New Approach to Distinguish Copper Molten Marks Based on Quantitative Microstructure Analysis Using Electron Backscatter Diffraction

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Received: 23 May 2020/**Accepted:** 27 November 2020/**Published online:** 11 January 2021

Abstract. Molten marks identified at a fire site can aid in determining the cause of a fire. Quantitative analysis of such molten marks has not yet been reported, despite several identification methods for molten marks being proposed based on optical micrographs. Herein, we propose a new methodology to establish the quantitative microstructure parameters of the molten marks using electron backscatter diffraction (EBSD). The globules were generated by heating the copper conductor to 1100°C in a non-energized state. Then, the arc beads were prepared by shorting the copper wire at 25°C and 900°C in an energized state. The globules did not show evident demarcation lines and indicated that the microstructure consisted of globular or dendritic grains; this demonstrated that their characteristics were distinctly different from those of arc beads. However, the primary arc beads (PABs) shorted at 25°C exhibited a strong (001) texture perpendicular to the demarcation line and comprised large fractions of columnar grains with a small grain aspect ratio (GAR). The microstructure of secondary arc beads (SABs) shorted at 900°C presented a mixture of elongated and equiaxed grains with a large GAR and no specific development of texture. The GAR and (001) fraction perpendicular to the demarcation line could be discriminant

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parameters of PABs and SABs. Consequently, EBSD methods can be newly applied for the classification of globules, PABs, and SABs based on the quantitative microstructure information and orientation distribution.

Keywords: Copper, Molten mark, Globule, Arc bead, Electron backscatter diffraction (EBSD), Microstructure

1. Introduction

Molten marks refer to rounded masses of melted copper. Molten marks are classified into two categories [1, 2]: globules melted by fire and the arc beads melted by electrical energy. Arc beads can be further divided into primary arc beads (PABs) and secondary arc beads (SABs). Globules are molten marks formed at high temperatures above the melting point (1083°C) of copper in a non-energized state. Arc beads are molten marks formed when a significant short-circuit current surges through an electrical wire. PABs are molten marks resulting from the initial short circuit that acts as a source of ignition. SABs are molten marks formed due to a short circuit that occurred in an energized state after the wire insulation had been burned off by the flames. Therefore, the molten marks identified at a fire site can aid in determining the cause of the fire.

Molten marks can be predicted to exhibit different solidification behavior according to the ambient temperature, heat transfer, and Joule heat. This is because ambient temperature causes changes in the solidification parameters such as temperature gradient and cooling rate. These changes eventually affect the microstructure, such as the grain size and shape, in the molten marks. Several classification methods for molten marks have been proposed, and metallographic studies have been actively conducted to analyze microstructure distinctions using the optical microscope [3–7]. Though the analysis of optical micrographs is a more scientific method than the observation of the appearance, it is difficult to derive quantitative discriminant parameters because optical microscopes have a limitation of resolution and cannot analyze crystallographic orientations of microstructures. To observe fine grains with sizes under several tens of microns, proficient researchers and proper etching conditions are integral. An automatic orientation mapping system based on the scanning electron microscope (SEM) was developed in the late 1990s, called electron backscatter diffraction (EBSD) [8]. EBSD measurement is widely used for micro-scale microstructure and crystallographic features, and quantitative microstructure analysis is possible from pixelated orientation information. In this study, using these advantages of EBSD, we propose a new method for quantitatively classifying molten marks. To this end, EBSD analysis of molten marks was performed and microstructure parameters were deduced to distinguish among globules, PABs, and SABs. It should be noted that this study is applicable to the limited conditions of bare wires contacting with direct flame. Other cases such as the insulated wiring exposed to thermal radiation in a compartment fire are beyond the scope of the study.

For the deduction of quantitative discriminant parameters, non-energized copper wires were melted at 1100°C to generate globules, and energized wires were

short-circuited at room temperature (25°C) and high temperature (900°C) to create PABs and SABs; the terms “PAB” and “SAB” used herein are limited to arc beads generated at 25 and 900°C, respectively. This study employed an EBSD technique to distinguish the molten marks based on the microstructure such as grain size, shape, and local orientation distribution; quantitative parameters were suggested to distinguish among globules, PABs, and SABs.

