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Microstructural characterization of aluminum alloys using Weck's reagent, part I: Applications

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ABSTRACT

This paper focuses on the applications of a color etchant for aluminum alloys named Weck's reagent. The Al phase shows different colors from location to location after being etched by Weck's reagent. It is proved that Weck's reagent is very sensitive to the micro-segregations of Ti, Si and Mg in Al alloys so that characterization of the micro-segregations can be qualitatively realized which is usually done by electronic probe techniques. With the help of this characterization method, we are able to evaluate solid fractions for the semi-solid processed Al alloy with a better accuracy by excluding the Al grain growth during water quenching. To understand this reagent better, the color change during etching is investigated by applying different etching times at room temperature (25 °C). Among those results, 12 s shows the best color contrast after etching. Finally, we repeat the 12 second etching for four times through repeating a polishing–etching process. The result exhibits that Weck's reagent has a satisfying re-producibility with stable color and color distribution for the four times etching result. The second part of this study covers the coloring mechanism of Weck's reagent by characterizing the etched surface via various characterization methods.

1. Introduction

Color etching is an effective technique to characterize the microstructures for various metals or alloys. The history of color etching is very long. Color micrographs were published as early as over ninety-some years ago [1]. Although nowadays the electron microscopy technology is playing an important role in material characterization such as composition analysis and grain structure detection, color etching, this "old technique" is still being used and studied recently. Several studies were carried out for cast iron or steels applying the color etching method [2–7]. Among those studies, some had made an effort in explaining the coloration mechanisms or the relationship between color and material properties [4,6,7]. It was found that the color difference could qualitatively represent the difference in chemical compositions [4] or grain orientations [6,7]. In the case of aluminum and its alloys, anodizing using Barker's reagent (1.8% HBF_4 in distilled water) is widely applied to reveal the grain structure. Despite the fact that in many cases, a film is formed on the specimen's surface after anodizing, no film is formed on aluminum alloys when anodized by Barker's reagent. Instead, the surface is furrowed which causes coloration effects under polarized light [1]. The color difference among grains is related to the different grain orientations. Another useful color etching method for aluminum alloys was developed by Weck and Leistner in the 1980s [8]. The reagent was called "Weck's reagent" which contains 4 g of $KMnO_4$, 1 g of NaOH and 100 mL of distilled water. This reagent can reveal micro-segregations inside aluminum grains. In the examples given by Weck's research [8], the dendritic aluminum grain formed during casting exhibits color difference between the central (early formed during solidification) and surrounding regions (later formed during solidification), which is believed to be a reflection of the micro-segregation formed during solidification of the primary Al phase. In our previous research on semi-solid processed A356 aluminum alloy [9], this reagent was used for etching not only the dendritic structure but also the spheroidal structure. The result was quite surprising. We found that the Al spheroidal grain obtained by partially re-melting the dendritic structure is a cored structure (Fig. 1). Inside the spheroidal grain, there was a dendritic structure visualized by Weck's reagent (Fig. 1b). Also, the growth of spheroidal grain during water quenching from semi-solid state was distinguished with the help of this reagent, so that the solid fraction could be more accurately evaluated by the exclusion of the growth when calculating the area fraction (detailed explanation will be given later in this paper). Achieving these interesting results, we moved our focus to the micro-segregation-sensitive nature of Weck's reagent [10]. By comparing the color difference with the result of electron probe micro-analysis (EPMA), it is confirmed that the micro-segregation of Ti in Al grains had a strong correlation with the color difference revealed by Weck's reagent (Fig. 1c). Ti was added to A356 Al alloy to refine the Al grain size. Due to the extremely low diffusion

