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A new etching technique for revealing the plastic deformation zone in an Al–Cu–Mg alloy

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Abstract A new etching technique for revealing the plastic deformation zone in an Al–Cu–Mg alloy has been developed. The etching with the proposed etching agent was conducted on the deformed sample after being heated to 673 K for 3 h. With this etching technique, the plastic deformation zone was clearly observed even under low magnification. This was due to the change of microstructural characteristics in the plastic deformation zone after the heating process, in which there is significant precipitation of Al_2Cu and Mg_2Si, caused by the high energy arising from the severe deformation.

Introduction

Aluminum alloys are widely utilized in various structures and components because of their good strength and low density [1]. Specifically, the stringent demands in aircraft manufacture for high-strength, low density and tough materials has been met by the use of aluminum alloys such as duralumin. In addition, many automobile parts have been manufactured with Al alloys instead of steel materials to reduce the weight of the car [2]. It is self-evident that understanding the mechanical properties and fatigue strength of such alloys is of significant importance in order to make structural integrity assessments. In particular, investigation of the fatigue behavior is important to assess the material fatigue life. There are several experimental reports in which the crack growth characteristics for aluminum alloys have been directly related to the severity of the plastic deformation around the crack tip [3–5]. In order to understand the effect of the extent of plastic strain on fatigue behavior, the deformation zone adjacent to the crack tip has been investigated by various experimental techniques, including etching techniques, photoelastic stress analysis and microhardness measurements [6–18]. The etching techniques proposed by Fry [6] and Morris [7] have been utilized by several researchers because of their simplicity. Their etching reagents are based on the observation that due to the segregation of impurity atoms at dislocation cores, etch-pit formation occurs where dislocations are located. The Fry etching technique has been used in the study of Evans and Lu [19], who observed plastic deformation zones in the bending of specimens made of silicon steel. With their approach, the plastic strain distributions were clearly obtained and are in good agreement with their theoretical estimates. Using Fry's solution, Birol [20] has also examined the severity of the plastic strain surrounding the fatigue crack in a specimen material of Fe–2.6%Si steel. It was found that the more severe the plastic deformation, the slower the crack growth rate. More recently, a new etching technique was proposed in a study by one of the authors [11, 12]. With this technique, the plastically deformed zone in a sample of SAE1015 steel was darkly etched whilst being brightly etched in the undeformed zone.

From the literature survey described above, various etching techniques have been developed to observe plastic deformation zones. However, it seems that there are material limitations to these etching techniques. Although high strength aluminum alloys, e.g., duralumin, have been extensively utilized for various engineering applications, there is apparently no etching technique available in this

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case. The purpose of this work was therefore to propose a new etching technique to detect the plastic deformation zone in aluminum alloys such as 2017 duralumin. In addition, an attempt was made to explain the etching mechanism for revealing the plastic deformation zone.

Experimental

Material and experimental procedures

In the present work, a cold-rolled 2017 aluminum alloy (Al–Cu–Mg) was selected. This was supplied in the form of a 25.4 × 25.4 mm rectangular bar. The chemical composition of the aluminum alloy is (wt%): 4.4 Cu, 0.5 Mg, 0.8 Mn, 0.8 Si and balanced with Al. The tensile properties of the alloy at room temperature are: 0.2% proof strength $\sigma_{0.2} = 69$ MPa, tensile strength $\sigma_{UTS} = 181$ MPa and elongation = 22%. A severe plastic deformation was introduced by imposing a hardened trapezoidally shaped rod, 700 HV, at a compressive force of 5 KN at a rate 1 mm/min on to the specimen in the form of a rectangular block (10 × 10 × 5 mm). An electro-servo-hydraulic system with a capacity of 100 KN was employed to carry out the compression test. Figure 1a, b shows SEM micrographs of the plastic deformation and undeformed zones. The micrographs are further enhanced by the high magnification inverse images (Fig. 1c, d), which clearly show the secondary phase characteristics. By comparison of the microstructure images of Fig. 1c, d, the difference in the microstructural characteristics is obvious. Due to the severer deformation, the microstructural morphology in the deformation zone is distorted.

