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THE INVESTIGATION OF GROUND GRANULATED BLAST FURNACE SLAG GEOPOLYMER AT HIGH TEMPERATURE BY USING ELECTRON BACKSCATTER DIFFRACTION ANALYSIS

This paper elucidated the potential of electron backscatter diffraction analysis for ground granulated blast furnace slag geopolymers at 1000°C heating temperature. The specimen was prepared through the mechanical ground with sandpaper and diamond pad before polished with diamond suspension. By using advanced technique electron backscatter diffraction, the microstructure analysis and elemental distribution were mapped. The details on the crystalline minerals, including gehlenite, mayenite, tobermorite and calcite were easily traced. Moreover, the experimental Kikuchi diffraction patterns were utilized to generate a self-consistent reference for the electron backscatter diffraction pattern matching. From the electron backscatter diffraction, the locally varying crystal orientation in slag geopolymers sample of monoclinic crystal observed in hedenbergite, orthorhombic crystal in tobermorite and hexagonal crystal in calcite at 1000°C heating temperature.

Keyword: Geopolymer; Ground granulated blast furnace slag; Electron backscatter diffraction

1. Introduction

Geopolymer is a kind of inorganic material with ceramic-like properties that is composed of amorphous phase and semi-crystalline phase [1-5]. The geopolymer technology was implemented to recycle abundant steel waste with pozzolanic properties, such as, ground granulated blast furnace slag [6-8]. Generally, geopolymer can be synthesized at elevated or ambient temperature for curing purpose, by activation ofaluminosilicate sources with an activator composed of silicate and aqueous hydroxide. After contact with alkaline activator solution, the precursor material is dissolve, and the polycondensation is occur simultaneously. Amorphous geopolymer are obtained at temperatures within the range of 20-90°C while crystalline phase formed in furnace at temperature up to 1200°C. Sintering at high temperatures contributes to the formation of ceramic product due to the formation of crystalline phase.

Literature indicates that the geopolymer phase was influenced by heating treatment at higher temperatures. Nepheline ceramics have formerly been formed through sintering-crystallization, controlled devitrification and vitrification process of fly ash and slag [9] in the temperature range of 600-1200°C. I.H. Azis et al. [10] obtained the formation of crystalline phase of gehlenite (Ca_2Al_2SiO), mayenite ($Ca_{12}Al_{14}O_{33}$) and larnite (Ca_2SiO_4) filled the slag-based geopolymer and formed an ordered structure up to 1000°C heating temperatures. Meanwhile, the crystallization of alkali activated slag/metakaolin up to 1200°C delivered worst performance of strength as study by Burciaga-Diaz et al. [11]. The details of crystalline structure in which can be explained by microstructure properties required for understanding the geopolymer behavior at high temperature environment.

The analysis of the microstructure material surface delivers essential information for the understanding of their characteristic. Electron backscatter diffraction (EBSD) is an advanced practical tool for micron scale crystallographic analysis of materials connected with the scanning electron microscope (SEM) [12]. EBSD provides resolved crystallographic data by measuring backscattered Kikuchi diffraction patterns formed by incoherent

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point sources of backscattered electrons within a single crystalline volume [13]. Kikuchi pattern possessed clear advantages compared to other diffraction techniques, such as provides a wide-angle view on the potential point group symmetries of the crystal phases that are probed locally by the electron beam. Commonly, the advanced technique of EBSD can still be utilized for orientation determination even of unspecified phase due to the ability that the experimental Kikuchi pattern is lesser basically to set of linear features extracted by Hough transform as one of the initial steps of the EBSD data processing [14]. Meanwhile, the sensitivity of EBSD system phase discrimination is limited with the geometrical images analysis Kikuchi patterns by the Hough Transform.

Previously, this advanced technique performed with scanning electron microscopy (SEM) was used to evaluate crystal orientation in ceramic samples [15-16], steel materials [17] and electronic materials [18-19] but which has not previously analyzed for geopolymer materials. The microstructure crystalline surface, Kikuchi pattern and phase fraction were characterized by using electron backscatter diffraction (EBSD) in this current work. Additionally, the details of the crystal orientation of each crystalline mineral formed in the samples also can be determined.