2. Materials and Methods

In order to generate globules, the wire was fixed to the holder in a non-energized state, and the ambient temperature was raised to 1100°C. The globules were formed after approximately 20 s from flame initiation. PABs were prepared by forming a short-circuit at room temperature (25°C) without using a gas burner. SABs were fabricated by forming a short-circuit after increasing the temperature to 900°C and holding for 2 min. After the short-circuit, the temperature was maintained for an additional 2 min. This method was referred to in the report by Weinschenk et al. [9], wherein an overcurrent fault or grounding fault occurred at approximately 900°C within 2 min on average after the flashover. Figure 1 shows a detailed experimental setup for preparing PABs and SABs. The test wires were fixed to both holders, and the left wire was slowly moved using the movement control device for the two wires to make contact with each other. The reason for adopting this method is to maintain the exposure time to the external flame and allow data to be obtained under fixed conditions. A gas burner (propane gas) was also installed at the bottom of the short-circuit point. The controller was used to adjust the size of the flame up to 2 cm in diameter and up to 10 cm in height, and its maximum temperature up to 1300°C (tolerance temperature: $\pm 50^\circ\text{C}$).

Depending on the magnitude of the short-circuit current, the amount of heat generated at the short-circuit point may affect the size and shape of the molten mark. Accordingly, a short-circuit limit device was manufactured by referring to the point contact arc test current according to the rated voltage of UL1699 [10]. This was done to obtain molten marks of similar size and shape. The short-circuit current was limited to a maximum of 500 A at a rated voltage of 220 V. A 220 V AC power source was used in the experiment with a frequency of 60 Hz. A molded case circuit breaker (MCCB) was used to protect the power system from

Figure 1. Schematic illustration of the short-circuit generator.

Figure 2. Ambient and copper conductor temperature measurement configuration.

forming a short-circuit. The MCCB, with a current rating of 30 A and an interrupting rating of 2.5 kA, was of the thermal and magnetic (TM) type. It was operated within 2 cycles with an overcurrent of 450 A or more.

The heat transfer condition was confirmed using K-type thermocouples by measuring the ambient temperature and the temperature of the copper conductor. The conductors were bare copper wires with a diameter of 1.6 mm, and K-type thermocouples (temperature range: -230 to 1250°C , limits of error: $\pm 2.2^{\circ}\text{C}$) were welded to a copper conductor to measure the temperature, as shown in Fig. 2. Three thermocouples were attached at the short-circuit point (point A) and copper conductor (points B and C). Positions A, B, and C were at 30-mm intervals. Temperature profiles were monitored for 3 min from flame initiation when the ambient temperature (point T in Fig. 2) was set to 900°C , which was the same condition used for SAB generation. The ambient temperature was measured simultaneously at a position 2-mm away from the short-circuit point (point A) in the horizontal direction.

Specimen preparation for EBSD is critical because diffracted electrons appear above 10–50 nm on the surface. The presence of contaminants, oxidation, and mechanical damage on the surface hinders the incidence of electrons onto the specimen. The flatness of the sample is necessary to ensure that the diffracted patterns are not obscured in a highly tilted sample. For this purpose, the molten mark shown in Fig. 3a was hot-mounted using conductive resin and then wet-ground with SiC papers and polished with diamond suspensions and colloidal silica suspensions (Fig. 3b). The sample preparation of copper and its alloys for EBSD analysis was introduced elsewhere [11, 12].

Field-emission scanning electron microscopes (FE-SEMs: Hitachi SU-70, Hitachi SU-6600, JEOL JSM-7900F) equipped with EBSD systems (EDAX Hikari, Oxford Instruments Symmetry) were used to measure the orientation of the specimens. The accelerating voltage of the FE-SEM was set to 15 or 20 kV, and the probe current was set to 15 nA. Mapping with a step size of $4\text{ }\mu\text{m}$ was performed.

Figure 3. a Appearance of molten mark and b cross-section of the molten mark after mounting and polishing.

The orientation varied significantly according to the direction of the specimen on the holder, especially when using EBSD to measure the orientation. The specimen refers to a molten mark secured to a flat cross-section through mounting and polishing. Accordingly, completely different orientation results can be obtained depending on how the specimen coordinate system is set, indicating that the reference coordinate of the molten mark should be clearly selected. The demarcation line between the unmelted and the melted zones in the molten mark was formed by the short circuit [2, 13]. Accordingly, the demarcation line can be set as the reference point of the specimen coordinate system. The specimen coordinate axis were set to the longitudinal direction (LD), perpendicular to the demarcation line.