An attempt has been made to develop a new etching technique for the identification of the plastic deformation zone. The essence of this etching approach is to distinguish the deformation zone by the different degrees of etching severity [21]. The etching technique proposed in the present work is briefly summarized as follows: (i) the specimens are compressed to produce a severe plastic deformation; (ii) the deformed samples are heated at different heating temperatures, e.g., 573, 673, and 773 K for

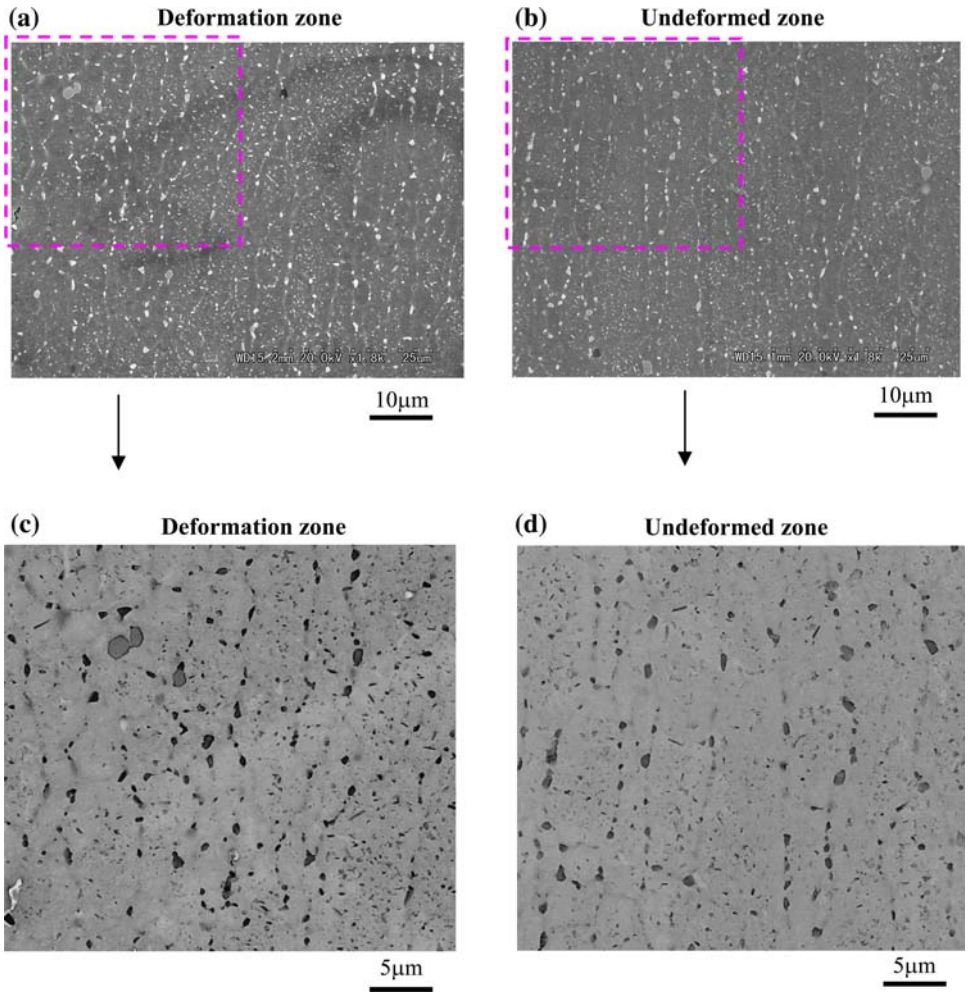

Fig. 1 SEM micrographs of 2017 duralumin in (**a**) and (**c**) the plastic deformation zone, (**b**) and (**d**) undeformed zone

3 h, respectively. The choice of heating temperature is determined by the recrystallization temperature of this alloy which is 723 K [2]. (iii) The specimen surface to be observed is ground to a mirror finish; (iv) the polished surface is then etched for 1 min using the proposed etchant, 4 mL HF, 6 mL HCl, 8 mL HNO_3, 80 mL H_2O and 50 mL ethyl alcohol. Note that the design of the etching solution has been based on the Dix-Keller reagent [8].