2. Material and method

2.1. Sample preparation and experimental details

The geopolymer samples were formed by activating the precursor material of ground granulated blast furnace slag (GGBFS; Ann Joo Integrated Steel Sdn. Bhd) with alkaline activator solution. The liquid alkaline activator was prepared by mixing sodium hydroxide (NaOH, 10M) with sodium silicate (Na_2SiO_3), supplied by South Pacific Chemical Industries Sdn. Bhd., Malaysia consists of 30.1 % SiO_2, 9.4% Na_2O, and 60.5% H_2O.

The sample was fabricated using solely GGBFS activated with pellet sodium hydroxide and sodium silicate solution, at solid-to-liquid and alkaline activator ratio of 3.0 and 2.5, respectively. The raw slag powder was stirred with an alkaline activator solution for 5 min before casting into a 2.5 mm diameter × 3 mm height cylinder at ambient temperature for 28 days. The hardened sample was heated at 1000°C (heating rate of 10°C/min) for 1 hour by using a standard electrically-heated furnace.

2.2. Characterization

For the EBSD technique, the sample is required to obtain a mirror-like surface. Thus, the details of the grinding method should be to be considered. For the first step, the slag sample was ground with abrasive paper (2000 mesh). Subsequently, electroplated diamond lapping plates or silica carbide abrasive paper with 20 μm, 74 μm, and 250 μm were used in the lapping process and then polished with a diamond suspension (diamond particle diameter: 9 μm, 6 μm, and 1 μm). The specimen was analyzed with scanning electron microscopy (SEM) and optical microscopy to confirm the mirror-like surface, as shown in Fig. 1a and 1b.

The sample was investigated by electron backscatter diffraction (EBSD) measurement using an acceleration voltage of 25 kV at a working distance of 24 mm. For EBSD analysis, specimens were titled at an angle of 70°, and the data was collected by using Emax Evolution software. All the details specimen's preparation for EBSD characterization is depicted in Fig. 2.

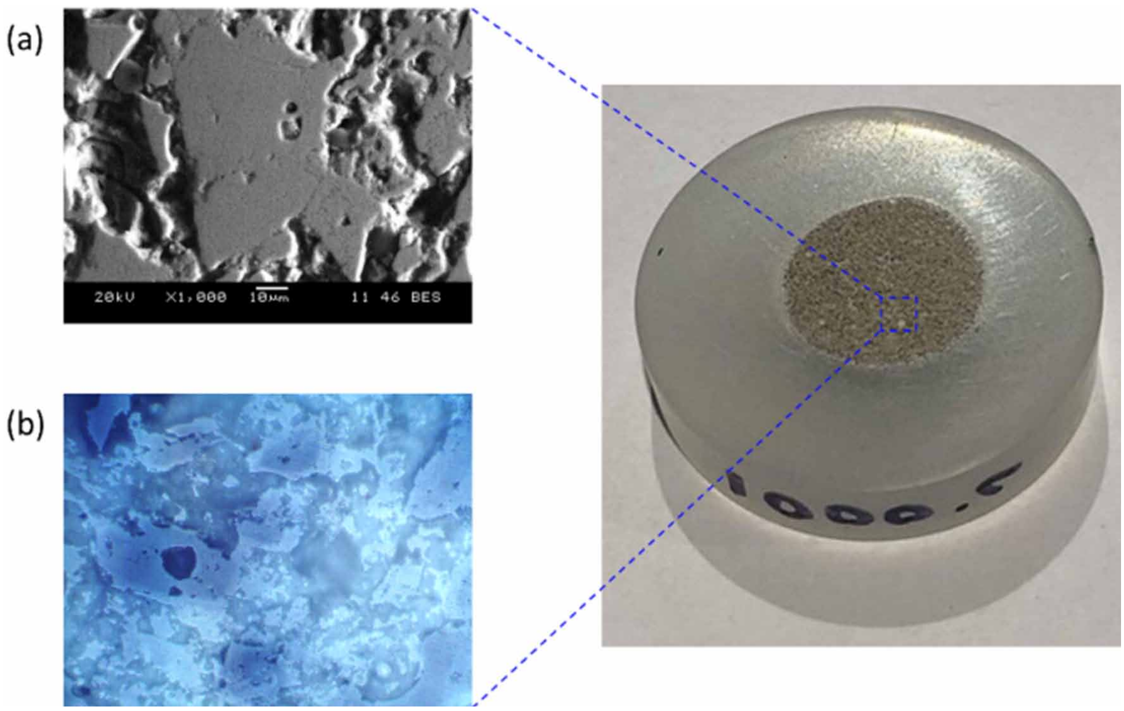

Fig. 1. Mirror-like surface of slag specimen by using (a) SEM analysis and (b) OM analysis

