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ANALYSIS OF SLAG PROPERTIES PRODUCED DURING THE HIGH-TEMPERATURE WASTE TREATMENT

Marian Lázár *, Mária Čarnogurská, Tomáš Brestovič, Natália Jasminská, Romana Dobáková
* Department of Power Engineering, Faculty of Mechanical Engineering, Technical University of Košice, 042 00 Košice, Slovak Republic

ABSTRACT
The publication aims to provide the basic information about the mechanical properties and phase transformation temperatures of the vitrified slag generated during the high-temperature smelting of a variety of inorganic wastes in a plasma reactor. In order to characterize the material, the hardness according to Vickers, micro hardness, and Young's modulus of elasticity and fracture toughness were monitored. The phase analysis of these samples was also performed.

KEYWORDS: vitrified slag, plasma technology, mechanical properties.

INTRODUCTION
The significance of the separation and the subsequent recovery or disposal of any waste has a great merit in any sector of the national economy. Waste often contains various valuable raw materials with relatively high energy and material potential. Recovery of raw materials and energy, just from waste, is of environmental importance in addition to economic importance. Equally worthwhile is the disposal of waste, which cannot be recovered at present by any other recycling processes.

One way to dispose of inorganic hazardous waste is to smelt it in a plasma reactor. During the high temperature treatment, the waste is smelted in a plasma reactor with a flux so as to form a glassy oxidic slag, which is then granulated or cast into molds. The glassy structure of the slag ensures the binding of some unwanted components in an extremely stable glass matrix. The output products of the process are also synthesis gas, alloy and particle matter obtained in gas (fly ash). Synthetic gas, representing the primary gaseous product of the smelting process is, after purification and with sufficient heat, usable for energy purposes. Both, the obtained metal alloy as well as the metal-containing fly ash is captured as a secondary raw material in the metallurgical industry. The use of vitrified slag on the market with the secondary products is more difficult than with any other primary products obtained in the smelting process.

The slag formed during the plasma waste smelting process is a multi-component system consisting of the major slag-forming oxides represented in the batch. In view of its possible future use, it is therefore necessary to know the mechanical properties as well as the phase changes occurring during its heating.

EXPERIMENTAL VERIFICATION OF THE SLAG PROPERTIES
The determination of the mechanical properties of the slag from the plasma treatment was preceded by the pre-treatment itself. The pre-treatment of samples of the cooled slag consisted of sanding and polishing the surface of the uneven surface of the slag which was created by the cooling process as well as the formation of internal stresses [1], [2].

The hardness of the vitrified glass slag was verified by the Vickers hardness test. For the indenting process, the diamond pyramid (indenter) with an angle of 136 ° was used along with the force of 9.81N, and 29.42 N, respectively. With a loading time of 10s, the hardness values in Vickers units (Tab. 1) [3] were obtained for each sample.

Table 1. Mechanical properties of slag

Mechanical property	Sample type		
	Sample I	Sample II	Sample III
Hardness according to Vickersa [HV]	673±20	618±23	620±18

Legend: Sample I - is slag obtained during the smelting of the asbestos-cement roofing material (ACRM) and of fly ash from the fluidized boilers (the cooling process took place in form of a free convection); Sample II - slag produced during the smelting of the ACRM and the fly ash from fluidized bed boilers (cooling process was carried

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out by free convection of the mold (15 min), followed by the immersion of the material in a water bath) Sample III - slag from the smelting of fly ash captured in the cleaning circuit incineration of municipal waste

In order to characterize the material in a wider range, an instrumental indentation analysis was carried out to capture the essential properties of the material examined under a very low force. The following were studied: Young's Elasticity Module (E) and Micro-hardness (H).