Finite element analysis

In order to examine the plastic strain distribution in the sample after the compressive test, finite element analysis (FEA) was conducted using the ANSYS 11 program. Figure 2 shows the FEA model for the present study based on the compression test as mentioned above. In this analysis, a two-dimensional finite element simulation with 8-node quad elements was used. The mesh size in the sample surrounding the trapezoidal rod was 0.01 mm. For our investigation the case of bilinear kinematics hardening was selected, where the initial slope of the stress–strain curve is taken as the elastic constant of the material, and after plastic yield the curve continues along the second slope defined by the tangent modulus. The following material properties were employed: elastic constant $E = 73$ GPa, tangent modulus $T = 7.3$ GPa [22], Poisson's ratio $v = 0.33$ and yield strength $\sigma_y = 69$ MPa.

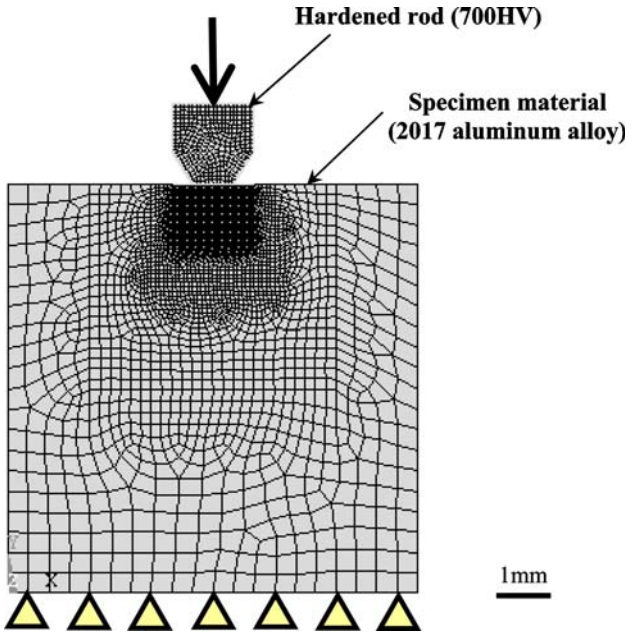

Fig. 2 FE analysis model used to determine the plastic strain distribution

Results and discussion

Figure 3 shows the macrostructure of the etched samples in the area surrounding the dented zone. After heating the samples to various temperatures, it was found that heating to 673 K results in a dark colored region in the vicinity of the dent zone as shown in Fig. 3b, c. In contrast there is no change of macrostructure for the samples heated to 573 and 773 K, as seen in Fig. 3a, d. From the profile of the dark area in Fig. 3b, c, we consider that this region can be attributed to the actual plastic deformation zone [11, 12]. In order to confirm this, microhardness measurements in the dark and bright regions in the sample were carried out after the compression test. Figure 4a shows the results of the microhardness mapping (see Fig. 4b). As can be seen, microhardness values between 100 HV and 130 HV are measured in the region of the sample surrounding the dent area, and these are about 1.25 times higher than that in other regions. Juijerm and Altenberger [23] have investigated the microhardness in the plastic deformation zone of a 6110 (Al–Mg–Si–Cu) alloy. In their examination, the hardness in the work-hardened zone was approximately 1.15 times higher than that in the undeformed zone, which is close to the present ratio of 1.25. Therefore, the discolored area on the etched surface shown in Fig. 3b, c could reflect the actual plastic deformation zone. In order to confirm this in terms of stress–strain distribution we have carried out a further FE analysis. Figure 5 presents the equivalent plastic strain distribution in the specimen. As can be seen, the shape of the plastic strain distribution is similar to the darkly etched zone as well as the hardened area. From this result, we are convinced that the dark area is associated with the actual plastic deformation.

As mentioned above, the plastic deformation zone was discolored, and this discoloration can be attributed to a different response to the etching procedure. In order to investigate the reason for this, we conducted a microstructural examination after etching. Figure 6 shows the SEM micrographs of the deformation and undeformed zones after heating to 573, 673, or 773 K for 3 h, respectively. By comparing Fig. 6e, f with Fig. 1a, b, recrystallization is evident when the sample is heated to 773 K, whereas indistinct microstructural changes are detected after heating to 573 K (Fig. 6a, b). In contrast, substantial microstructural changes were observed in the deformation zone after heating to 673 K, in which many pits appeared on the sample surface, as shown in Fig. 6c. This characteristic cannot be observed in the undeformed sample (Fig. 6d). It is considered from this result that the rough sample surface, caused by the pit formation, could correlate the plastic deformation zone with the dark color as presented in Fig. 3b, c.