Post-processing of EBSD results was performed using EDAX TSL OIM 7.3 software. The grain was set such that the minimum grain size was 2 pixels, and the tolerance angle was 5° . Grain confidence index (CI) standardization and neighbor orientation correlation were adopted, including a tolerance angle of 5° for the clean-up. The mounting resin sand pores inside the molten marks were removed during post-processing.

3. Experimental Results and Discussion

Figure 4 shows the temperature change of the ambient temperature (point T) and the copper conductor temperature (points A, B, and C) with respect to the passage of time from flame initiation. The ambient temperature was measured 2 mm away from the short-circuit point. The ambient temperature reached 900°C after 42.2 s from flame initiation and the temperature at A followed that of point T with a small difference. The average temperatures at T and A after 42.2 s were 910 and 888°C , respectively, and the difference between them was 22°C . This result was due to the heat conduction from A to C, and the temperature difference between A and C was approximately 687°C , which resulted in the release of a significant amount of heat. The graph in Fig. 4 shows that the temperature at the high temperature region of T and A fluctuates significantly, while it is rela-

Figure 4. Temperature profiles of ambient temperature and temperature of copper conductor at points A, B, and C.

tively stable at points B and C. However, the minimum arc temperature is approximately 6000 K when a short circuit occurs [14]; the temperature difference between T and A can therefore be considered to be negligible. Therefore, it is determined that the measured ambient temperature at T can be replaced with the temperature of the copper conductor at the short-circuit point A.

Figure 5. The appearance of globules and beads: a globules, b PABs, and c SABs.

Figure 6. The orientation map of globules and beads: a globules and color key (upper side), b PABs, and c SABs. Demarcation lines are indicated by white dotted lines.

Figure 5 shows the appearances of the globules and arc beads generated by the experiment. The globules exhibited an unclear demarcation line between the unmelted and the melted zone, and gradual necking (Fig. 5a). In contrast, the arc beads demonstrated a clear demarcation line and re-solidification waves and drawing lines appeared on the conductor surfaces as indicated in Fig. 5b, c. The globules and beads produced during the experiment show no significant difference in appearance from the molten marks generated in an actual fire [13, 15].

LD orientation maps measured by EBSD are shown in Fig. 6. The color in the orientation maps indicates the orientation parallel to the LD, which is a perpendicular demarcation line according to the color key (Fig. 6a). The microstructure of the globules comprised globular or dendritic grains and a clear demarcation line was not observed in the gradual neck. Contrastingly, the arc beads in Fig. 6b, c exhibit outstanding demarcation lines depicted using white dotted lines. Similarly, the demarcation lines are shown from an external appearance in Fig. 5b, c. Therefore, globules could be distinguished clearly from the appearance and microstructure observed by the optical microscopes and EBSD analysis [2, 16, 17]. Globules were prepared by exposure to higher temperatures than arc beads and consequently, grain growth was instigated in a neck. Figure 6 shows that the average grain size in the globule's neck was larger than that of arc beads. Since drawn and annealed copper wires have strong (111) fiber textures along the LD [18, 19], weak (111) fiber texture near globules' neck remained even after high temperature exposure.

Roby et al. [20] reported that the demarcation line became unclear when melted and solidified again by an external flame after forming an arc bead. In addition, the arc beads can form without any direct flame impingement or convective heat transfer when insulated wire is exposed to thermal radiation. Novak et al. [21] presented that the arc beads formed in the radiant heat of 26–55 kW/m². Meanwhile the heat flux increased up to 140 kW/m² in a short-circuit by the direct flame [9]. The different heat flux can change the microstructure of molten marks. The arc beads formed by remelting or thermal radiation were not considered because the deduction of distinguishable factors of molten marks, categorized as globules, PABs, and SABs, is the main purpose of this study.

Figures 7 and 8 show the LD orientation maps, the crystal direction maps, and pole figures (PFs) for respective representative three specimens in PABs and SABs. In order to exclude the influence of wire texture in the necks, the pixels outside of the molten marks were removed for EBSD analysis in PABs and SABs. From here, EBSD data of only molten marks was considered. The crystal direction maps indicated the (001) component parallel to the LD with a tolerance angle of 15°, with red parts belonging to (001)//LD. The (001), (011), and (111) PFs were considered for precise texture analysis. The LD in PFs is normal to figures.

