MICROSTRUCTURE IN Al-Ti BASE ALLOYS AFFECTED BY A REPEATED WORKING PROCESS

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Abstract Research program of applying repeated working process similar to ECAP (Equal Channel Angular Pressing) to Al-Ti base alloys (Al-10mol%Ti-3.6mol%Mn Al-10mol%Ti-3.2mol%Cr) is underway to obtain a fine dispersion of second phase particles with high volume fraction. As a first step, in this study, a repeated working process was applied to the Al-Ti-Mn alloy ingot at 600℃. Although the bolts clamping the dies were seriously deformed at the first pass and further working became impossible, significant refinement of the second phase particles was observed. It was found from X-ray diffraction analysis that two intermetallic phases, Al_6Mn (or $Al_{13}Cr_2$) and Al_3Ti with DO_{22} structure, were present together with aluminum matrix in the ingots, and that they slightly increased in amount when annealed at 650℃ for 24h.

Keywords: *repeated working, second phase, refinement, aluminum-titanium base alloy,*

1. Introduction

Fine dispersion of second phase particles with high volume fraction by means of heat treatment is limited in aluminum alloys, since there is no allotropic transformation and there are few alloying elements that have large solid solubility. Although to break through this drawback rapid solidification and mechanical alloying processes have been attempted so far, none of them has yet been commercialized, because both processes are involved with powder metallurgy route, resulting in serious disadvantage such as impurity pick-up and high cost. However, mechanical alloying is based on repeated working of a great number of cycles[1], and thus seems possible to be applied to the bulk material. A kind of repeated working process, Equal Channel Angular Pressing (ECAP) illustrated in Fig.1, has been already applied to bulk aluminum base materials consisting of single phase (pure aluminum and Al-3mol%Mg alloy), resulting in an increase in dislocation density and a reduction in grain size[2-4].

Research project has been started by the authors in which a repeated working process similar to ECAP is applied to Al-Ti base alloys that contain coarse second phase particles with high volume fraction in the as-cast state, in order to refine the dispersion of the particles. The reason for selecting titanium as a primary alloying element is that it does not seriously harm the light weight, an advantage of aluminum, and that coarsening of the second phase particle was expected to be inhibited even in hot working because of extremely small diffusivity of titanium in aluminum[5]. It is thus expected that a new lightweight material with high heat resistance can be obtained.

In Al-Ti binary alloy, the second phase that exists with aluminum matrix in equilibrium, is Al_3Ti, having poor deformability caused by its tetragonal DO_{22} structure. When the second phase particle is hard with lower deformability, a marked refining effect of repeated working can not be expected since the deformation will be restricted in the matrix after the morphology of the particle becomes equiaxed. In contrast, when deformed to some extent together with the matrix, the particle will be elongated and then cracked, leading to a more marked refinement. These features are schematically illustrated in Fig.2. Addition of Mn or Cr to Al_3Ti monolithic intermetallic compound was reported to induce replacement of Al with the additive, forming $Al_{66}Ti_{25}Mn_9$ or $Al_{67}Ti_{25}Cr_8$ and changing the structure into a cubic $L1_2$[6-8] which is frequently associated with higher deformability.

In this study, Al-10mol%Ti-3.6mol%Mn and Al-10mol%Ti-3.2mol%Cr alloys, having the same Ti/Mn and Ti/Cr ratios as the monolithic $L1_2$ compounds, have been used as specimens. It is reported that a metastable $L1_2$ Al_3Ti phase forms during rapid solidification in Al-Ti binary alloys[9]. Hence, the alloys might contain the $L1_2$ $(Al,Mn)_3Ti$ and $(Al,Cr)_3Ti$ compounds as second phases, respectively, possibly having greater deformability, although the Al-Ti-Mn and Al-Ti-Cr ternary phase diagrams near the aluminum corner do not seem to have been well established

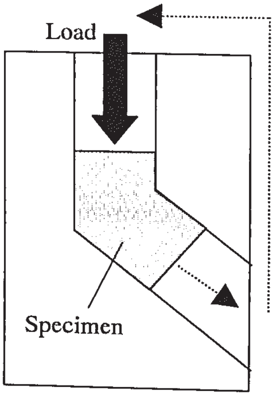


Fig.1 Schematic illustration for ECAP process.

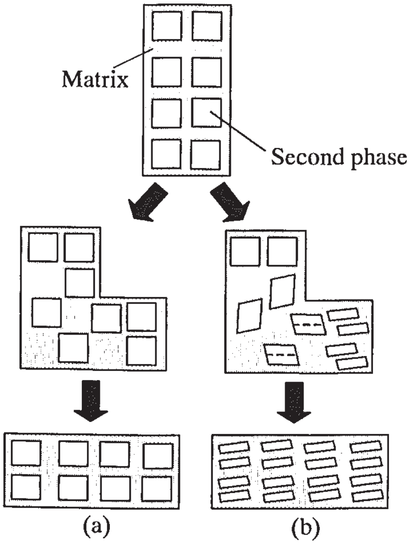


Fig.2 Schematic illustration showing the difference in the dispersion of the second phase particles arising from the deformability of the particles when a two-phase alloy is side extruded. (a) with hard and undeformable second phase particles, (b) with second phase particles that are deformable to some extent together with the matrix.

yet. This paper deals the results of microstructural characterization obtained by an attempt to apply a repeated working process to these alloy ingots.