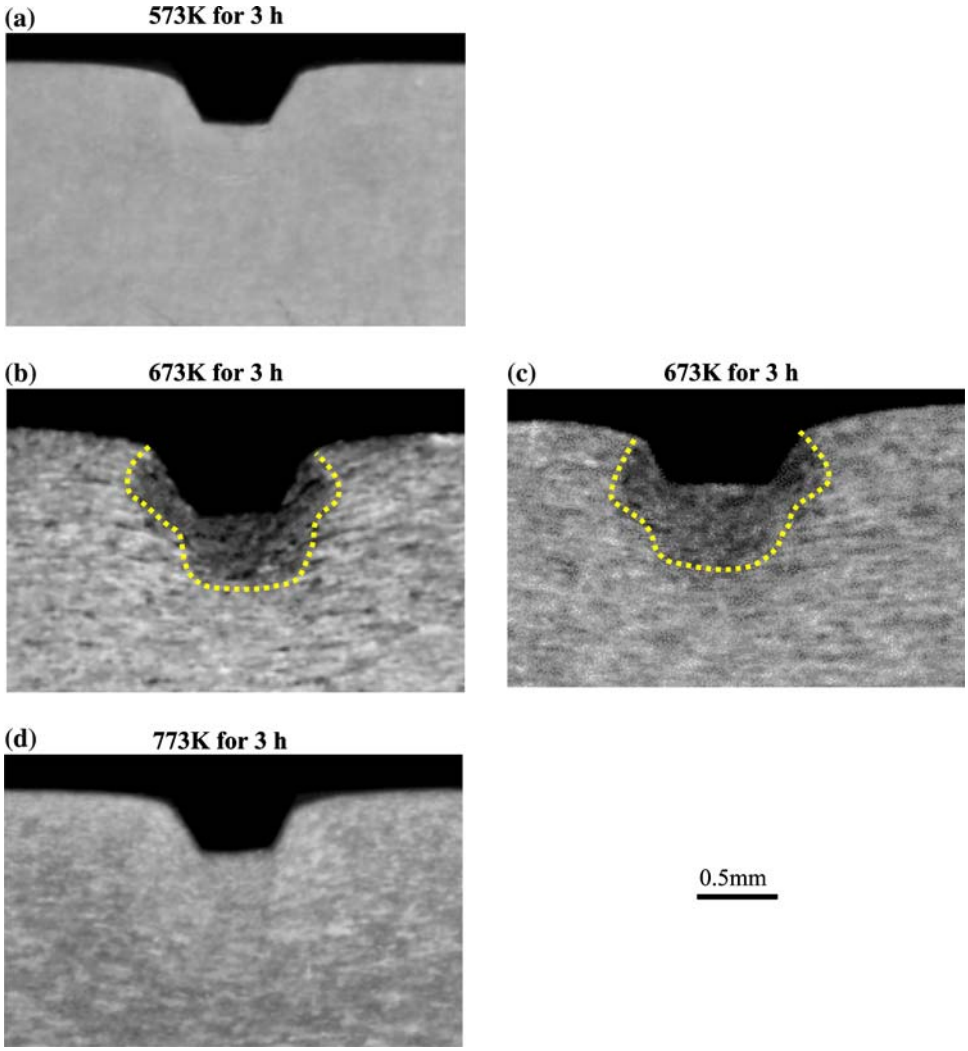

Fig. 3 Optical macrographs of 2017 duralumin after etching. The sample was heated to (**a**) 573 K for 3 h, (**b**) and (**c**) 673 K for 3 h, (**d**) 773 K for 3 h. (The dark region is the plastic deformation zone indicated by the dashed line)

In order to understand the reason for pit formation in the deformation zone (Fig. 6c), the microstructural characteristics for the 673 K sample were further examined. Figure 7a, b displays the SEM microstructure image of the 673 K sample. It should be noted that the microstructures in Fig. 7 were exposed after etching for a short period of time (10 s). As seen in Fig. 7, a large number of precipitation particles are detected. It can also be seen in Fig. 7 that some of the particles seem to drop from the matrix during the etching process, as indicated by the arrows, and