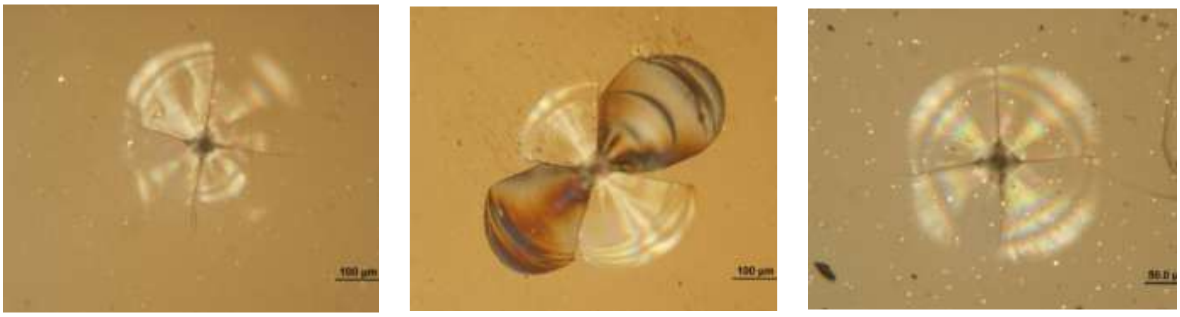
Knowing the temperature characterizing the phase transformations of the vitrified slag obtained from the analyzed plasma treatment of waste is necessary information for designing, for example, the manufacturing process of a further recovery of the slag into the form of an insulating material. To determine the phase composition and phase transformations of slags produced in the high-temperature melting process of inorganic wastes, the following analyses were carried out:

- XRD analysis (X-ray diffraction),
- TG analysis (thermogravimetric analysis),
- DSC (differential scanning calorimetry).

RESULTS AND DISCUSSION

Phase analysis of slag was performed by using a Philips X'PERT PRO X-ray diffractometer. For the x-ray diffraction measurements, the Cu anode (wavelength Kα1 = 0.154056 nm; K α2 = 0.154439 nm at IKα2 / IKα1 intensity = 0.5) was used. The 2Theta (2Θ) measuring range was 20 to 100o at step of 0.03342o (measurement time at each step of 40s). Prior to the measurement, the powdered samples were poured into the sampler and its surface was finely aligned. Based on the observations and results of the X-ray diffraction recording, Sample II is amorphous with a very small proportion of the crystalline phase of gehlenite. Sample III is also amorphous. Sample I, the cooling of which was in the form of a free convection in the cast iron mold in the air, is polycrystalline. It consists of a phase known under the mineralogical designation of gehlenite of the composition Al1.25Ca1.96Fe0.12Mg0.24Na0.05O7Si1.39 [4, 5].

Sample I shows the highest hardness values under both forces. This property is attributed to the arrangement of the polycrystalline structure, which contains crystalline and amorphous phase joined by a smooth transition. The photographic documentation of the typical deformation of the slag surface caused by the regular quadrangular pyramid is shown in Fig. 1. The indentation of the tip in a perpendicular direction resulted in a set of fissures typical of brittle materials. High brittleness was also confirmed by the development of lateral cracks leading to the splitting of larger pieces from the surface (Figure 1).



Sample I Sample II Sample III

Figure.1 Deformation of a slag sample caused by a regular four-pillar needle

In the process of determining the level of hardness, the instrumented indentation prefers the triangular Berkovich indenter, which was also used for experiments performed on samples of the vitrified slag. The values of the micro-hardness and Young's modulus of elasticity were compared with the values of the calibration sample, which is the (vitreous) silica glass. The load force was 50 mN with 5 a second hold time. The typical shape of the indenter measuring the micro-hardness for Sample I is captured in Fig. 2. The measured values of micro-hardness and Young's modulus of elasticity are shown in Tab. 2

Table 2. Micro-hardness and Young's elasticity modulus of vitrified slag samples

Mechanical property	Sample type			
	Sample I	Sample II	Sample III	Vitreous silica
Micro-hardness [HV0,005]	1102±39	971±51	958±21	1111±24

