Preparation Method for Metallographic Specimen of Iron-Carbon and Silicon-aluminium Alloy

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Abstracts: Metallographic analysis is one of the means of testing and analyzing materials. To conduct metallographic analysis, we must prepare samples that can be used for microscopic observation. It is important to select and prepare representative samples, which is directly related to the correctness of microstructure analysis of metallography specimens. In this paper, the preparation process of the ferrocarbon alloy metallographic sample was analyzed and studied in detail, and some common defects were pointed out. The preparation process includes sampling, inlaying, polishing, polishing, and corrosion. Then, taking a silicon aluminum alloy (YZAlSi10) as an example, the preparation process is described, and the matters needing attention in the preparation process and the elimination of metallographic defects are analyzed. Finally, the accurate and clear microstructure of the metallographic structure is restored.

1. Instruction

The metallographic technique can be used to reveal the types, properties, quantity, size, shape, distribution, and combination of each phase and tissue defect in microstructure, which can be used for quantitative characterization. The microstructure observation of iron-carbon alloy is the basis for the study and analysis of the properties of steel materials. In metallographic observation, the preparation of a metallographic specimen is an important link in obtaining clear organization and drawing correct conclusions \cite{1}. Stefanescu D M prepared the cast samples of the alloy composition Al-4% Cu to study the effect of the surface refining process (SRP) on hardness, wear rate, and coefficient of friction of Al-4Cu \cite{2}. Kovacheva R measured the graphite shape factors on the metallographic samples of iron-carbon-silicon alloys, and analyzed their evolution as a function of the chemical composition and the solid fraction \cite{3}. Qing-Hong H E described results obtained with a new method for electropolishing metallographic specimens of zinc and zinc alloys. Conventional metallographic preparation of the specimens was followed and is described elsewhere in \cite{4}. Lee E S. described some issues that should pay attention to in the preparation of ZL101A metallographic specimen using the manual method. A method of electrolytic polishing was studied in \cite{5}. The several steps of preparation of metallographic specimen consists are sampling, inlaying, grinding, polishing, and etching.

1.1 Sample Selection and Inlaying

The common sampling defects are such as lack of representativeness, creation of the metallographic illusion, and unrecording of the sampling location and method \cite{6}. For hard materials, you can use a grinding wheel slicer or wire cutting. For hard and brittle materials, a hammer can be used. We should pay attention to whether the organization of the sample is changed due to improper operation. And if the material size is too small or irregular in shape or needs to protect the edge of the sample, we can adapt...
the method of mosaic. The metallographic specimen after mosaic is shown in Fig.1.

1.2 Sample grinding and polishing

1.2.1 Grinding

The grinding of the metallographic specimen is divided into coarse grinding and fine grinding. Rough grinding is usually carried out on a grinder. During the grinding process, the sample must move slowly along the radial direction of the grinding wheel, to prevent the surface of the grinding wheel from forming grooves. During the grinding process, the sample needs to be continuously immersed in water cooling, to prevent the change of the tissue. In addition, gloves are not allowed in rough grinding, to avoid hands being sucked into the grinder. Fine grinding is divided into two types: manual grinding and mechanical grinding. The operation process of manual grinding is shown in Fig.2. Lay the sandpaper on the glass panel. Hold the glass plate with one hand, and with the other hand holding the sample do a one-way grind in the sandpaper.

Manual grinding needs to replace different types of metallographic sandpaper. Metallographic sandpaper can be divided into many types from coarse to fine. The specification can be seen in Table 1.

| Granularity | 400# | 500# | 600# | 800# | 1000# | 1200# |
|-------------|------|------|------|------|-------|-------|
| Model       | M28  | M20  | M14  | M10  | M7    | M5    |
| Grit size/μm| 28~20| 20~14| 14~10| 10~7 | 7~5   | 5~3.5 |

The following points need to be noted when handing grinding. When choosing sandpaper, the size of the sand grain is small to large. Force the sample evenly as grinding on the same sandpaper. The grinding crack length should not be too large. When the sand grains on the sandpaper become dull, it is not suitable to continue to be used, otherwise, the rolling pressure generated by the sand particles on the metal surface will increase the surface deformation. Then grind the sample rotated in 90°in the polishing of the sample is to eliminate the fine grinding marks left by fine grinding to make it smooth and glossy. Clean the sample and the glass before replacing the sandpaper, in case the coarse grains are brought into the fine sandpaper. To avoid the effect of the heat of the metal tissue on the organization's authenticity.

