A NOVEL LIGHT OPTICAL METALLOGRAPHIC TECHNIQUE FOR REVEALING GRAIN STRUCTURES OF ALUMINUM ALLOYS

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ABSTRACT A new metallographic technique has been developed for revealing the grain structures of 2000, 5000, and 7000 series aluminum alloys. This technique consists of four steps:

(1) The aluminum alloy is solution heat treated and water quenched.
(2) An optimized precipitation heat treatment then produces relatively coarse precipitates at grain boundaries and fine precipitates in the grain interior.
(3) The samples are then polished and deliberately overetched. The chemical attack at the grain boundaries is accentuated because of increased susceptibility of intergranular corrosion of the alloy after the heat treatment specified in steps 1 and 2.
(4) The overetched sample is then lightly polished to remove etch pitting and etched precipitates in the grain interior.

The grain contrast of samples prepared with this technique is far superior to that of any existing etching technique for aluminum alloys.

Key Words: Grain Structure, Aluminum Alloys, Metallography, Intergranular Corrosion

1. INTRODUCTION

Determination of the grain size, shape and distribution in polycrystalline materials is probably the single most important metallographic measurement because of the influence of grain size on mechanical properties. Many methods are available to reveal grain structure of aluminum and its alloys [1-5]. However, chemical etching methods using etchants such as Keller's reagent [2] and Graff-Sargent's reagent [4] do not always yield high grain-boundary contrast, because of the variation in the depth of grain boundary steps produced by different rates of chemical attack of neighboring grains during chemical etching. As a result, not all grain boundaries are clearly delineated. Grain contrast also varies with alloys and heat treatment conditions when the standard chemical etching and anodizing techniques are used. Furthermore, if the grain structure of an anodized sample is very fine, contrast between neighboring grains becomes so low that an accurate measurement of grain size is almost impossible. Therefore, an improved technique for revealing grain structures of aluminum alloys is greatly needed.

2. EXPERIMENTAL PROCEDURE

The aluminum alloys used in this investigation were commercial 2124, 5083, and 7475. All materials were received in the form of cold-rolled sheets, ~1.5 mm thick. The new technique for preparing metallographic samples consists of four steps. In step 1, the cold-rolled sheets were salt bath annealed and cold water quenched. The annealing temperature for each of the alloys was high enough to solutionize the principal alloying elements (i.e., Cu, Mg, and Zn). The subsequent water quenching kept these elements in supersaturated solid solution.

Step 2 was a precipitation heat treatment to produce relatively coarse precipitates at grain boundaries and fine precipitates in the grain interior. These precipitates were $CuAl_2$ + Al_2CuMg, Mg_2Al_3, and $MgZn_2$, respectively, in the 2124, 5083, and 7475 alloys. The relatively coarse grain boundary precipitates increased galvanic corrosion effect and accelerated the chemical attack at grain boundaries during subsequent etching. This led to good grain contrast. To optimize the precipitation heat treatment, the annealed and water-quenched sheet samples were heated at various temperatures for various holding times.

In step 3, metallographic specimens were prepared from the heat treated sheet samples and polished according to standard techniques. The polished specimens were then deliberately overetched by doubling or tripling the normal etching time. The etchant used was a solution of 10% H_3PO_4 and 90% H_2O at ~50°C. Compared with normal etching, overetching produced much wider and deeper grooves at grain boundaries, resulting in improved grain contrast.

In the final step (4), the overetched specimens were repolished lightly (20-60 s) with MgO powder in 3% sodium sulfide solution to remove corrosion pitting and etched precipitates in the grain interior. After this final step, an exceptionally high contrast grain structure was revealed.

Steps 3 and 4 could be repeated as many times as needed to produce excellent grain contrast. For comparison of the new technique with a commonly used etching technique, samples were also etched for ~10 s in Keller's reagent containing 1.5% HCl, 1% HF (from 48% concentrated, commercially purchased HF), 2.5% HNO_3, and 95% H_2O.

3. RESULTS AND DISCUSSION

3.1 Metallurgical Basis of the New Technique

The essence of this new technique is the use of precipitates to decorate grain boundaries. To better understand the metallurgical aspects of the technique, a careful examination of the phase diagrams of the three alloy systems is necessary. Figures 1 (a) and (b) show the limits of solid solubility of Al-Cu-Mg (2000 series alloys), Al-Mg (5000 series alloys), respectively [6].

As shown in Fig. 1(a), for 2000 series alloys, which are rich in copper, the ternary eutectic point is 507°C. As the temperature decreases below the solidus, the aluminum-rich apex of the three-phase triangle moves toward the aluminum corner of the diagram. It can be seen that the constituents separating from solid solution are S-Al_2CuMg and θ-$CuAl_2$ or the metastable S' and θ' at relatively low temperatures.