Young's modulus of elasticity [GPa]	115±1	124,7±7	109±0,8	70±0,6

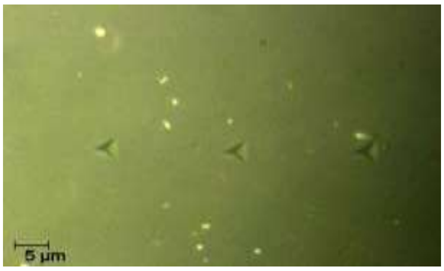


Figure 2. Shape of the indenter measuring the micro-hardness for Sample I

The calculation of the fracture toughness by the indentation method is based on the formula designed for the cracks of the semi elliptical crack shape, typical of brittle materials [6].

$$K_{IC} = 0{,}016 \cdot \left(\frac{E}{H}\right)^{0,5} \cdot \left(\frac{F_p}{x^{1,5}}\right) \qquad (1)$$

Where E is Young's modulus of elasticity (GPa), H - hardness (GPa), Fp - indentation force (N), x - crack length (m).
Low fracture toughness values are in a direct relation to the brittleness of the material. The measured values are shown in Tab. 3.

Table 3. Fracture toughness of vitrified slag specimens

fracture toughness	Sample type		
	Sample I	Sample II	Sample III
$K_{IC}\left(MPa \cdot m^{\frac{1}{2}}\right)$	0,274±0,03	0,264±0,03	0,074±0,01

Phase transformation temperature analysis was performed using the simultaneous thermal analyzer (NETZSCH). The DTA-DSC-TG thermal density analyzer Jupiter STA 449 F1 series was used to determine the temperatures of the slag phase transformations. The slag samples placed in Al2O3 crucibles were heated under an argon atmosphere at a heating rate of 10 ° C • min-1. The maximum heating temperature was 1400°C.

In the case of Sample II, the crystallization process was initiated at 898.9°C (Fig.3). Its melting point is 1158.4°C. Phase changes in Sample I (Fig. 4) commence from 909.4°C. The melting point is 1181°C. The phase transformation temperature analysis of the Samples III speak of a slight endothermic transformation in the temperature range of about 735°C, which can be considered as the start-up temperature of the phase changes in the analyzed sample. The start of the melting of the sample is set at 1115°C.