1.2.2 Polishing

The polishing is to eliminate the fine grinding marks left by fine grinding, and make them bright, not marking flat mirror surfaces. The methods include mechanical polishing, electrolytic polishing, and chemical polishing [7]. Among them, mechanical polishing is the most commonly used stage.

When polishing, the sample is perpendicular to the radial direction and reciprocates along the radial direction of the disk. Add an appropriate amount of polishing fluid or water to maintain the humidity of the polishing disc and avoid dry polishing. Polishing time is usually 3-5min. A polishing cloth is selected according to the hardness of the material. At the end of polishing, the polished surface of the specimen shows a mirror appearance. From sampling, rough grinding, fine grading to polishing, the surface of the specimen is shown in Fig.3.
1.3 Etching
The polished specimen appears as a shiny surface under the microscope. Scratches, watermarks, inclusions, cracks, etc. can be seen on the surface, as shown in FIG. 4. After the sample is chemically corroded, the chemical structure of the sample can be precipitated to analyze the metallographic structure [8].

![Fig.3 preparation of low carbon steel sample](image)

The depth of etching should be determined according to the characteristics of the tissue and the magnification of the microscope. The time of etching is different for different materials and different treatment conditions. The problems that are easy to appear in the erosion are the rusty spots if the cleaning is not in time, the phenomenon of inclusions tail if the cleaning is not in place, the spots of the alcohol wiping are not in place, as shown in Fig.5. The phenomenon of shallow or excessive erosion is shown in Fig.6.

![Fig.4 the surface of the specimen after polishing (containing metal inclusions)](image)

![Fig.5 Comparison of dry samples when alcohol is not volatile in time](image)

![Fig.6 eroded deep (20 steel)](image)

2. Preparation of metallographic samples of silicon and aluminum alloy
Aluminum alloy has a wide range of use in electrical engineering, space and space industry, general mechanical engineering, and light industry [9]. When the aluminum alloy is melted, it is easy to produce Al₂O₃. It not only deteriorates the casting properties of the alloy but also reduces the impact toughness and fatigue limit of the alloy [10-13]. The microstructure of Si Al alloy can be understood by a metallographic microscope, the casting effect of Si Al alloy can be understood, and the defects of the microstructure can be revealed. Taking YZAISi10 as an example, metallographic specimens were prepared and analyzed.

2.1 Sample grinding
For the good samples of aluminum alloy, it needs to chamfer the sample, 40# or 80# sand paper can be used for chamfering. 45°chamfering is more appropriate as the soft material. In manual grinding, sandpaper is selected from low to high. Compared with the grinding of iron-carbon alloy specimens, the pressing force is lighter. The sandpaper can be used from 400# to 1200#. As the aluminum alloy is soft, the inclusion will fall off during the grinding process, resulting in scratching the surface of the sample. Kerosene or water can be dripped on sandpaper to prevent the falling sand from being embedded in the metal surface, resulting in a false appearance. There are inevitably some shrinkage holes in the casting process of aluminum alloys. When grinding, there will be some holes on the surface of the sample. As shown in Figure 7-1.
Fig. 7-1 After polishing (macro)  Fig. 7-2 After erosion (500×)

Fig. 7 macro and micro comparison of specimens before and after etching

2.2 Sample polishing

Rinse the ground samples with water and polish them on the polishing machine. The speed of the polishing machine should not be too high and the speed is 600r/min-1200r/min. The polish is used as a polish for polishing cloth. The polishing paste can be evenly spread on the disc, and the light pressure sample moves along the polishing disk in the radial direction and moves evenly; It is necessary to control the humidity of the polishing. When the polished surface is not worn and bright as a mirror, the polishing is finished.

2.3 Sample etching

As different aluminum alloys needed different etching treatments [12]. As described earlier, the preparation process of the Si Al alloy specimen is shown in Table 2. Note that HF aqueous solution is highly corrosive and safety should be paid attention to in operation.