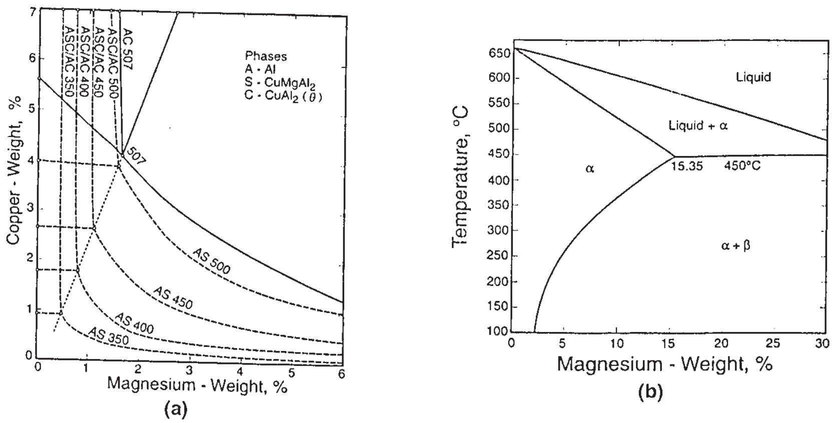

Fig. 1 The limits of solid solubility of (a) Al-Cu-Mg, and (b) Al-Mg.

With aluminum-rich Al-Mg alloys, Fig. 1(b), the eutectic reaction lies at 35% Mg and 450°C. One of the eutectic constituents is designated β or Mg_2Al_3. The aluminum-rich solid solution can contain as much as 15.35% Mg at the eutectic temperature, decreasing to 4.5% at 250°C and about 2% at 100°C.

For 7475 alloy, the majority of the constituents separating from the solid solution are the η-$MgZn_2$ phase or the metastable η' at relatively low temperatures. This phase, which is also capable of dissolving significant amounts of Cu, is completely taken into solid solution at 460°C and above.

As can be seen from the phase diagrams, when these aluminum alloys are solution heat treated, water quenched, and precipitation heat treated, precipitation will occur from supersaturated solid solution. Grain boundary precipitates are much coarser than the precipitates in the grain interior because of faster solute diffusion at grain boundaries. During etching of the polished samples, the relatively coarse and continuous grain boundary precipitates will accentuate the chemical attack at grain boundaries and delineate them uniformly. Overetching produces wide and deep grooves at grain boundaries, improving grain structure contrast. However, overetching also reveals the fine precipitates in the grain interior. Therefore, a light repolish is needed to clean up the grain interior and produce clearly delineated grain boundaries.

3.2 Optimization of the Temperature and Time in the Precipitation Heat Treatment

Figures 2(a)-(d) show the grain structures of 2124 alloy samples prepared metallographically using the new technique. The optimum temperature of the one-hour precipitation heat treatment for

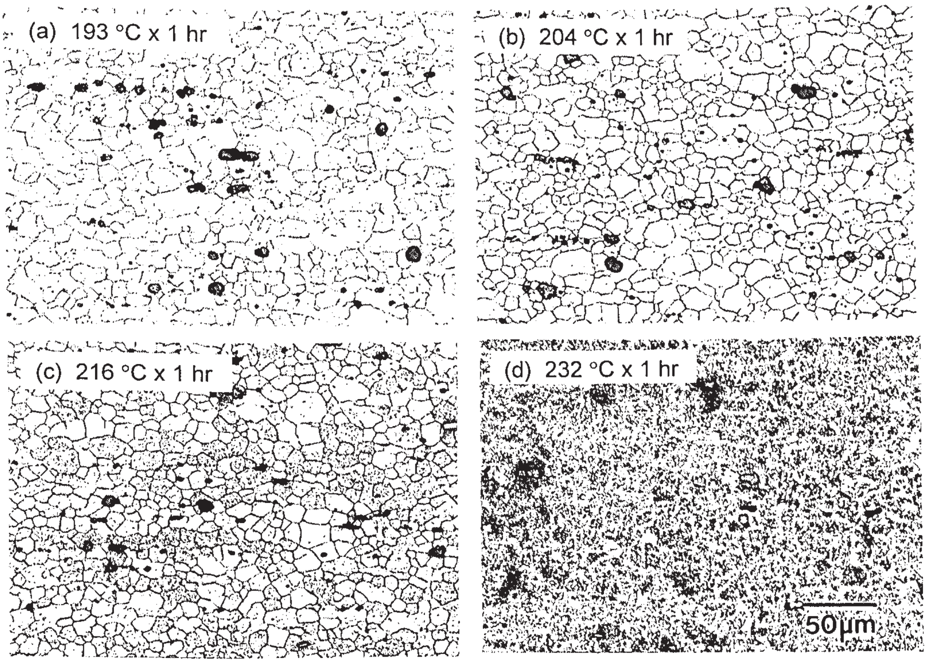

Fig. 2 Grain structures of 2124 samples salt bath annealed at 493°C for 15 mins, water quenched, and precipitation heat treated for 1 hr at (a) 193, (b) 204, (c) 216, and (d) 232°C.