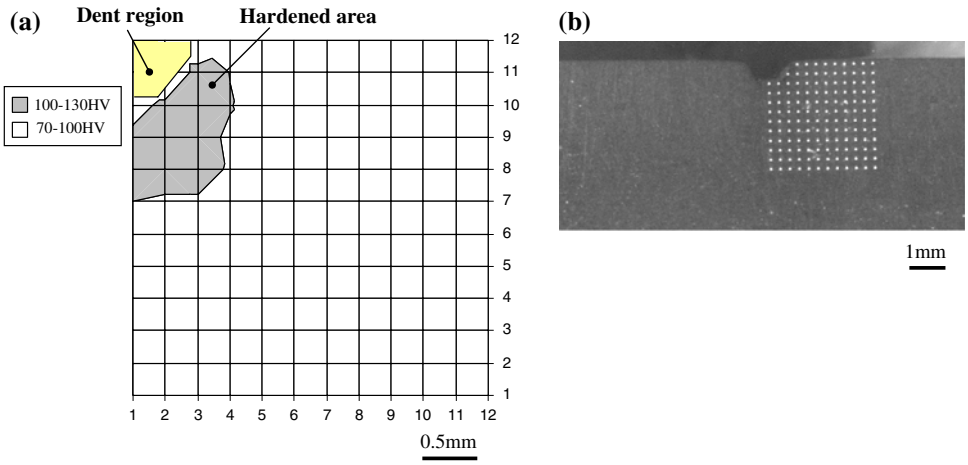

Fig. 4 (**a**) Microhardness mapping of the deformed and undeformed zones after the compression test, (**b**) the position of the hardness test as indicated by the white dot

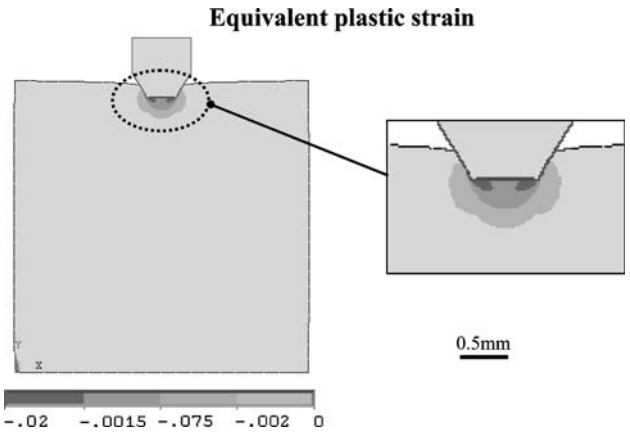

Fig. 5 FEA equivalent plastic strain distribution obtained by compressive stress

this leads to pit formation as mentioned in Fig. 6c. From this result, it is considered that the change of microstructural characteristics in Fig. 6c can be attributed to the presence of many precipitation particles, generated by the high energy arising from the severe plastic deformation [11, 21]. It should be pointed out that the precipitation particles may possibly be attributed to copper and magnesium, given the chemical composition of this alloy. In order to verify this, an EDX analysis was carried out. The results of the EDX analysis are displayed in Fig. 8. It is seen that considerable quantities of copper and magnesium can be detected in the precipitation particles. From this analysis, we consider the particles to be related to Al_2Cu and Mg_2Si [1].

Conclusions

Based upon the above experimental and numerical results, the following conclusions can be drawn:

