown in Table 1.

Table 1 Chemical compositions of waste

	Cast iron slag	Steelmaking slag + dust of electrostatic precipitators
Unbound SiO ₂	20.78	51.62
Sum SiO ₂	47.03	57.48
Al ₂ O ₃	13.32	2.19
Fe ₂ O ₃	9.11	33.73
TiO ₂	0.10	0.33
CaO	21.12	1.53
MgO	2.55	0.47
SO ₃	0.65	0.14
n.n.n.	2.63	2.93
Na ₂ O	0.37	1
K ₂ O	0.39	-
Σ	97.27	100.26

2.2. Mixing

The main components of slag are oxides of silicon, calcium, magnesium, aluminum, and titanium. X-ray analysis showed the presence of cristobalite, and glandular compounds in the waste. Raw mixtures were calculated from the main oxides contained in the waste. They were blended with a siliceous or calcium component to obtain a composition corresponding to the composition of the glass. The main raw materials for obtaining the initial glass were cast iron slag and quartz sand (Wang et al., 2019). To get glass crystals, 1, 1.5, and 2% Cr₂O₃ were added to the charge. The mixtures were mixed, placed in corundum crucibles, and melted in a furnace at 1200 - 1400oC for 3 hours. Table 2 shows the optimal glass compositions corresponding to low melting and crystallization temperatures.

Table 2 Glasses' material composition, %

Glasses' material composition, %
65% cast iron slag + 33.5% quartz sand +1.5% Cr ₂ O ₃
70% cast iron slag + 28.5% quartz sand +1.5% Cr ₂ O ₃
72% cast iron slag + 26.5% quartz sand +1.5% Cr ₂ O ₃
72% cast iron slag + 26.5% quartz sand +2%Cr ₂ O ₃
65% slag and dust of electrostatic precipitators + 32% tuff + 2% Cr ₂ O ₃
70% slag and dust of electrostatic precipitators + 27% tuff + 2%Cr ₂ O ₃

When using cast iron slags, the mixtures were melted at T 1200 - 1350°C. The resulting glasses were sharply cooled and then subjected to temperature treatment at 700 - 800°C for 3 hours. During the experiments, it was noted that at a temperature of 1200°C, glasses were already obtained. After heat treatment, the samples were subjected to petrographic studies. The X-ray method and electron microscopy were used to investigate the structure

of the crystalline glasses. The Vickers method was used on the PMT device to determine the microhardness of the samples. The crystalline characteristics of glasses were researched, and the heat treatment mode used to generate glass crystals with the necessary structure and composition was identified.

3. Results and Discussion

The compositions and melting points corresponding to the lowest melting temperature are shown in Table 3. Table 3 clearly shows that the melting point decreases as the percentage of quartz sand reduce from 33.5 – 26.5%. The temperature of crystallization has the same tendency. The glass-crystal materials based on metallurgical slags had the maximum microhardness ($655 \cdot 10^{-6}$ kg /mm²) when 70% cast iron slag + 28.5% quartz sand + 1.5% Cr₂O₃ were used.

Table 3 Properties of glass-crystal materials based on metallurgical slags

The material composition of the glasses, %	Melting point, °C	Crystallization temperature, °C	Microhardness, kg /mm ²
65% cast iron slag + 33.5% quartz sand + 1.5% Cr ₂ O ₃	1270	750	$585 \cdot 10^{-6}$
70% cast iron slag + 28.5% quartz sand + 1.5% Cr ₂ O ₃	1250	740	$655 \cdot 10^{-6}$
72% cast iron slag + 26.5% quartz sand + 1.5% Cr ₂ O ₃	1250	740	$565 \cdot 10^{-6}$
72% cast iron slag + 26.5% quartz sand + 2% Cr ₂ O ₃	1200	710	$540 \cdot 10^{-6}$
65% slag and dust of electrostatic precipitators + 32% tuff + 2% Cr ₂ O ₃	1200	710	$560 \cdot 10^{-6}$
70% slag and dust of electrostatic precipitators + 27% tuff + 2% Cr ₂ O ₃	1250	710	$565 \cdot 10^{-6}$