revealing grain structure in the 2124 alloy was found to be about 204°C, Fig. 2(b). At 193°C and lower temperatures, precipitation heat treating for 1h did not produce clearly and continuously decorated grain boundaries. At 232°C and above, uniform attack of the grain interiors occurred and, as a result, grain contrast was low.

In the 5083 alloy, grain structure could not be clearly revealed by heat treating for 1h at any temperature below 250°C which is the solutionizing temperature of Mg_2Al_3. This is because the diffusivity of Mg in Al is low at below 250°C. Among the samples heat treated for 24h at 121, 132 and 143°C, the samples heat treated at the higher temperatures had better grain contrast. Slightly better grain contrast was obtained in the samples heat treated for longer times (e.g., 72h) at 132°C.

The optimum temperature range of the 1h precipitation heat treatment for revealing grain structure in the 7475 alloy was found to be relatively wide, 182-216°C. Such a wide range of precipitation heat-treating temperatures is a result of the high content of solute elements in this alloy. At 232°C, the grain boundary grooves became so wide that it was difficult to resolve very fine grains.

The relation between the optimum precipitation heat-treating temperature and the time for revealing grain boundaries is determined by the following factors:

(1) The diffusion rate in aluminum of those principal alloying elements that form the precipitates
(2) The solid solubility and the amount of alloying elements available for precipitation at a given heat treatment temperature
(3) Etching characteristics of the grain boundaries decorated with those precipitates

The principal alloying elements, Cu, Mg and Zn, have relatively high rates of diffusion in aluminum. For precipitates to grow by diffusion to a size large enough to effectively accentuate chemical attack at grain boundaries, the temperature and time required would obey the following Arrhenius type of equation:

$$D_O \cdot \exp[-Q/(R \cdot T)] \cdot t = \text{constant} \quad \text{...... (1)}$$

where D_O is the diffusion coefficient, Q the activation energy, R the constant, 8.31 J/(mol·K), T the temperature in kelvins, and t the time. In diffusional processes including both grain boundary diffusion and lattice diffusion, lattice diffusion is the rate-limiting step. Equation (1) is plotted schematically in Fig. 3 as temperature - time, or T-t, curves.

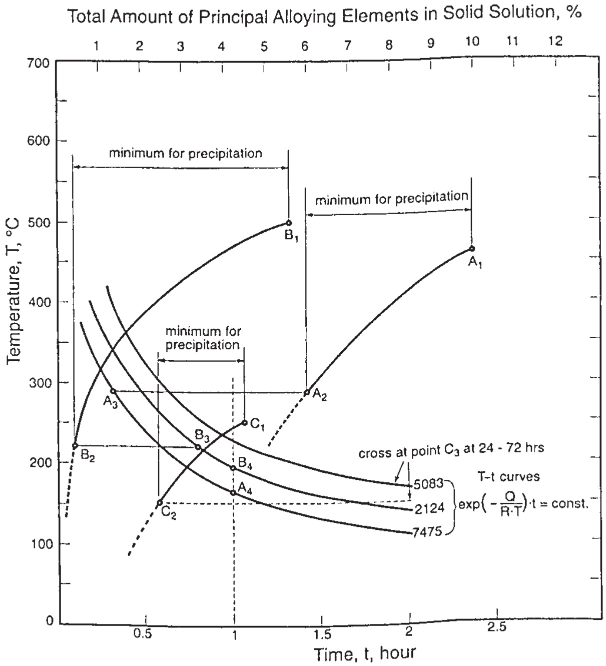

Fig. 3 Schematic T-t curves for optimum precipitation heat treatment to reveal grain structure and the solid solubility curves of 7475, 2124, and 5083 alloys.

The shift of the T-t curves between 5083, 2124, and 7475 alloys corresponds to the differences in the levels of the principal elements available for precipitation, in the type of precipitates, and in the etching characteristics of the grain boundaries decorated with these precipitates.

The total amount of principal alloying elements that can separate from solid solution to form precipitates at a specific heat treating temperature is the alloy composition minus the solid solubility at that temperature. The solid solubility of all principal elements in each of the three alloys is plotted schematically as a function of temperature in Fig. 3.

