New combination of polishing and etching technique for revealing grain structure of an annealed aluminum (AA1235) alloy

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Commercially pure aluminum alloy AA1235 is commonly used for foil production, and in this production process intermediate annealing is important for the controlling of final texture and mechanical properties of the foil. To determine the correct recrystallization temperature, a study of optical microstructures at different temperatures and times is essential. However, a limited work has been done to reveal recrystallized microstructure of commercial purity aluminum alloy AA1235. Some investigations [1–3] were performed on AA1200 type alloy. However, authors did not report any optical metallography study. Other authors, who worked on commercially pure aluminum [4] or polycrystalline aluminum (99.99% pure) [5], also did not mention the details of the optical metallography technique of these alloys.

There are many proposed methods to reveal grain structure of aluminum and its alloys, e.g., mechanical polishing, electrolytic polishing, chemical etching, and electro-etching [6–8]. In this study, a variety of combination of polishing and etching methods have been used to evaluate their effect on the grain boundary revelation of aluminum alloy AA1235, and the best possible procedure has been proposed along with the development of a modified Poulton's reagent-solution for chemical etching.

Aluminum packaging alloy AA1235 containing Al–0.67Fe–0.16Si–0.01Ti (all in wt.%) has been used in this investigation in annealed condition (at 480 °C for 2 hr). For optical metallography the following sample preparation methods were applied on the annealed specimens:

1. mounting of the sample,
2. grinding and polishing, and
3. etching.

During the mechanical polishing of thin metal sheet mounting of samples is essential to hold it by hand. However, there are some limitations in conventional mounting method. At first, a proper selection of mounting materials and technique must be followed for appropriate protection and preservation of the specimen. Mounting material must have sufficient hardness, similar grinding and polishing characteristics of the sample, resistance to physical distortion due to the heat generation during grinding and polishing, ability to withstand exposures to lubricants, solvents and etchants, penetration to small pores, crevices and the other surface irregularities in the specimen, electrical conductivity for SEM examination, non-toxicity and economical availability [8]. In the current paper conventional mounting methods were avoided since electrolytic polishing would be used and a simple mounting method was followed. For this technique only a small aluminum cylindrical block and double-sided adhesive tape are needed. Aluminum sample was just fixed on the aluminum block by double-sided adhesive tape (Fig. 1).

Rolled surface of the Al-alloy is quite smooth. Therefore, initial grinding was started on the 600 grit SiC paper. During grinding kerosene oil was applied periodically to grinding papers to avoid the sticking of SiC particles at the surface of softer Al-alloys.

Mechanical polishing method consists of following three steps:

1. rough polishing with alumina abrasive particles (5 μm), liquid soap and distilled water on the cotton cloth of medium nap at the 300 rpm wheel speed;
2. polishing with 3 μm diamond paste on the velvet cloth;
3. final polishing with 1 μm diamond paste on the velvet cloth.

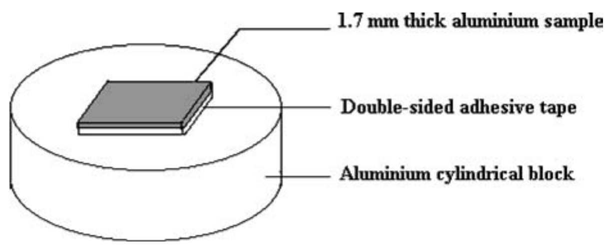

Figure 1 Mounting of the specimen for grinding and polishing.

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TABLE I Details of different types of etching technique

Etching type	Name of the etchant	Composition	Method	Etching time
Chemical	Modified Keller's reagent [6]	10 ml HNO_3 (conc.), 1.5 ml HCl (conc.), 1.0 ml HF (48%), 87.5 ml distilled water	Immersion	30 s
Chemical	NaOH–Na_2CO_3 solution [6]	2 g NaOH, 4 g Na_2CO_3, 94 ml distilled water	Immersion	60 s
Chemical	Poulton's reagent-solution [8]	50 ml Poulton's reagent (12 ml HCl (conc.), 6 ml HNO_3 (conc.), 1 ml distilled water, 1 ml HF (48%)), 25 ml HNO_3 (conc.), 40 ml of solution of 3 g chromic acid per 10 ml of distilled water	Immersion	3 min
Chemical	Modified Poulton's reagent-solution	50 ml Poulton's reagent, 25 ml HNO_3 (conc.), 40 ml of solution of 1 g chromic acid per10 ml of distilled water	Immersion	10 s
Electrolytic	Barker's reagent [8]	4–5 ml HBF_4 (48%), 200 ml distilled water	20 V DC (0.2 A/cm^2), cathode-stainless steel anode-specimen	80 s