Table 2 Metallographic preparation recipe for YZAlSi10 alloy

| Steps       | Coarse grinding | Fine grinding | Polishing | Etching            |
|-------------|-----------------|---------------|-----------|--------------------|
| materials   | SiC sandpaper   | SiC sandpaper | navy cloth| tampon, Wood clips |
| Granularity | 400, 600, 800   | 1000, 1200    | 2.5 μm Grinding paste, water | Absolute alcohol /0.5%HF aqueous solution |
| Lubricants / | water           | water         |           |                    |
| eroding     |                 |               |           |                    |
| agents      |                 |               |           |                    |
| rotation rate|                 |               | 1500 r/min|                    |
| strength    | Soft reduction  | Soft reduction| Soft reduction| Soft reduction |
| Length of   | 10 min          | 10 min        | 3-5 min   | 15-20 s            |
| time        |                 |               |           |                    |

Fig. 7-2 is the microstructure of the YZAlSi10 specimen after etching. It can be seen from the picture that the eutectic of the aluminum alloy can be displayed. There are a small number of scratches in the microstructure, a straight black line, which can pass through several grains. It can also be seen that there are many small, large, and small black regions in the microstructure, which is the hole produced by the casting, which is presented after the sample is prepared.

A good specimen of Si Al alloy can be obtained clearly from the microstructure diagram. As shown in Fig. 8.

Fig. 8 microstructure diagram of silicon aluminum alloy (500×)
3. Conclusion
The microscopic observation of the metallographic sample is directly related to the test and judgment of the properties of the sample. The microstructure of different materials in different treatment states is different. When making metallographic specimens, we need to continuously inquire about relevant information and analyze it repeatedly with existing materials. Especially for the problems arising in the preparation process, we need to use a rigorous scientific attitude to analyze the causes, and the process of problems, and analyze and improve them. Making full use of advanced metallographic analysis instruments and technologies, the method of metallographic specimen preparation is perfect. The preparation of metallographic specimens can provide powerful help for metallographic microscopic analysis.

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Reference

[1] Gopi V, Sellamuthu R, Arul S. (2014) Measurement of Hardness, Wear Rate and Coefficient of Friction of Surface Refined Al-Cu Alloy [J]. Procedia Engineering, 97:1355-1360.
[2] Stefanescu D M, Alonso G, Larrañaga P, et al. (2016) On the stable eutectic solidification of iron–carbon–silicon alloys[J]. Acta Materialia, 103:103-114.
[3] Kovacheva R, Gidikova N, Lilova A. (1992) A new electropolishing technique for metallographic specimen preparation of zinc and zinc alloys[J]. Materials Characterization, 28(4):205-211.
[4] Qing-Hong H E. (2014) Issues in the Preparation of ZL101A Metallographic Specimen[J]. Foundry Equipment & Technology.
[5] Lee E S. (2000) Machining Characteristics of the Electropolishing of Stainless Steel (STS316L)[J]. International Journal of Advanced Manufacturing Technology, 16(8):591-599.
[6] Wang L, Yang P, Li C R. (2006) Metallographic experimental technology [M]. Beijing: Metallurgical Industry Press, 73-85
[7] Zipperian D C, Diaz D. (2000) Metallographic specimen preparation[J]. Advanced Materials & Processes, 158(4):52-54.
[8] Saklakoğlu I E. (2009) Surface morphology and tribological behavior of AlSi10 alloys treated by plasma immersion ion implantation for automotive applications[J]. Journal of Materials Processing Technology, 209(4):1796-1802.
[9] Uli N, Kuppuswamy R, Amran M F. (2011) A survey of some metallographic etching reagents for restoration of obliterated engraved marks on aluminium-silicon alloy surfaces[J]. Forensic Science International, 208(1-3):66.
[10] Boncina T, Zupanic F. (2016) Dynamic Deep Etching and Particle Extraction for High-Strength Aluminum Alloys[J].
[11] Panchenko I, Mueller M, Wolter K J. (2010) Metallographic preparation of the SnAgCu solders for optical microscopy and EBSD Investigations[C]// International Spring Seminar on Electronics Technology, Iss. IEEE,18-23.
[12] Collini L, Nicoletto G, Konečná R. (2008) Microstructure and mechanical properties of pearlitic gray cast iron[J]. Materials Science and Engineering: A, 488(1-2): 529-539.
[13] Akimov I V, Sylloveryuk V P, Volchok I P, et al. (2013) Influence of the shape of graphite inclusions on the mechanical properties of iron–carbon alloys[J]. Materials Science, 48(5): 620-627.