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Microscopic Investigation of a Copper Molten Mark by Optical Microscopy (OM) and Atomic Force Microscopy (AFM)

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Abstract

A wide variety of physical and chemical detecting methods have been proposed for discriminating between and electric arc bead that caused a fire, versus one that was caused by the fire itself. The simplest proposed method claims that examination of the molten marks in a bead under a microscope will suffice to make the distinction. Generally, copper molten marks of the bead are examined by using optical (OM) and scanning electron microscopy (SEM). In this paper, OM and AFM were employed to investigate a molten mark formed in laboratory. AFM observation reveals that AFM could be an auxiliary method to investigate the copper molten mark formed in the fire in order to confirm the reasons of the fire.

Keywords: AFM; copper molten mark ; fire investigation ; forensic science

1. Introduction

In recent years, the validity and authority of the identification of the fire causes especially knotty and complicated fire disaster [1,2] have been paid more and more attention. The proportion of electrical fire is highest in all fire disasters in China according to the statistic related data of China fire services from 2003 to 2010 [3]. An electrical fire is any fire that caused by electrical short circuits, overloaded circuits or faulty electrical equipment. Anything that causes excessive current flow has the ability to create fire, including short circuits. In most cases, where an electric fire happened, electric molten marks in the electric arc bead that may be the cause of the fire could be found in the fire scene. Various methods have been studied for identifying these electrical molten marks to be either the cause of a fire or one caused by the flames of the fire including analyzing their appearance [4], the voids in the molten marks [5], carbonized residue [6], surface analysis [7, 8], and so on. The simplest method, which

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distinguished the microstructure formed in different condition, was analyzing the microstructure of the molten marks under a microscope (after preliminary preparation which may including cleaning or etching surface, and other techniques for preparing cross-section). Generally, microstructures of molten marks are examined by using optical (OM) [9] and scanning electron microscopy (SEM) [10]. While OM may not provide the necessary resolution sometimes, the microstructures of molten marks may present three-dimensional features, as a result of etching, which is not always possible to be observed by SEM.

The atomic force microscopy (AFM), capable of generating 3D images, is now a well-established technique for imaging surface topography with high resolution and can be used to study the microstructure of materials in a nanometer range. This powerful technology allows material scientists new insight into the diversity of microstructures and defects. As an alternative, this paper describes the use of atomic force microscopy (AFM) in understanding micro-scale feature of a copper residue formed in typical condition in laboratory.

2. Experiments

After an electrical fire, all kinds of broken copper wires can be found in the debris. At the point of severance, the ends could show numerous forms such as beads, fragile fracture, plastic deformation, pointed ends, and numerous other characteristics. The copper molten marks could be due to a very high current, the heat of the fire, or a combination of these, as well as mechanical fractures, either at fire temperature or nearer to room temperature, with or without current. Electrically initiated fires can be caused by old as well as new wiring in different potentially hazardous conditions, such as loose wires on outlets, overloaded extension cords, broken wires with intermittent connection, deteriorated wiring, damaged and abused cord sets, corroded plugs and sockets, damaged insulation, etc.

In this study, in order to form a typical copper molten mark, new flexible PVC-coated copper cord (single-strand, allowable current: 20 A at 25 ºC, diameter is 1 mm2 [4]) was used. An experimental station to form typical short-circuit copper molten marks was constructed. Figure 1 is the schematic illustration of the experimental circuit. A single electrical circuit, composed of power source (AC 220 V), a switch, sample copper wire, electrical applications and current probe, was wired in the experimental station. The electrical molten marks that were formed between two PVC-wounded copper cords in a container, when brought in contact (twisted) with each other. The wrapping insulated polyvinylchloride of the wires was burnt out by the petrolic burner flames, and then the copper wires in the two cords were contact with each other, short circuit happened.

An electric arc between two current-carrying copper wires produced some extremely high temperatures. If an arc occurred and suitably conditions were present, a fire may ensue. Since the temperature of an electric arc was greatly in excess of the melting temperature of copper (1085 ºC), portions of the copper wires may melt in such an event. In the air cooling condition, the copper molten mark formed.