In above method, the first step takes around 5 min and second and third steps take 10–20 min each. Adhesive tape was cut to the same size of the sample. There was very little possibility of catching adhesive particles on the tape. After mechanical polishing, the adhesive tape was removed from the sample carefully with the help of kerosene.

Electrolytic polishing method consists of following two steps:

1. rough polishing with alumina abrasive particles (5 μm), liquid soap, and distilled water on the cotton cloth of medium nap at the 300 rpm wheel speed;
2. electrolytic polishing using electrolyte consisting of 800 ml ethanol, 140 ml distilled water, and 60 ml perchloric acid (60%) at 30 V DC and 15 °C temperature for 15 s in ELECTROMET-II.[1] The observed current density during electrolytic polishing was 3 A/cm^2.

In the above method, the first step takes around 5 min and the second step takes about 15 s. After each polishing step, sample was washed under a stream of cold water with liquid soap and was finally washed with ethyl alcohol and dried with warm air.

Two etching methods, chemical and electrolytic, were tried to reveal the grain structure of this alloy. For chemical etching four solutions, e.g., modified Keller's reagent, NaOH–Na_2CO_3 solution and two types of Poulton's reagent-solution (original and modified by us), and for electrolytic etching Barker's reagent were used. Composition and other conditions of the etching are given in Table I.

Nine combinations of polishing and etching methods were applied to develop good grain-contrast of the alloy (Table II). After etching samples were washed under a stream of cold water for 20 min, rinsed in ethanol, and then dried with warm air. Then etched samples were studied under Leica DMRX[2] optical microscope.

[1] Registered trademark of Buehler Ltd., Lake Bluff, Illinois.
[2] Registered trademark of Leica Microsystems, Wetzlar, Germany.

TABLE II Different combinations of polishing and etching methods

Combination no.	Combinations
1	Mechanical polishing and etching by modified Keller's reagent
2	Electrolytic polishing and etching by modified Keller's reagent
3	Mechanical polishing and etching by NaOH–Na_2CO_3 solution
4	Electrolytic polishing and etching by NaOH–Na_2CO_3 solution
5	Mechanical polishing and etching by Barker's reagent
6	Electrolytic polishing and etching by Barker's reagent
7	Mechanical polishing and etching by Poulton's reagent-solution
8	Electrolytic polishing and etching by Poulton's reagent-solution
9	Electrolytic polishing and etching by modified Poulton's reagent-solution

Specimens were prepared by both mechanical and electro-polishing methods. Electro-polished samples are more scratch free than mechanical polished samples. After polishing, samples were at first etched with modified Keller's reagent and observed under an optical microscope (Fig. 2). Keller's reagent could not produce good grain contrast for both mechanical and electro-polished samples, and therefore this reagent was not suitable for such samples.

Some authors [6] recommend NaOH–Na_2CO_3 solution for etching of pure aluminum to reveal good grain structure. Therefore, this solution was also tried to observe grain boundaries. This etchant was applied for both mechanical and electro-polished samples and was unsuccessful in revealing proper grain structure.