As shown in Fig. 7, the thermal gradient in the PABs was the steepest in the direction perpendicular to the demarcation line because of the large temperature difference between the unmelted wire and the melted bead [22]. Therefore, the LD became the preferred growth direction of the columnar grains. In cubic material, including copper, the preferred solidification growth direction is known as the [001] direction [8]. The columnar grains with (001)//LD developed at the arc bead, as shown in the LD orientation map of Fig. 7. Although the small grains that nucleated near the demarcation line have a relatively diverse orientation distribution, a large number of columnar grains with a high growth rate occupied most of the PABs. The average area fraction occupied by the (001)//LD component for 10 specimens was 65.4%.

In this study, the reference axis of PF was established by assuming that the LD is the central axis of a cylindrical coordinate, and the arc bead was formed in an axisymmetric shape. PF is a method of displaying a crystal plane in the form of dots or contours on a stereo projection to know the crystallographic orientations based on the set specimen coordinate axis. The crystal orientation distribution of the entire arc bead can be expressed as a density contour [8]. The intensity of the density contour was calculated using a series expansion of generalized spherical harmonics. As mentioned above, it could be deduced that the (001)//LD texture developed in the PFs as well as in the LD orientation map and crystal orientation map.

It is noteworthy that the (001) fiber texture (PFs in Fig. 7a), or (rotated) cube texture (PFs in Fig. 7b, c) formed in PABs. Here, it was assumed that the arc beads are symmetrical to the shape and that the heat pass has axial symmetry in the reference axis of the LD. However, the actual PABs were not perfectly spherical and were not exactly symmetric to the LD. The reason is that the boundary conditions of external parameters such as the melt flow, temperature gradient, and heat dissipation (radiation), change the microstructure during the solidification.

Figure 7. The LD orientation maps, the crystal direction maps of $(001)//LD$ (tolerance angle = 15°) and PFs of PABs: a-c show strong texture of $(001)//LD$.

Figure 8. The LD orientation maps, the crystal direction maps of (001)//LD (tolerance angle = 15°), and PFs of SABs: a-c show the weakening of (001)//LD component.

Therefore, the development of a specific texture or the growth of columnar grains that deviate from (001)//LD needs to be clarified with respect to microstructure and texture evolution through further research using mesoscale modeling. The EBSD results confirmed that (001)//LD columnar grains were strongly developed in PABs.

In contrast, the SABs exhibited a weak (001)//LD texture and a low fraction of (001)//LD in Fig. 8. The recrystallization and grain growth in the copper wires, considering their thermal history, occurred when the temperature was maintained at 900°C, causing the arc beads to form by the short circuit. At the time of the short circuit, the formation process of arc beads was similar between the PABs and SABs. Nevertheless, the thermal gradient of SABs was more gradual than that of PABs, so the columnar grain growth was somewhat suppressed. After a short circuit, the recrystallization and grain growth occurred again in the SABs during isothermal holding at 900°C for 2 min. Therefore, SABs demonstrated various orientations and coarse grains due to recrystallization and grain growth. The average fraction of the (001)//LD component in the 10 specimens was 19.45%. This value was much smaller than that of PABs, indicating that the (001)//LD texture was weakened in SABs.

The question remains: why do some grains maintain the columnar structure parallel to the LD even after the recrystallization and grain growth in SABs? It is known that the columnar structure formed by short-circuiting cannot be completely broken down to equiaxed grains after the recrystallization without external deformation. In the solidified structure of a single-phase metal without deformation, the internal accumulated thermal strain is the main driving force of recrystallization, sometimes called stress-induced recrystallization [23, 24]. This kind of recrystallization is not accompanied by drastic microstructural change. Therefore, in the case of SABs, it can be assumed that the columnar structure was partially changed, and grain rotation occurred during the recrystallization and growth. The microstructure of the PABs comprised mainly of the columnar grains with strong (001)//LD texture, but SABs were composed primarily of a mixture of the columnar and equiaxed grains without a specific texture. Accordingly, a fraction of the

Figure 9. GAD maps of PABs.

Figure 10. GAR maps of SABs.

(001)//LD component can be selected as a factor to discriminate between PABs and SABs.