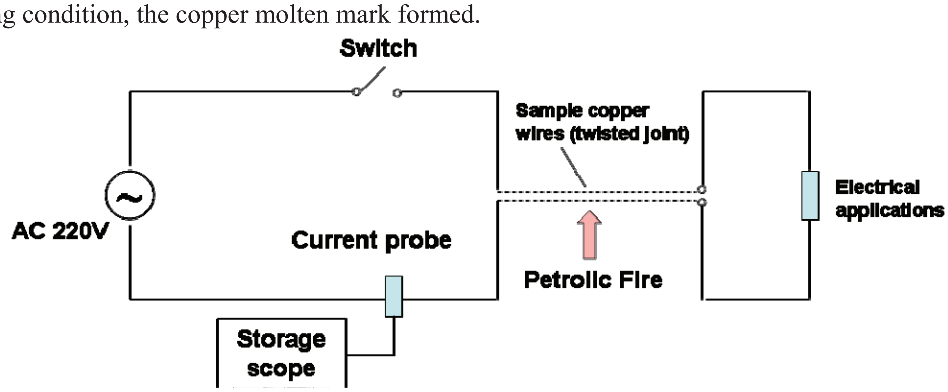


Fig. 1 Experimental circuit

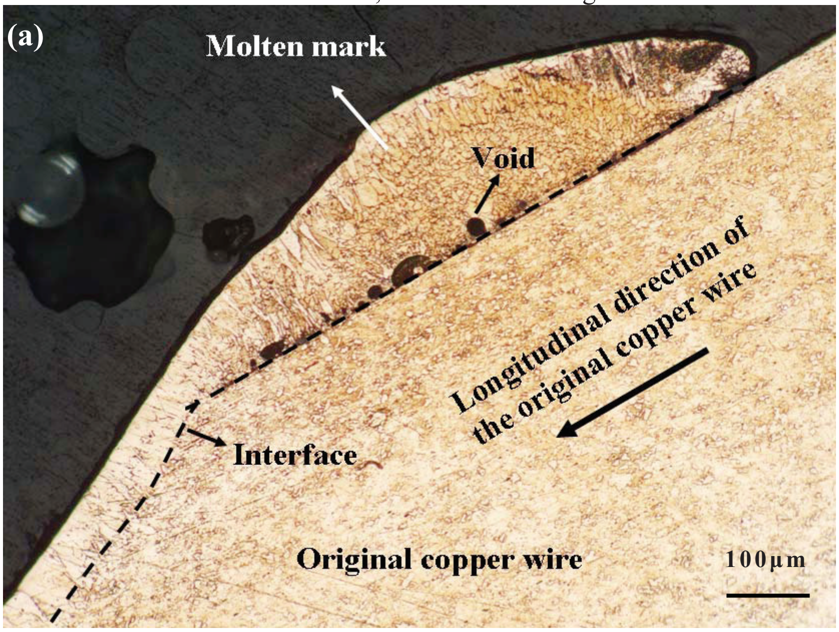
The microstructure of the molten mark was evaluated by applying ordinary procedures of metallographic preparation. Electrical copper molten sample was prepared by grinding, mechanical polishing and chemical etching with solution of 25 ml of alcohol, 12.5 ml of HCl and 0.25 g of $FeCl_3$. The sample was investigated by optical microscopy (OM) and atomic force microscopy (AFM). The OM investigation was carried out in OLYMPUS

PMG3 equipment, while in the AFM, images were acquired using a TopoMetrix Discoverer instrument, in the contact mode operation.

3. Results and Discussion

3.1. Optical observation

Micrographs of the longitude-section microstructure in the mark domain were shown in Fig. 2. Fig. 2 (a) presented solidified copper molten mark and part of original copper wire obtained by OM for 200×. It was apparent that the morphology of the grains in the molten mark was quite different from the original copper wire. The microstructure of the sample showed that, as an initial counterpart, columnar and isometric grains were distributed in the molten mark which was attached to the original copper wire. Many voids were existed in the interface. Fig. 2 (b) showed the magnified image of 500 ×. It could be found that microstructure at the edge of the molten mark, showed larger columnar grains which growth direction was directed to the core of the copper wire, however, microstructure in the other domain in the molten mark, showed isometric grains.



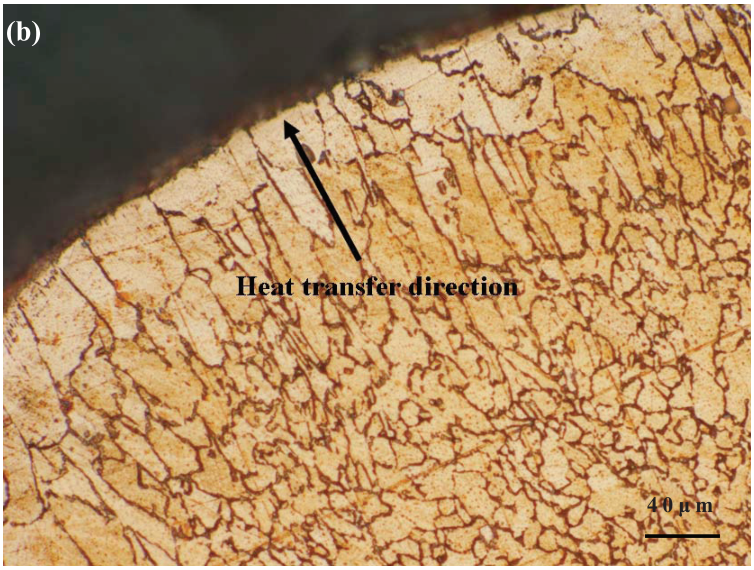


Fig. 2 Typical microstructures of copper molten mark under optical microscopy with etching, (a) 200×, (b) 500×.