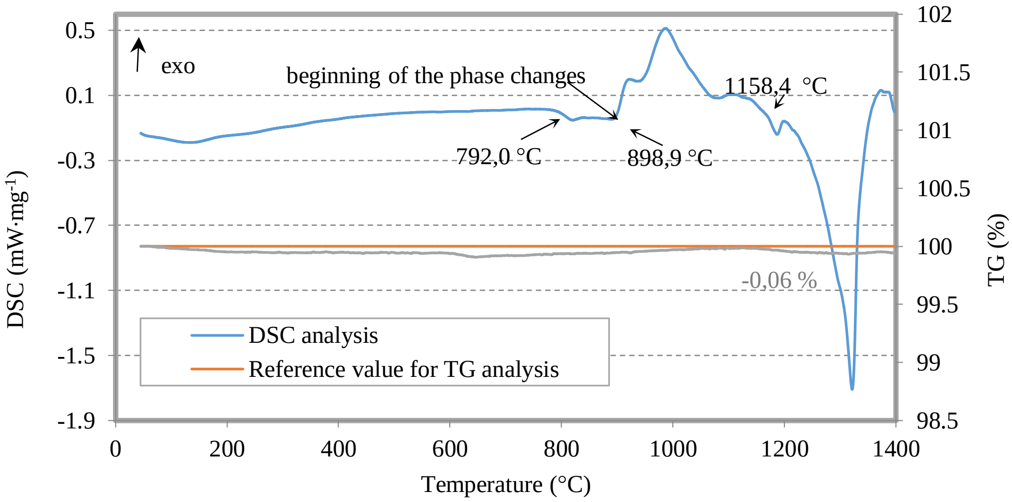


Figure. 3 Recording from the DSC and TG analysis of the Sample II

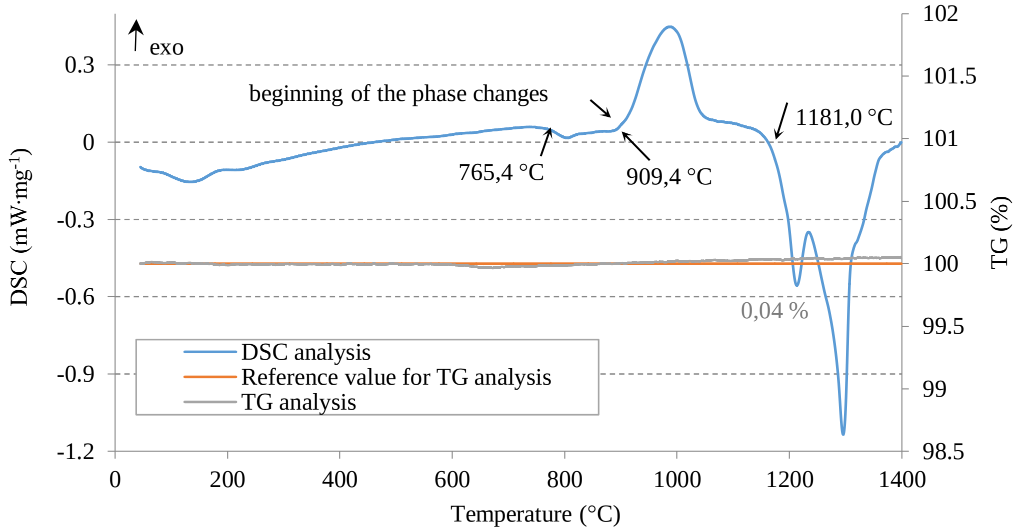


Figure. 4 Recording from the DSC and TG analysis of Samples I

In both specimens (Sample I and Sample II) it was observed that a slight endothermic change occurred before the phase changes themselves: Sample II was at 792° while Sample I was at 765°C. This change is followed by an exothermic transformation itself - crystallization or other phase transformation.

The weight loss of the monitored samples over time and temperature was monitored by the thermogravimetric measurement. Thermogravimetric records did not show any significant changes in the weight of slag samples obtained by the melting process of ACRM and fly ash from the fluidized boilers, whereby confirming the high chemical stability of slags. Total weight loss for Sample II was 0.06 wt. %. A weight loss of 0.04% was recorded for Sample I. %. In the case of Sample III it is possible to talk about a weight increase of about 0.05% by weight. This increase could be triggered by the oxidation of the sample as the suction of the atmosphere from the furnace chamber before the start of the experiment was not 100% (about 99%).

CONCLUSION

Recently, the EU has been aiming to reduce the environmental and health risks associated with waste management and disposal. Deviating from the most widespread way of waste management, such as landfilling, requires, in addition to legislative changes, in particular a change of the perception of society as a whole. Selection of the methodology for the treatment of a particular type of waste is significantly affected, in addition to its physical and chemical properties, by the economic development of the country, as well as by the population's attitude toward the environment and by the cost of its disposal. It is the task, for the above reasons, to search for such processing practices whereby the material and energy content of waste is capitalized as much as possible, with the minimum amount of otherwise unusable fraction of waste going to landfills.

By the high-temperature pre-treatment of inorganic wastes analyzed in this paper a vitrified slag is obtained - a product that is inert, innocuous and environmentally friendly according to current legislation. However, its further appreciation is conditioned by its mechanical and thermo-chemical properties.

By analyzing the results obtained, it can be stated that from the point of view of the pre-treatment of slag, the difficulty of its crushing is comparable to the difficulty of preparing the glass powder from the waste glass. The use of slag in the construction industry in the form of a filler substitute for concrete mixtures and in the form of raw materials suitable for road construction therefore requires optimization of the ratio of the composition of the individual compounds as well as the change in the cooling process of the slag. An interesting area of exploitation of the molten slag is its application in the production of insulating materials (foam glass, mineral wool). Research in the area is subject to current scientific research.

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