1. The localized plastic deformation in the 2017 duralumin sample can be revealed via the proposed etching

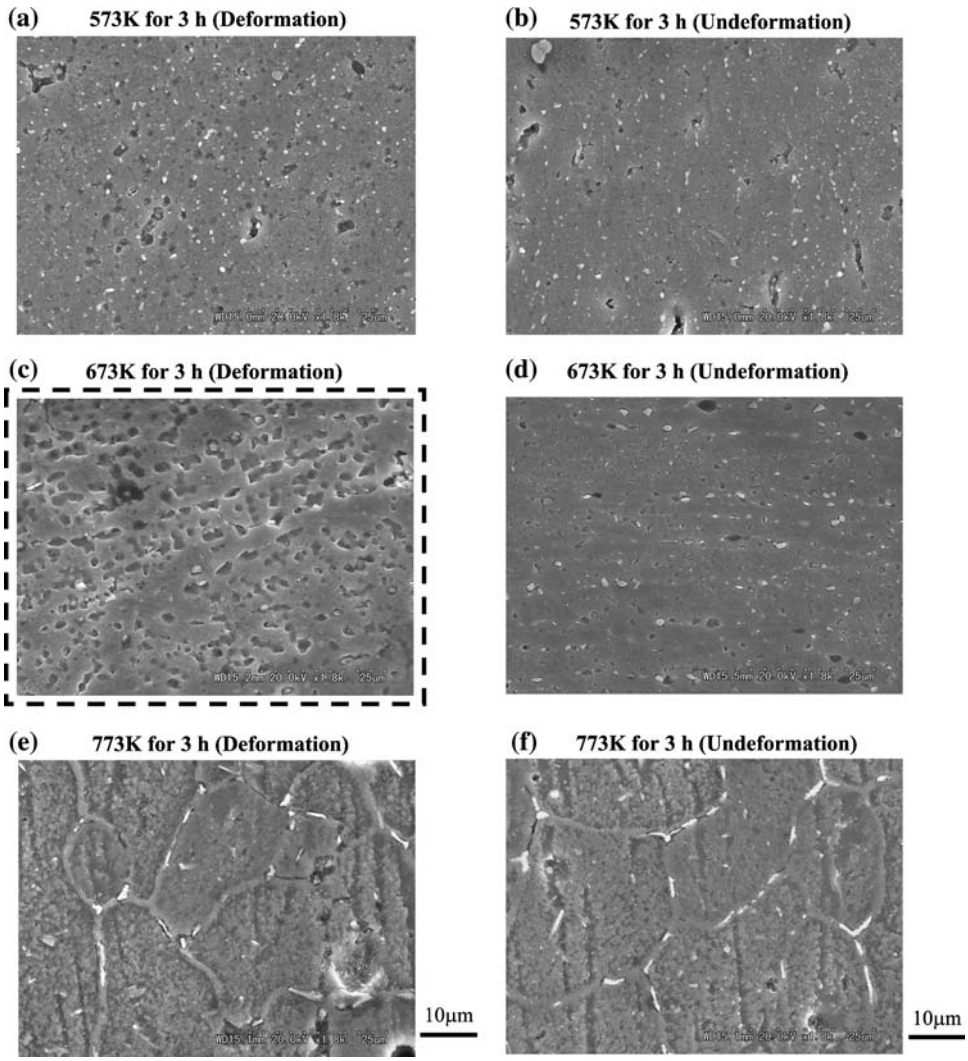

Fig. 6 SEM micrographs in the deformed and undeformed zones after etching for 1 min: (**a**) and (**b**) 573 K sample, (**c**) and (**d**) 673 K sample, (**e**) and (**f**) 773 K sample

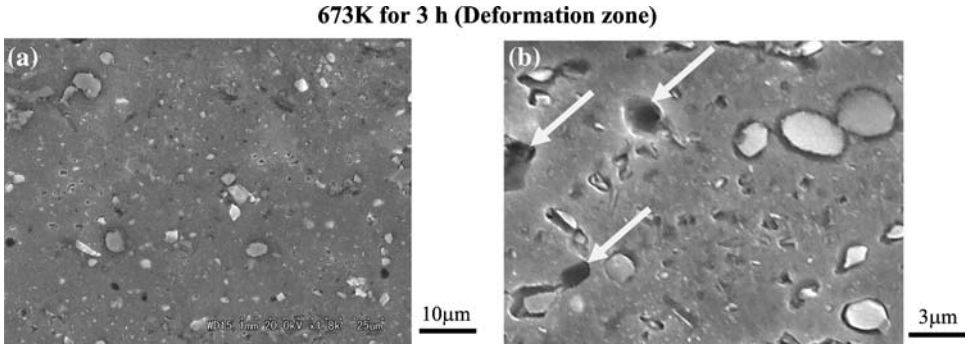

Fig. 7 (**a**) and (**b**) SEM micrographs of the 673 K sample in the deformation zones after etching for 10 s, showing the increase of precipitated particle size

technique which is as follows: (i) the samples deformed plastically are heated to 673 K for 3 h then allowed to cool in the furnace; (ii) the sample surface is polished to a mirror finish; (iii) the polished surface is etched with 4 mL HF, 6 mL HCl, 8 mL HNO_3, 80 mL H_2O, and 50 mL ethyl alcohol for 1 min. This design of this proposed etching reagent was based on the Dix-Keller solution.