Generally, alloys with low alloy content do not respond well to chemical etching method [8]. For these alloys, use of the electrolytic etching method is generally advised. Since electrolytic etching produces a thin anodic film on the surface of the specimen and when this specimen is viewed with a plane-polarized light, a good grain contrast

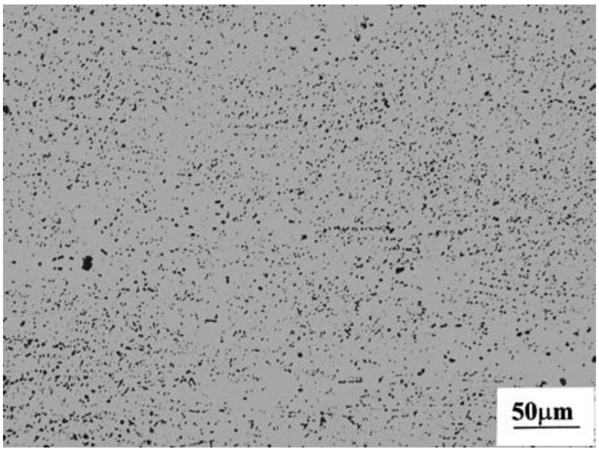

Figure 2 Microstructure of a specimen prepared by electrolytic polishing and etching with Keller's reagent.

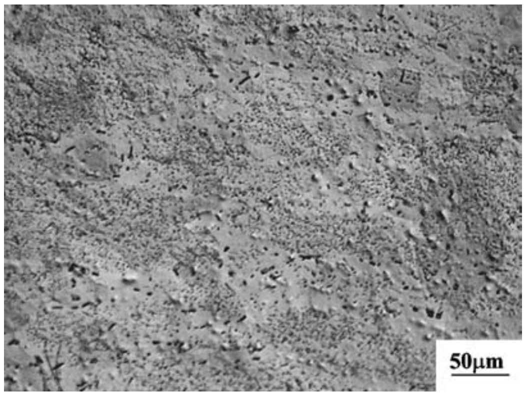

Figure 4 Microstructure of a specimen prepared by electrolytic polishing and etching with Barker's reagent under a polarized light, as observed under a polarized illumination.

can be observed [10, 11]. Some authors [9] use a combination of electrolytic polishing and electrolytic etching in a 1.8% HBF_4 solution (Barker's reagent) using a current of 0.1–0.4 A/cm^2 for commercially pure aluminum. Hence, Barker's reagent was used for both types of polished sample (Figs 3 and 4). But, grain boundaries are still not clearly revealed.

Finally chemical etching method by two types of Poulton's reagent-solutions (original and modified) was applied on both types of polished samples. Mechanically polished samples show better result than earlier attempt (Fig. 5). However, it does not reveal clear grain structures for the microstructural study. Fig. 6 shows the best grain contrast than any other previous methods, still microstructure is not appreciably clear for evaluation of recrystallized volume fraction.

Therefore, a modified Poulton's reagent-solution is developed (Table I) during the course of this study. In this modified etchant, 40 ml of solution of 1 g chromic acid per 10 ml of distilled water is used with 50 ml Poulton's reagent and 25 ml HNO_3 (conc.). Previously it was 40 ml of solution of 3 g chromic acid per 10 ml of distilled water with 50 ml Poulton's reagent and 25 ml HNO_3 (conc.). After applying this new etchant on the electro-polished sample, grain boundaries are revealed within 3–5 s and microstructure is reasonably free from pits (Fig. 7). Therefore, by lowering the density of chromic acid in Poulton's

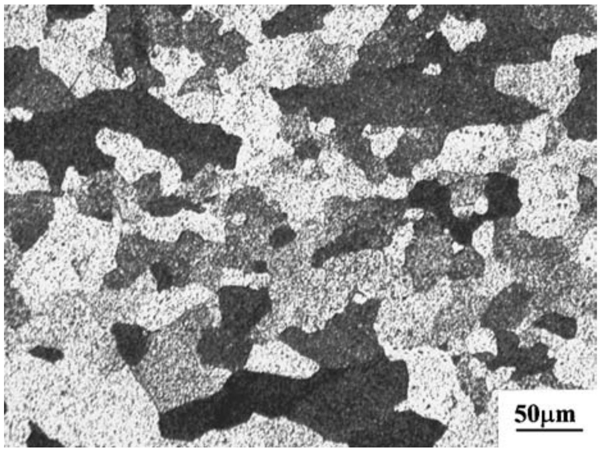

Figure 5 Microstructure of a specimen prepared by mechanical polishing and etching with modified Poulton's reagent-solution.