The Scanning Electron Microscopy (SEM) pictures of samples of crystallized slag-based glasses are shown in Figures 1 and 5. Cast-iron slags produced the lowest-melting-point glasses. The temperature of glass synthesis was 1300°C. The fine crystal structure of the glasses was obtained by heat treatment at a crystallization temperature of 800°C. Figure 1 depicts the result of crystallization of cast iron slag-based glasses, crystals of α tridymite, diopside (CaO·MgO·2SiO₂) (d = 2.98; 2.523; 1.744; 1.616; 1.418). X-ray analysis also revealed the existence of MgO·Al₂O₃ spinel (d = 2.41; 1.41; 2.01).

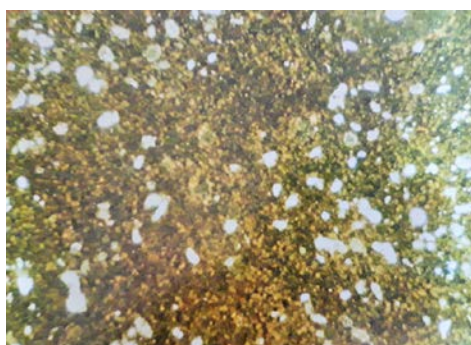


Figure 1 SEM of glass based on cast iron slag, x280

Glass-crystalline materials were created from a mixture of slag and dust from electrostatic precipitators, and the main minerals of which are: spinel (white octahedra) as shown in figures (2 and 3); iron compounds hematite - 2Fe₂O₃, (d = 2.69; 2.51; 1.69),

hedenbergite - $\text{FeO} \cdot \text{CaO} \cdot 2\text{SiO}_2$ ($d = 2.98; 2.23; 2.94$). X-ray analysis also revealed traces of augite - $\text{Ca} [\text{Mg}, \text{Fe}^{2+}, \text{Al}, \text{Fe}^{3+} (\text{Si}, \text{Al})_2\text{O}_6]$ ($d = 1.41; 1.32; 1.07$).

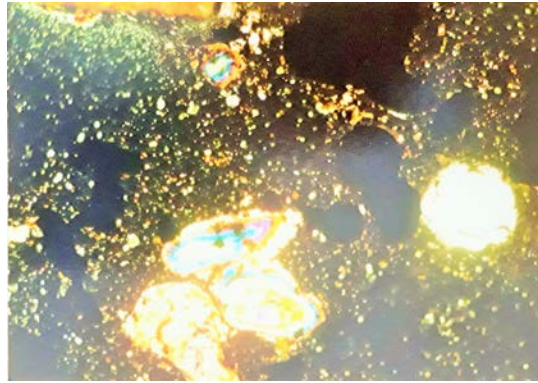


Figure 2 SEM of crystallized glasses based on slag and dust of electrostatic precipitators, x280

Figure 3 shows the structure of glass-crystal materials based on a mixture of electro filter slags and dust with a 2% Cr_2O_3 addition. While figure 2 reveals how the structure of samples without Cr_2O_3 varies and becomes more fine-grained.

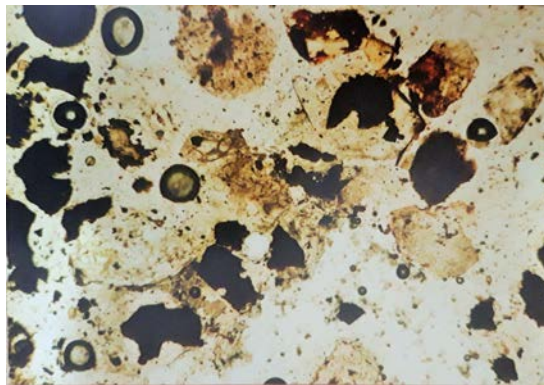


Figure 3 SEM of crystalline glasses based on electrostatic precipitator slag and dust with 2% Cr_2O_3 , x280

