Microstructure Analysis of Al-5%Mg Alloy Semi-Solid Slurry by Weck's Reagent

Kamolwat Prapasajchavet, Yohei Harada and Shinji Kumai

Department of Metallurgy and Ceramics Science, Tokyo Institute of Technology, Tokyo 152-8552, Japan

Spheroidization of Al grains is required for the production of semi-solid slurry. In this research, Al-5%Mg aluminum alloy with dendrite structure was plastically deformed with 33% compression and partially melted in a semi-solid state in order to obtain globular solid grains. The microstructure evolution during partial melting was investigated by the quenched microstructure etched with Weck's reagent. EPMA analyses by X-ray Wavelength Dispersive Spectroscopy (WDS) were carried out to evaluate Mg content in globular solid grain. Imaging with secondary electrons by SEM was applied to characterize the etched surface. In the as-etched optical micrograph, the microstructure was revealed with different color and contrast. During microstructure evolution of compressed specimen, the deformed grain changed gradually to the spheroidal shape due to the partial melting at the semi-solid temperature range. The original shape of the globular Al grains at the semi-solid state could be visualized clearly in the quenched microstructure by eliminating the growth layer of Al grains formed during water quenching. The solid fractions were measured for the entire semi-solid temperature range. SEM images showed that the surface topology of the specimen is corresponding to the variation in color and contrast in the optical micrograph.

Keywords: Aluminum alloys, Semi-solid process, Solid fraction, Color etching, Weck's reagent

1. Introduction

Al-Mg alloy has the high strength and good corrosion resistance, however, this alloy shows a poor castability. By using conventional casting process, casting defects and solidification cracking often formed. The effective method to produce sound products is necessary.

Semi-solid metal process is the forming technique of the cast products from the semi-solid state at temperature between solidus and liquidus line. The semi-solid slurry consists of spheroidal solid grains suspended in the liquid phase at semi-solid temperature during forming [1]. The present process can decrease shrinkage porosity due to reduced processing temperature and improve the castability for this alloy system.

In the present research, Weck's reagent (Distilled water: 100mL; KMnO₄ 4g; NaOH 1g) is utilized to visualize the microstructure of Al-Mg alloy semi-solid slurry obtained by recrystallization and partial melting process for understanding the microstructure evolution during partial melting. The solid fractions in the semi-solid temperature range were also precisely evaluated.

2. Experiment

Al-5%Mg billet was plastically deformed at room temperature with 33% compression and partially melted to semi-solid temperature range: 580-630°C. The polished surface of as-cast, as-compressed and semi-solid specimen were etched with Weck's reagent for approximately 12s (\pm 1s) at room temperature to examine the microstructure. Microstructure was observed by optical microscope. To investigate the microstructure evolution during partial melting, the compressed specimens were heated to various temperatures from the below solidus line to the temperature range of semi-solid state. The solid fractions were measured for the entire semi-solid temperature ranging from 585°C to 625°C. Imaging with secondary electrons by SEM was applied to characterize the etched surface. EPMA analyses by X-ray Wavelength Dispersive Spectroscopy (WDS) were carried out to evaluate Mg content in solid grain.

3. Results and Discussion

The optical micrograph observation showed the as cast specimen exhibited dendritic structure. For the as compressed specimen, dendritic branches were deformed and changed gradually to the spheroidal shape due to the partial remelting at the semi-solid temperature range [2] shown in Fig.1.

The dark brown area and the white area corresponded to the globular solid grain and liquid phase at the semi-solid state, respectively. While, the light brown area indicated the solid growth layer during water quenching shown in Fig.2.

By excluding the growth layer of Al grains formed during water quenching, the original shape of the globular Al grains at the semi-solid state could be visualized clearly [3]. The entrapped liquid inside the globular solid grains was considered as the solid phase at semi-solid state in order to evaluate the precise solid fraction which comparable to the actual situation during dies filling: red solid fraction curve shown in Fig.3.

As shown in Fig.4, SEM images showed that the surface topology of the specimen is corresponding to the variation in color and contrast in the optical micrograph. The dark brown area and the light brown area in the optical micrograph corresponded to the rough surface and smooth surface in SEM image, respectively.

Optical micrograph showed a variation in color and contrast, light brown and dark brown in globular solid grain. In contrast, the Mg content obtained by EPMA analysis showed the homogenous Mg distribution in the globular solid grain. This might suggest that Weck's reagent possibly has better sensitivity in detecting the local Mg content than EPMA analysis. In order to investigate this topic, coloring mechanism and precise investigation concerning the relationship between Mg composition and color (contrast) of the color-etched optical micrograph is necessary [4].

4. Conclusion

By etching Weck's Reagent, the original solid phase and liquid phase at semi-solid state were distinguished clearly with different color and contrast. The microstructure evolution during partial melting could be investigated. The present color metallography was effective for correct estimation of the solid-liquid ratio in Al-5%Mg alloy semi-solid slurry.

Acknowledgement

The authors would like to thank Nissan Motor Co., Ltd for supplying the starting material and technical supports in this research.

References

[1] Flemings MC (1991) Metall Trans A 22A:957 [2] L. Gao, Y. Harada, S. Kumai, J. Mater. Sci. 47 (2012) 6553-6564.

[3] L. Gao, Y. Harada, S. Kumai, J. Mater. Sci. 49 (2014) 1286-1296.

[4] L. Gao, Y. Harada, S. Kumai, Materials Characterization. 107(2015) 434-452.



Fig.1 Microstructure of (a) as cast (b) as compressed and (c) semi-solid specimen



Fig.2 Detection of grain growth during quenching



Fig.3 Solid fraction curve of Al-5% Mg alloy

f_S : read from phase diagram

 f_{S} : 1- f_{L} (liquid outside and inside the grain) f_S : 1-f_L(only liquid outside the grain)





(c) Mapping distribution by EPMA

Fig.4 Relationship between color and contrast, surface topology and Mg distribution