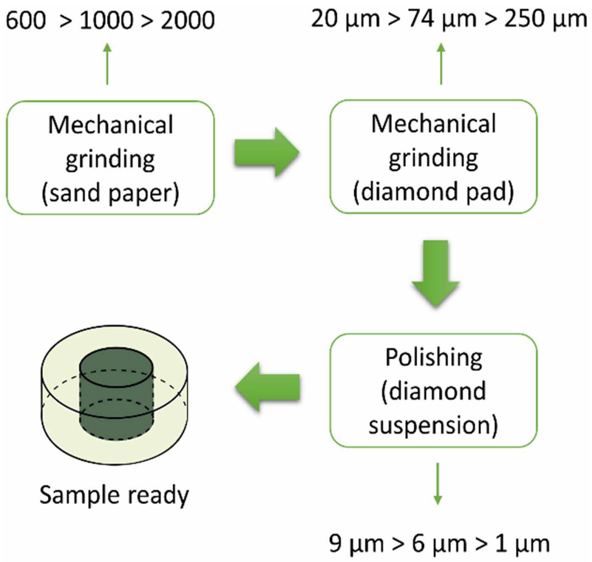

Fig. 2. Grinding and polishing process of slag specimen for EBSD analysis

3. Result and discussion

3.1. Microstructural and element distribution

The heated slag specimen was subjected to scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX) to analyze the elemental composition and surface structure. Fig. 3 illustrates the distribution of Si, Al, Ca, O, Fe, and Mg elements; signifying Al and O are mostly located within the heated slag geopolymers. As noted in the SEM images, the appearance of pores has been due to hydration at higher heating temperatures. Through the use of EBSD-EDX analysis, the distribution of elements that could reflect the mineral within the slag geopolymer structure can be determined.

Table 1 tabulates the details of phase mineral within the heated slag geopolymers at 1000°C. The table shows the appearance of gehlenite ($Ca_2Al[AlSiO_7]$), mayenite ($Ca_{12}Al_{14}O_{33}$), calcite ($CaCO_3$), tobermorite ($Ca_5Si_6O_{16}(OH)_2.4H_2O$), diopside ($MgCaSi_2O_6$) and hedenbergite ($CaFeSi_2O_6$). This was correlate with the SEM-EDX analysis as depicted in Fig. 1.

TABLE 1

Details of phase mineral of the slag geopolymers at 1000°C

Phase	a	b	c	Space Group
Gehlenite	7.74 Å	7.74 Å	5.05 Å	113
Mayenite	11.98 Å	11.98 Å	11.98 Å	220
Calcite	4.99 Å	4.99 Å	17.06 Å	167
Tobermorite	5.59 Å	3.70 Å	22.78 Å	44
Diopside	9.73 Å	8.91 Å	5.26 Å	15
Hedenbergite	9.81 Å	8.99 Å	5.22 Å	15

3.2. Kikuchi Pattern and crystal orientation

The image quality (IQ) mapping of the slag geopolymers sample at 1000°C heating temperature was shown in Fig. 4a.