It was traditionally suggested that the microstructure of the molten mark had a relationship with the solidified condition (thermal history). The microstructure of the original copper wire, not heated to beyond melted point, showed fine longitudinal striations caused by the wire-drawing process. In the molten mark, solidification of the molten copper which was formed by the electric arc during short circuit happened in the tip of the copper wire and then flowed to the side of the wire. It was also suggested that the thermal exchange of the melt with its interface of the air is mostly dominated by the convection. It was because of the combination of rather large temperature gradient and proper growth rate at the outer edge in the molten copper in the present experiment, the solidified structures outside the molten mark, in which molten copper solidified firstly, treated were predominantly columnar grains. The columnar grains grew in a direction which was antiparallel to that of the heat flow. Then, because of the same of the growth and heat flow direction, which would induce equiaxed growth, the isometric grains having random orientations were nucleated in the remaining, slightly-undercooled liquid.

3.2. AFM observation

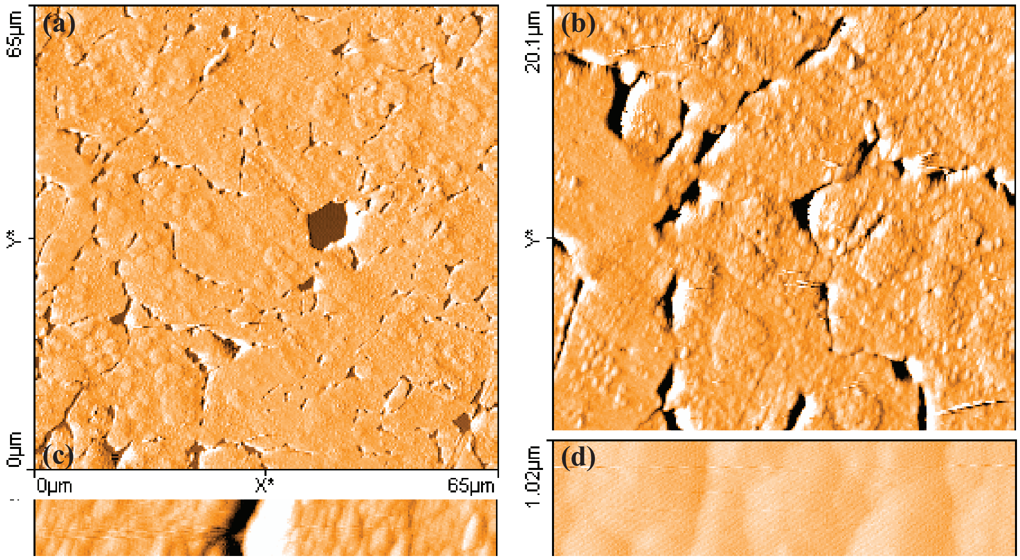


Fig. 3 Typical microstructures of copper molten mark under atomic force microscopy (AFM) with etching, (a) 65×65 µm, (b) 20.1×20.1 µm, (c) 5.01×5.01 µm, 1.02×1.02 µm.

Structural detail of copper molten mark covers a lot of information which could illustrate the temperature and chemical circumstance in a fire disaster. Therefore, different microscopic techniques like optical microscopy (OM), scanning electron microscopy (SEM) are used for visualizing these microstructures. Usually, examination of a metallic structure is carried out by using optical microscopy. AFM is a very useful supplement to this technique with scan sizes ranging from about 100 um down to several nanometers. A force sensor, consisting of a Si3N4-contilever with a tip radius of about 30 nm, gave lateral and vertical resolution of 2 nm. Each image was taken in nearly 10 minutes and consists of 512×512 points. Image processing and quantitative evaluation of particle size distributions was done with a software system.

This large dynamic range was illustrated in Fig. 3 with four pictures, where the microstructures of the copper molten mark were visualized clearly with the AFM. The microstructures consisted of three parts: grains, surrounded by grain boundaries, grain boundaries and voids. By monitoring the copper molten mark in a series of AFM images, including 65 μm, 20.1 μm, 5.01 μm and 1.02 μm scans, the details of the grains, grain boundaries and voids could be seen. It could also be seen that there were deeper worn traces on the rubbing surface of copper wire molten mark (as shown in Fig.3 (a)), the wear scar is similar to "ploughing" and there were some adhesion traces (grain boundary) of grains (black places shown in Fig.3 (a)). The grain boundaries were easily identified in all of the images since the height differences on the boundaries were much larger than the average surface roughness. In Fig. 3 (b), it could be seen that there were many independent particles in the grains. In the magnified micrographs (Fig. 3 (c) and (d)), the small sesame-like particles of about several hundred nanometer dispersed uniformly in the grains could be seen clearly.

Though this was just a rough estimation, in the current observation, the results still showed that the AFM results, in particular the high resolution plots, provided several details of the copper molten mark.

4. Conclusions

Reliable, localized measurements of microstructure of a copper molten mark are of great relevance for identifying the cause of fire, and for investigating the conditions of fire process. Both optical microscopy (the common used method) and atomic force microscopy were employed to investigate a typical copper molten mark formed in laboratory. The results obtained allow one to deduce that AFM is a simple and auxiliary method to investigate the copper molten mark formed in the fire. AFM could provide unique capabilities relative to other microscopies for its superior resolution.

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