2. Experimental Procedures

From raw materials of 99.99%Al, 99.9%Ti, 99.9%Mn and 99.9%Cr, Al-10mol%Ti-3.6mol%Mn and Al-10mol%Ti-3.2mol%Cr alloys were induction melted under argon atmosphere and then cast in an iron mold. Pieces of 10×20×65mm were cut from the ingots. To reduce the amount of defects caused by solidification, the pieces were inserted in a set of closed dies, soaked at 600℃ for 1h together with the dies, hot-pressed at a load of about 100kN on an hydraulytic universal testing machine in longitudinal direction at room temperature immediately after the soaking without any artificial cooling.

Prior to repeated working, uniaxial compression was applied to the specimens of 8×8×8mm cut from the hot-pressed pieces on a hydrolytic compression-testing machine with a load capacity of 2MN by different reductions up to 88%. The compressed samples as well as the as-cast ingots were subjected to microstructural characterization by means of optical microscopy and X-ray diffraction after filing. Repeated working was conducted using a set of dies and other components illustrated in Fig.3. In a similar manner to that in hot-pressing, a 10×20×65mm piece cut from the Al-Ti-Mn alloy ingot was soaked in the dies at 600℃ for 1h, and then side extruded at room temperature immediately after the soaking without any artificial working. The strain obtained by this side extrusion process of 1 pass is about 1.15[10]. However, during the first pass of the side extrusion, the bolts clamping the dies were seriously deformed and damaged because of the lack in high-temperature strength of the bolts. Consequently, complete side extrusion of even a single pass was not achieved. For the deformed region of the incompletely side-extruded sample, transmission electron microscopy using Jeol 2010 was carried out as well as optical microscopy.

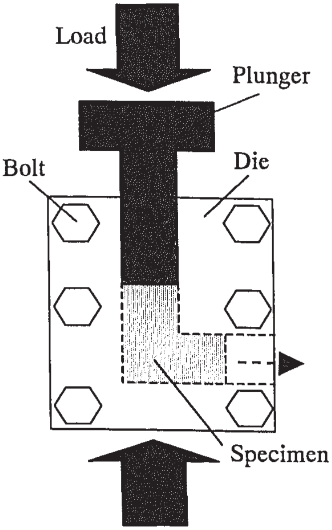


Fig.3 Dies and related components used in the repeated working process.

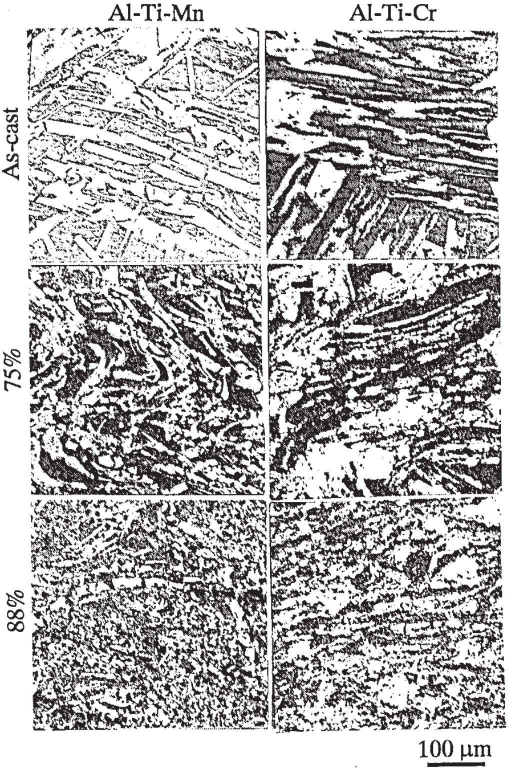


Fig.4 Optical micrographs of the as-cast ingots and the specimens uniaxially compressed by the indicated reduction.

3. Experimental Results and Discussion

Figure 4 shows optical micrograph of the two alloy specimens uniaxially compressed at room temperature by different reductions, along with those of the as-cast ingots. As-cast ingots of both alloys contain coarse second phase particles (light area) of about 50µm in width and 200µm in length. It is evident that these particles are markedly refined as the reduction is increased. However, further reduction was not attainable in the uniaxial compression arising from the limit both in the load capacity of the machine and in the deformability of the specimen; many side cracks were observed in the specimen deformed by 88%.