Figures 9 and 10 show the grain aspect ratio (GAR) maps of PABs and SABs, respectively. The GAR is given by the length of the minor axis divided by the length of the major axis of an ellipse fit to grain [25]. The GAR varies from approaching zero for a very elongated particle, such as a grain in a cold-worked metal and columnar structure, to near unity for an equiaxed grain. In the PABs, small grains were nucleated at the demarcation line, while the columnar grains grew parallel to the LD inside the molten marks. Therefore, the PABs consisted of mostly columnar grains with small GARs, with 10 specimens having an average GAR of 0.1327. In SABs, the grains that formed at the demarcation line were small, but the grains became larger when at a distance from the demarcation line. The columnar and equiaxed grains were mixed in SABs, and their GAR was larger than that of PABs. The average GAR of 10 specimens was 0.2832 in SABs. The small grains were visible on the surface of the molten marks in PABs and SABs, which were observed as nucleated grains in the 1- μm -thick oxide layer (Cu_2O) formed at high temperatures on the surface [26]. As described earlier, the columnar grains grown in the (001) direction preferentially occupied most of the PABs due to a steep thermal gradient. However, in SABs, the columnar and equiaxed grains were mixed because the (001)//LD texture was weakened by recrystallization and grain growth. For this reason, the grain shape of PABs and SABs were different. Therefore, since the differences between the microstructures of PABs and SABs were proved metallographically, it is possible to select GAR as a quantitative factor to discriminate between the PABs and SABs.

Based on the experimental results, the GAR and fraction of (001)//LD were selected as factors to quantitatively discriminate between PABs and SABs, as shown in Fig. 11. The discriminant line (dotted line) was shown on the graph using linear discriminant analysis (LDA) [27, 28] and the classification for the PABs (bottom right) and SABs (top left) can be applicable based on the GAR and fraction of (001)//LD. Nevertheless, one should note that this discriminant

Figure 11. PAB and SAB distribution according to the fraction of (001)//LD and GAR.

methodology is limited to molten marks produced at 25 and 900°C using a bare wire diameter of 1.6 mm. Further studies should be conducted to analyze molten marks formed at different temperatures, wire diameters, and insulators. It is expected that more data under various conditions can provide precise discriminant guidelines. Notably, this study shows the possibility of quantitative discrimination of molten marks based on the microstructure characteristics and orientation distribution using EBSD analysis.

4. Conclusion

In this study, we used EBSD analysis to quantitatively identify copper molten marks. The crystal orientation information of the globules and arc beads (PABs, SABs) was obtained and the characteristics of the microstructure were analyzed to deduce the discriminant parameters. The results can be summarized as follows:

- The globules exhibited gradual necking in appearance and indistinct demarcation lines in the EBSD results. However, the arc beads demonstrated apparent demarcation lines which were caused by rapid microstructural change.
- The microstructure of PABs had most of their columnar grains parallel to the LD (perpendicular to the demarcation line). In the orientation distribution, the (001)//LD texture was highly developed while the GAR was small because columnar grains primarily appeared.
- The microstructures of SABs consisted of a mixture of columnar and equiaxed grains due to recrystallization and grain growth after the short-circuit. The (001)//LD texture was weak, and the GAR was larger than the values for PABs.
- Based on the experimental results, the discriminant function was presented using the fraction of (001)//LD and the GAR. The discriminant factors were

derived from the distribution of the GAR and the fraction of (001)//LD in PABs and SABs.

In conclusion, the possibility of quantitative discrimination of molten marks was confirmed by analyzing the microstructure and orientation distribution using EBSD. Since this study was limited to bare copper wire (diameter 1.6 mm), it cannot be applied to all molten mark analysis. However, we hope that this study provided an opportunity to lay the foundation for further quantitative analysis of molten marks. In the future, we plan to expand the range of molten marks that can be analyzed by conducting experiments under various environmental conditions.

5. Availability of Data and Material

The data related to this work can be obtained from the corresponding author upon reasonable request.

Acknowledgments

This work was supported by the Fundamental Research Program grant funded by the Korea Electrical Safety Corporation (KESCO) (No. 2019-0101). J.-H. Kang was supported by the Fundamental Research Program of the Korea Institute of Materials Science (KIMS) (Grant No. PNK7070).

Author Contributions

All authors contributed to the study conception and design. Material preparation, data collection, and analysis were performed by JP, J-HK, EPL, YHK, and SBB. The manuscript was written and revised by JP and J-HK, and all authors commented and advised on the manuscript. All authors read and approved the final manuscript.

Funding

Korea Electrical Safety Corporation (KESCO) (No. 2019-0101), Korea Institute of Materials Science (KIMS) (Grant No. PNK7070).

Code Availability

Not applicable.

Compliance with Ethical Standards

Conflict of interest The authors declare neither a conflict of interest nor competing interests.

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