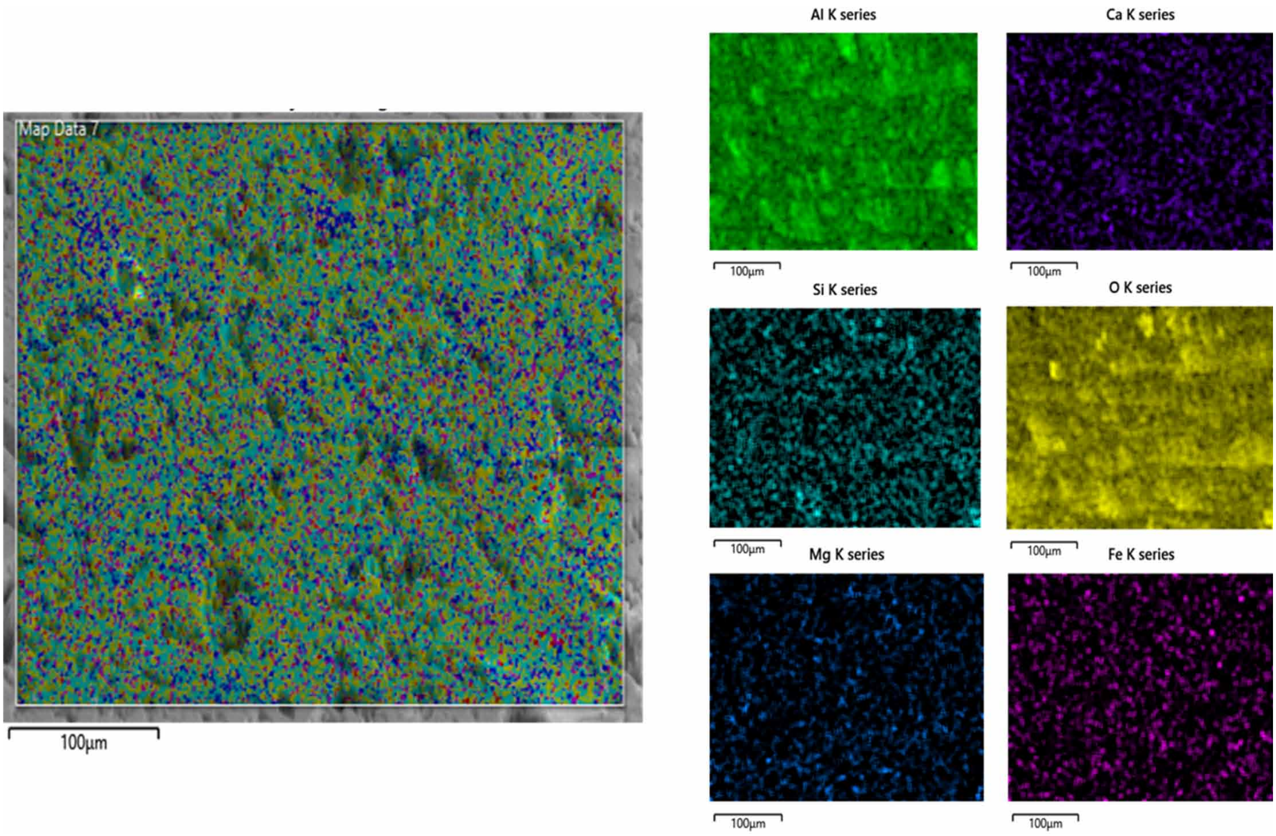

Fig. 3. EBSD-EDS elements mapping of the slag geopolymers at 1000°C

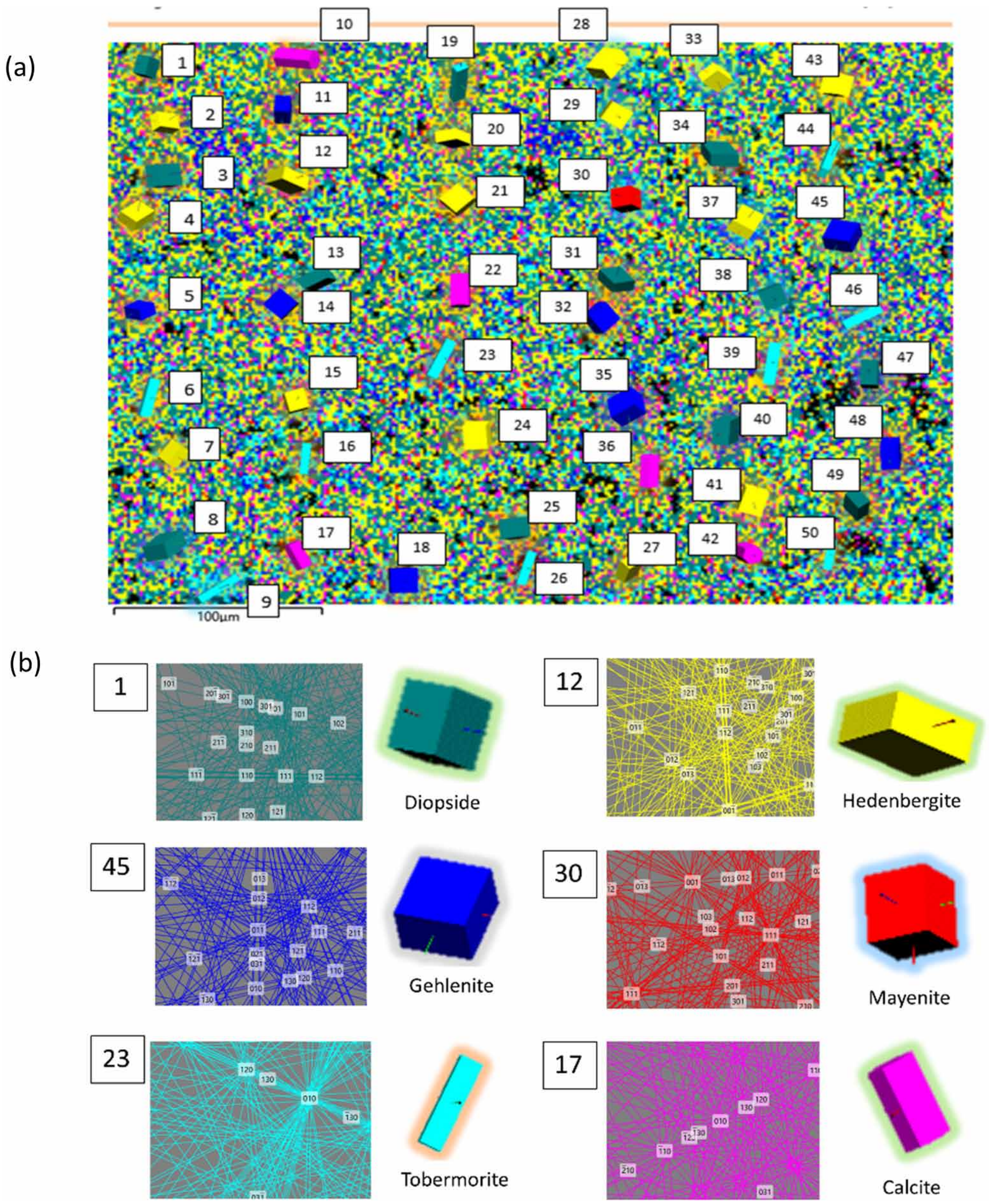

Fig. 4. EBSD analysis of (a) crystal orientation and (b) kikuchi pattern of slag geopolymer at 1000°C heating temperature

In the IQ maps, the black color denotes a zero solution, which are points of no indexing solution for the corresponding pattern. The various colors represent the crystal minerals distribution in the integrated area. Crystalline phase identification of individual mineral is localized in different colors in different areas within the sample. By referring to Fig. 4b, gehlenite (blue color) and hedenbergite (yellow color) dominated the sample surface of phase fraction.

The relatively equiaxed grain obtained with the surface of the whole sample of slag geopolymers consisting of diopside, mayenite, calcite, and tobermorite at 1000°C heating temperature details of crystal orientation are presented. All of these calcium-based minerals commonly formed in the eutectic mixture [20]. Otherwise, electron backscatter diffraction technique can provide details on the misorientation angle, inverse pole figure, texture analysis, grain boundaries and strain localization which are not been carried out in this paper.

4. Conclusions

The crystalline phase of geopolymers are potential to be analyzed by using electron backscatter diffraction (EBSD). However, the samples preparation needs to be considered in details. The mirror-like surface is essentially required. The elemental mapping, phase fraction, grain boundaries and crystal orientation could be obtained by using this advanced technique. By referring to this current work, in summary, ground granulated blast furnace slag geopolymers heated at 1000°C formed crystalline phase. The appearance of calcium-based mineral such astobermorite, calcite and mayenite were located within the sample surface.

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