Segregation is significant in the crystallization process because it accelerates under certain ideal conditions (Sycheva, 2016, 2017, 2019; Sycheva & Poljakova, 2013, 2016; Saraswati et al., 2018). Figure 4 shows delamination that occurred during the heat treatment. Segregation most likely has a positive effect on the process of crystal nucleation and volume crystallization (Fredericci et al., 2000; Dargaud et al., 2011; Strokova et al., 2020), as shown in Figure 5.

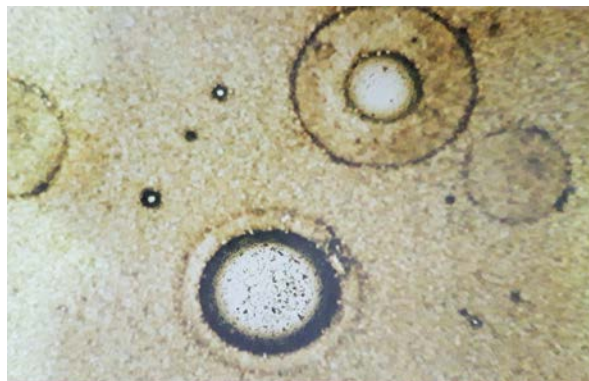


Figure 4 SEM: Micro liquation of glasses, x280

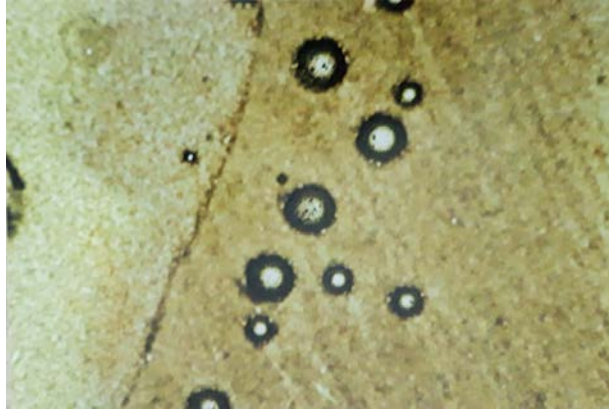


Figure 5 SEM: The nucleation of crystals in glasses, x280

It is assumed that Fe^{+2} ions located at sites of silicon-oxygen temperatures depolymerize the silicon-acid structure, and the melting point decreases. The melting temperature of the compositions rises as the acidity of the slag rises. With increasing acidity, silicon-oxygen complexes increase, with high binding energy between the anions and the surrounding ions. As a result, metal ions cannot freely move and disrupt the structure of systems.

Infrared spectra were recorded on an FT-IR Spectrometer Nicolet iS10 4000.0 – 400.0 cm^{-1} . FTIR spectra of iron slag + quartz sand + Cr_2O_3 are displayed in Figure 6.

