Color Metallography of A356 Aluminum Alloy Castings using Weck's Reagent

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A color etching method using Weck's reagent (3.81mol%KMnO4, 0.15mol%NaOH water solution) was developed for both the optical microscopy and micro-segregation characterization of A356 aluminum alloy (Al-7wt%Si-0.35wt%Mg) castings. The coloring mechanism of Weck's reagent was also elucidated. In this study, colored microstructures of A356 alloy (with 0.1wt% Ti added) were compared with the solute distributions measured by EPMA analyses to make clear the relationship between color and segregation. It was also evidenced that the tracking the distribution of Ti in Al phase during the spheroidization of Al phase at semi-solid state made possible. These examples prove that color etching with Weck's reagent is an efficient way of characterizing micro-segregations in Al alloy castings. The accurate evaluation of solid fraction of A356 alloy slurry using Weck's reagent was also achieved. With the help of Weck's reagent, the grain growth happened during rapid cooling of the alloy from semi-solid state to room temperature was clearly visualized. Thus the overestimation of solid fraction by conventional method was avoided by excluding the area of grain growth. Additionally, the spheroidization process of dendritic Al phase was observed in detail using Weck's reagent and EBSD analysis and the spheroidization mechanism was provided. Furthermore, the etched specimen surface was characterized by various observation and analysis methods to study the coloring mechanism of Weck's reagent.

Keywords: Color etching, A356 alloys, semi-solid casting, micro-segregation, spheroidizing mechanism.

1. Introduction

The present study is concerning the characterization of A356 alloys processed at semi-solid state through color metallography by using Weck's reagent. Applications of Weck's reagent were summarized, and then the coloring mechanism of the etching was studied by analyzing the etched surface using various kinds of advanced analyzing techniques.

2. Correlation between the micro-segregation in Al phase and color revealed by Weck's reagent

The detailed correlation between color and micro-segregation in A356 alloys was studied. EPMA mapping mode was applied to characterize the solute distribution and compared to optical micrograph after etching with Weck's reagent. The results show that micro-segregation of Ti, Si and Mg in Al phase can all lead to a color difference (Fig. 1(a-d)). Besides in dendritic Al structure, micro-segregation also exists in spherical Al grains obtained by semi-solid process (Fig. 1(e-f)). The spherical grain growth occurred when cooling it from semi-solid state to room temperature can also be visualized by Weck's reagent. [1]

3. Accurate solid fraction evaluation of A356 alloy at semi-solid state by Weck's reagent

As shown above, grain growth during quenching from semi-solid state could be visuallized by Weck's reagent. This finding can help to evaluate solid fraction at semi-solid state with a better accuracy by exclusing this part when calculating the area fraction of solid particles in the optical micrograph. The result for A356 alloy using this method were compared with both the traditional method without excluding the grain growth during queching and the results from lever rule referring to Al-Si binary phase diagram. It is shown that the results obtained with exclusion of the grain growth during water quenching agree well with the results calculated from Al-Si binary phase diagram. The results obtained without consideration of the grain growth showed an overestimation over the above two methods. [2, 3]

4. Spheroidization mechanism of Al phase at semi-solid state by compression and partial re-melting process

The microstructural evolution of Al grains from dendritic to spheroidal was focused. Since the materials were deformed and heated to semi-solid

state, recrystallization occurred during heating. Al grains were refined compared to the specimen without deformation before heating. Weck's reagent finds its application again in this study. By etching the specimen which is transforming from dendritic to spheroidal, compressed dendritic Al grains are found separated and when the eutectic structure starts to melt, liquid phase penetrates into those separation sites and spheroidized Al grains are formed (Fig. 2). According to EBSD analysis, high angle grain boundaries are found inside Al phase when the compressed specimen is heated. This suggests that Weck's reagent is also able to reveal grain boundaries in Al phase clearly. [3]

5. Coloring mechanism of Weck's reagent studied by characterization of the etched surface

Coloring mechanism of Weck's reagent was studied by various characterizations and analyses of the etched surface. Surface observation by SEM and laser microscopy found that the locational change of color is related to the different surface roughness which is in the order of submicron. More detailed observation by TEM, STEM-EDS and SIMS showed that an amorphous film contains $MnO₂$ is formed on the Al surface via the conversion coating occurred during the etching with Weck's reagent. Depending on the different concentration of solute in Al (micro-segregation), the $MnO₂$ thin film with different thickness and roughness is formed on the specimen's surface. The higher concentration of Ti can promote the film growth, which is confirmed by the surface observation. It is concluded that the color of the film is basically brown. But the brightness of brown is strongly influenced by the film's thickness and the roughness of the interface between the film and substrate. As a result, reflection of light from both the film surface and interface between film and substrate (Al alloy) interfered, causing the different colors observed by optical microscopy (Fig. 3). [4]

References

[1] Li. Gao, Y. Harada and S. Kumai: Materials Science Forum, 794-796, pp. 9-14, 2014. [2] Li. Gao, Y. Harada and S. Kumai: Journal of Materials Science, 49(3), pp. 1286-1296, 2014. [3] Li. Gao, Y. Harada and S. Kumai: Materials Characterization, 107, pp. 426-433, 2015. [4] Li. Gao, Y. Harada and S. Kumai: Materials

Characterization, 107, pp. 434-452, 2015.

Fig. 1 Optical micrograph and EPMA mapping for solute distribution.

Fig. 2 Optical micrographs of Al grains showing spheroidizing process.

Fig. 3 View of the cross section of specimen with correlation to change of Ti concentration.