The solubility curve of the 7475 alloy starts at point A_1 which is defined by the total amount of principal alloying elements in the alloy and the minimum solution heat temperature, 460°C. To obtain the desired etching response, the precipitation temperature must be low enough (e.g., at point A_2), that the solubility is reduced, creating sufficient Zn, Mg, and Cu for precipitation. At this temperature, the required soaking time is represented by point A_3 on the T-t curve. If a typical precipitation heat-treating time is limited to 1 h or less, then the optimum temperature range of the precipitation heat treatment for 7475 alloy is given by A_3A_4.

Similarly, B_3B_4 is the optimum temperature range for the 2124 alloy, which is a rather narrow range. This has been shown by the experimental results presented earlier. However, if the heat treating time is not limited to 1 h, the 2124 alloy can be heat treated at temperatures lower than B_4 for longer times to obtain better grain boundary contrast.

In the 5083 alloy, the horizontal line passing through C_2 crosses the T-t curve at point C_3 which corresponds to a soak time of 24-72 h. The much longer soaking time than is necessary for 7475 and 2124 is due to the low solutionizing temperature of Mg_2Al_3 and the low Mg diffusion rate at low temperatures.

3.3 Applicability of the Technique to Aluminum Alloys in Different Tempers and of Different Compositions and to Other Materials

This new technique is applicable to most aluminum alloy tempers for revealing grain or subgrain structure or both, regardless of whether the material is recrystallized, recovered or partially recrystallized. The heat treatment steps (i.e., steps 1 and 2) may be modified depending on the alloy tempers:

(1) *F-temper or H-temper.* If the material is in the cold worked condition, as in the present investigation, it must be annealed at a temperature that will not only recrystallize the cold worked structure, but also keep the principal alloying elements in solid solution.

(2) *O-temper.* The material must be resolution heat treated, and the temperature and time in this heat treatment should be kept to a minimum in order to minimize grain growth.

(3) *W- and T-temper.* In such cases, step 1 of this four-step technique is not needed because the material has already been solution heat treated and quenched.

The optimum temperature and time in the precipitation heat treatment for achieving good grain boundary contrast depends on the alloy composition even if the alloys fall into the same alloy series.

This technique is not sensitive to the metal-working process by which a material is fabricated (e.g., extruded, rolled, drawn, forged, etc.). The concept of this technique should also be applicable to 6000 series aluminum alloys, to aluminum metal-matrix composites, and to other non-aluminum single phase alloys in which revealing grain boundaries may be difficult.

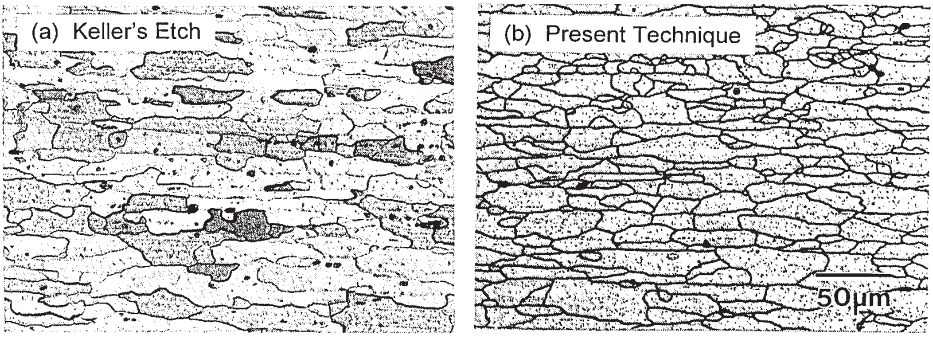

Fig. 4 Comparison of typical grain structures of a 7475 aluminum alloy using (a) Keller's etch and (b) the present technique.

3.4 Comparison with Other Etching Techniques

Figure 4 is a comparison of typical grain structure of a 7475 aluminum alloy obtained by using the present technique and Keller's etch. In Keller's etch, Fig. 4(a), grains were attacked at different rates, depending on their crystallographic orientation, producing steps at the grain boundaries. Because of the variation in the depth and orientation of these grain boundary steps, not all grain boundaries are clearly revealed in the micrograph. In comparison, the new technique produces distinct, continuous grain boundaries, Fig. 4(b). The binary black boundary - white matrix contrast produced by this new technique is ideally suited to automated image analysis. Grain structures revealed by Keller's etch cannot be used for automated systems.

4. CONCLUSIONS

A new metallographic technique has been developed for revealing the grain structures of common 2000, 5000, and 7000 series aluminum alloys. This technique consists of four steps: (1) solution heat treatment and cold water quench; (2) precipitation heat treatment; (3) polishing and overetching; and (4) light repolishing. The grain contrast of samples prepared with the new technique is far superior to any existing etching techniques reported in the literature.

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