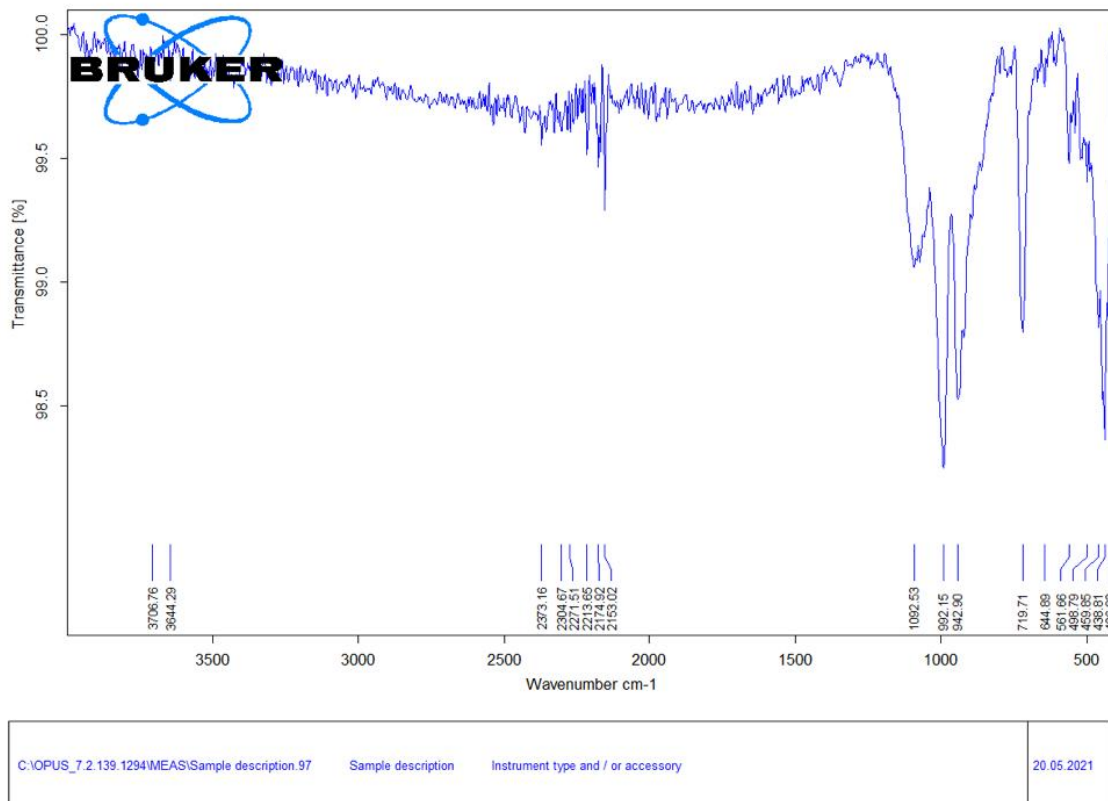


Figure 6 FTIR spectrum of iron slag + quartz sand + Cr_2O_3

The FTIR spectrum showed peaks at 992, 942, and 712 cm^{-1} (–Si-O stretching). Thus far, data show patterns such as a shift in the Si-O band (712 to 992 cm^{-1}) towards a higher wavenumber with increasing silica content. [Sowmya and Sankaranarayanan \(2004\)](#) conducted similar research in 2004. The peaks between 900 – 1000 cm^{-1} are assigned to (–

Si-O-Me), the same result as reported in the work of Arkles (2015), and at 2118 cm^{-1} is assigned to (-vestal oscillation area). The bands between 400 and 440 cm^{-1} indicate the existence of metals in the glass; they are inherent in the glass's structure. The metal oxides in the slag are mixed into the form of glass. The bands from 1000 to 1200 cm^{-1} show the degree of polymerization of the silicon-oxygen framework of the melt. The lower the intensity and the intensity ratio to half-width at half-height, the greater the superheating of the melt above the liquidus.

4. Conclusions

Cast iron slags are often used to make pyroxene glasses. All types of low-melting glasses are explained, in conclusion, by the fact that iron ions depolymerize the structure of melts and glasses, reduce the viscosity and temperature of structural changes, increase the tendency of melts to microliqutation, and actively participate in the nucleation of crystals. The crystallization temperatures of glasses of optimal composition are determined. Based on the conducted research, the compositions of glass-crystal materials were developed. As a result of studying these compositions, the optimal glass formation and crystallization model of glasses was established, and the microhardness of slag glass-crystalline materials was investigated. It was found that the hardness of the obtained glass-crystal materials increases in the presence of chromium oxide. The research revealed the possibility of synthesizing glass-crystal materials from cast-iron slags and dust of electro-filters.

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