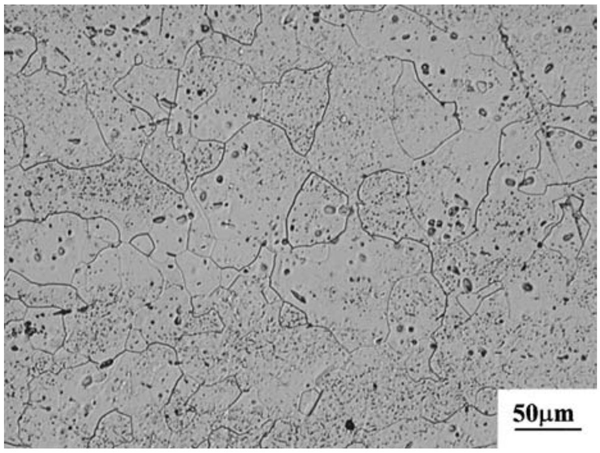

Figure 6 Microstructure of a specimen prepared by electrolytic polishing and etching with Poulton's reagent-solution.

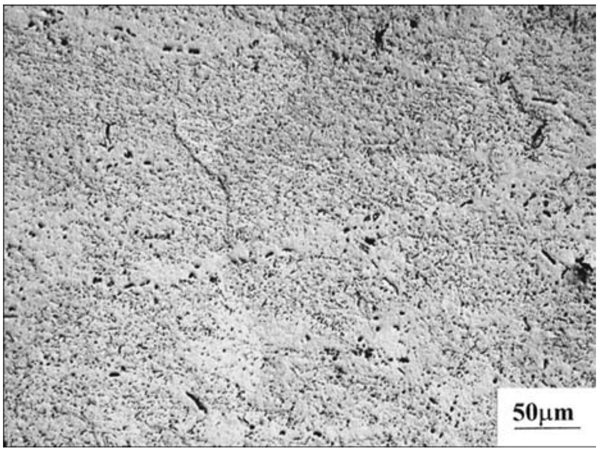

Figure 3 Microstructure of a specimen prepared by mechanical polishing and etching with Barker's reagent, as observed under a polarized light illumination.

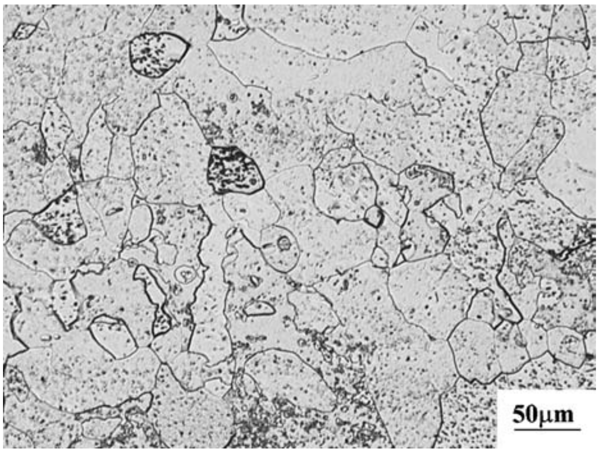

Figure 7 Microstructure of a specimen prepared by electrolytic polishing and etching with modified Poulton's reagent-solution.

reagent-solution grain boundary revelation is significantly improved over any other published information.

So, a new combination of metallographic procedures for annealed aluminum alloy AA1235 has been developed in the present research work. This technique can be used for other similar aluminum alloys. Mounting method is much simpler and easier than any other recommended methods. It also saves time. Mechanical polishing takes around 25–30 min, whereas electrolytic polishing takes only 5 min. Above all, electrolytic polishing gives much better surface finish than any other technique. Several etchants have been used for electropolished samples to reveal the recrystallized grains, and it is observed that Poulton's reagent-solution reveals grain boundary of aluminum alloy AA1235 more efficiently than any other etchant (like Keller's reagent) for aluminum alloys. Modified Poulton's reagent-solution, as developed during the course of this study, gives even better result than the original Poulton's reagent-solution. This superior gain contrast will be helpful for further recrystallization study of the alloy.

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