2. After etching, the plastic deformation zone was darkly etched, but lightly etched in the undeformed zone. From the discoloration, the plastic deformation zone could be observed clearly even at low magnification. The deformation zone as revealed in this way was in good agreement with the distribution of the strain hardening regions obtained by microhardness measurements and finite element analysis.

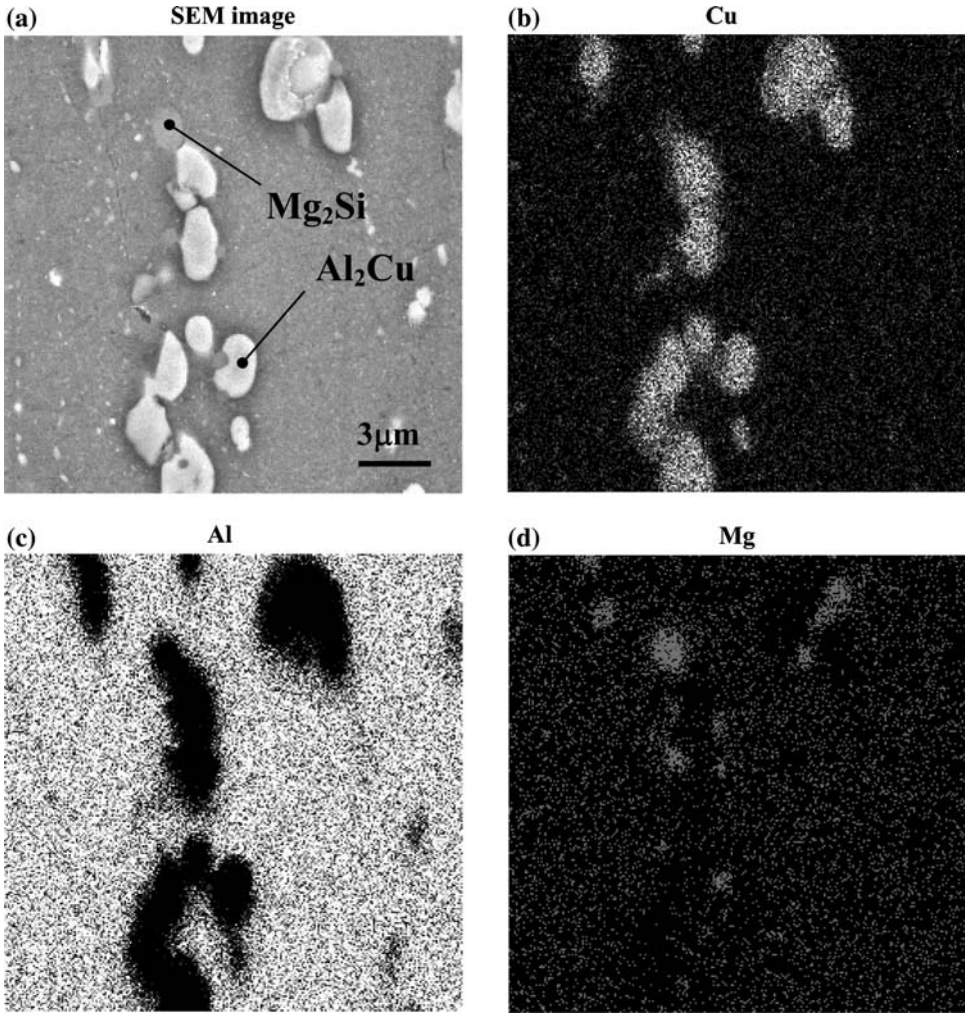

Fig. 8 EDX mapping of the sample after heating to 673 K for 3 h, showing precipitated Al_2Cu and Mg_2Si

3. The etching mechanism for exposing the plastic deformation zone was influenced by the change of microstructure in the deformation zone, where a number of precipitation particles, such as Al_2Cu and Mg_2Si, were detected. The precipitated particles were generated by the high energy resulting from the severe plastic deformation.

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