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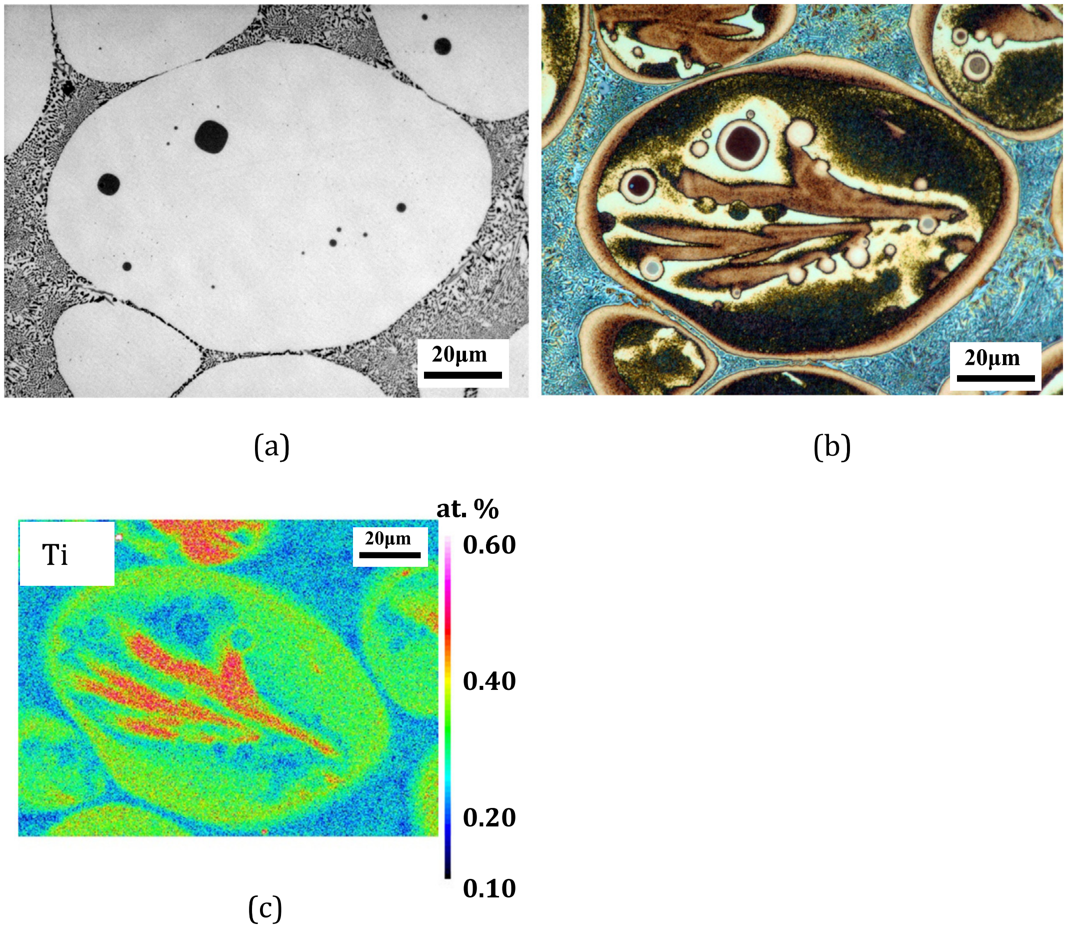

Fig. 1. Previous results showing a spheroidal Al grain before (a) and after (b) etching with Weck's reagent, as well as the Ti micro-segregation detected by EPMA mapping (c).

coefficient of Ti in Al, its micro-segregation could be preserved after semi-solid treatment at high temperature.

Besides A356 Al alloy, Weck's reagent has been proved to work for other Al alloys such as Al–Cu alloy and Al–Si–Cu alloy, etc. [8]. In those researches, all dendritic Al grains present a color difference after etching. However, since Ti is very commonly added into Al alloy as a grain refiner, it is difficult to know if elements other than Ti have any contribution to the color difference or not. Therefore, the attempt of etching Ti-free Al alloys with Weck's reagent is required.

This paper is divided into two parts, in this first part, besides demonstrating various applications of Weck's reagent, we would like to present more interesting results such as the color change depending on the etching time and the re-producibility of using Weck's reagent, etc. While in the second part of this study, we focus on the coloring mechanism of Weck's reagent through detailed observations of the etched surface of Al alloys. By showing these results related to the nature of Weck's reagent, we hope our readers can have a comprehensive understanding of this color etching technique, as well as a wider application of this technique in the future.

2. Materials and methods

Besides A356 aluminum with Ti added, there are three Al alloys used in this research which are Ti-free. All of these four alloys' compositions are listed in Table 1. The compositions of A356 Al alloys (Ti-contained and Ti-free) and Al–7Si binary alloy were measured by an Optical Emission Spectrometer while the Al–5Mg binary alloy's composition was measured by a Sequential Type ICP Emission Spectrometer.

Spheroidization of Al grains in A356 Al alloys with or without Ti added was obtained by re-melting those alloys to semi-solid state after cold compression by 33%. The cold compression was done in Nissan Motor Co., Ltd's factory. Cold compression was done in order to introduce enough strain to the material so that recrystallization could occur during heating and re-melting therefore the grain could be refined and more spheroidized. For the cored microstructure observation, specimens were quickly water quenched when the target semi-solid temperature was reached in order to restrain the homogenization of solute in the Al phase by diffusion. While for solid fraction evaluation, specimens were isothermally held at certain semi-solid temperatures for 5 min to make sure those specimens were heated homogeneously. Through isothermal holding, the solid fraction of the specimen should be close to equilibrium state.

Specimens were polished via standard metallographic techniques, finished using Struers OPS colloidal silica. Subsequently, specimens were immersed in Weck's reagent for approximately 12 s at room temperature. Then the microstructure was observed by optical microscopy (OM) without any filters or analyzers. In order to study the influence of etching time, one specimen was etched for five times, for 4 s, 8 s, 12 s, 20 s and 28 s. Before each time of etching, slight polishing of the previous etched surface was done to remove the etching layer. The

Table 1
Main chemical compositions of the materials used in this research (wt.%).

Alloy	Si	Mg	Fe	Ti	Sr	Mn	Al
Ti-contained A356	6.90	0.39	0.10	0.14	0.025	<0.10	Bal.
Ti-free A356	7.04	0.43	0.13	0.0006	0.0024	0.006	Bal.
Al–7Si	7.23	0.003	0.053	0.009	0.0001	0.008	Bal.
Al–5Mg	ND	5.22	0.094	ND	ND	ND	Bal.

ND: Not detected by ICP.

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