Then an attempt to apply repeated working by the side extrusion shown in Fig.3 to the Al-Ti-Mn alloy ingot was made at 600℃. As mentioned earlier, however, the bolts fixing the two dies in Fig.3 were seriously deformed and damaged during the first pass from lack of the high-temperature

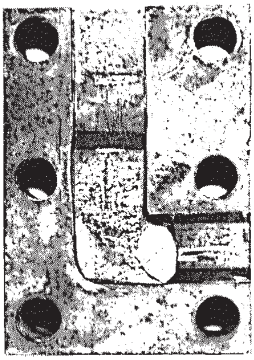
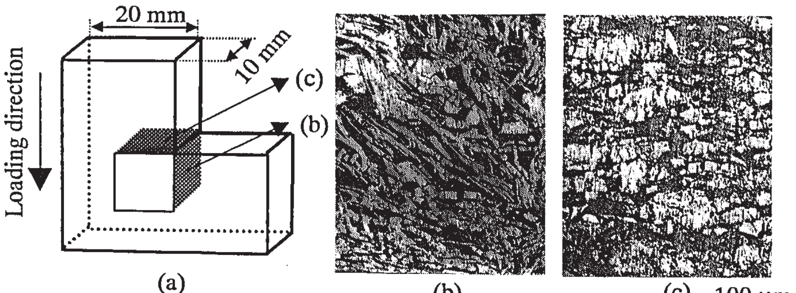
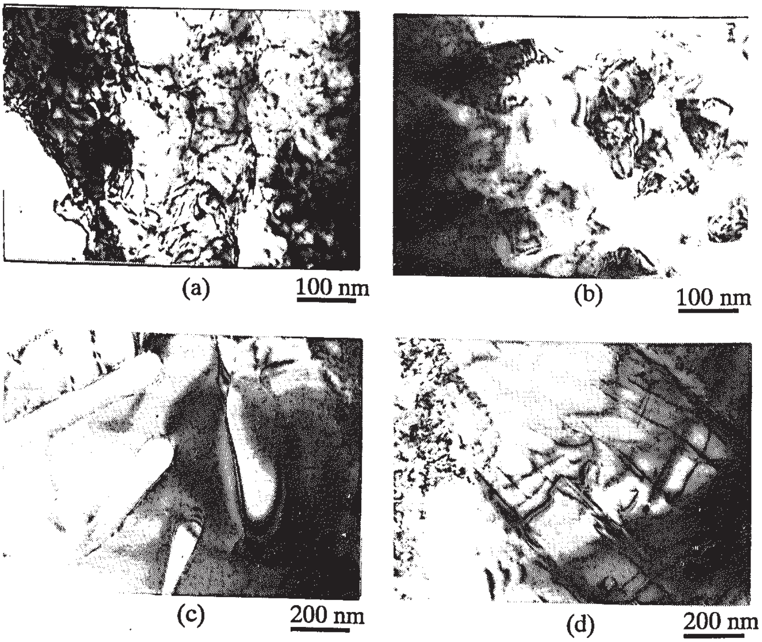


Fig.5 Photograph showing the change in shape of the Al-Ti-Mn alloy specimen by incomplete side extrusion.



(a) (b) (c) 100 μm

Fig. 6 Microstructures of the specimen shown in Fig.5. (a): schematic illustration of the sample subjected to observation. (b,c):optical micrographs taken from the are a indicated in (a).



(a) 100 nm (b) 100 nm (c) 200 nm (d) 200 nm

Fig.7 Transmission electron micrographs of the specimen corresponding to Fig.6(C).

strength, and thus even single (non-repeated) extrusion was not completed. Figure 5 shows the change in specimen shape caused by this incomplete extrusion. New set of dies is being designed by modifying the shape and the way of fixing. The microstructures of the specimen shown in Fig.5 were observed from two directions, as illustrated in Fig.6(a), and are shown in Fig.6(b) and (c). In comparison to the microstructure prior to the extrusion, *i.e.*, as-cast structure shown in Fig.4, second phase particles are confirmed to be slightly but significantly refined by the incomplete extrusion. Therefore, by increasing the reduction through this repeated working process using deformation-free dies and bolts, sufficient refinement of the second phase particles will be achieved.

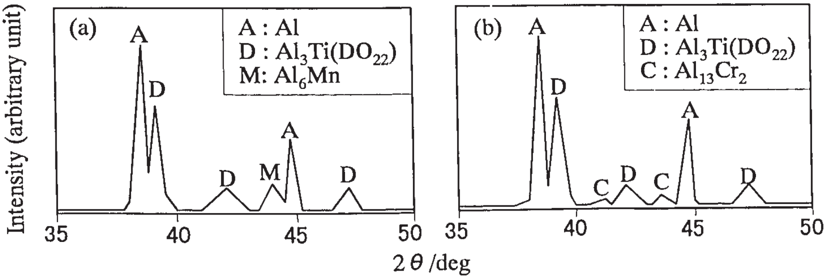


Fig.8 X-ray diffraction patterns obtained from as-cast ingots using CuK_α. (a): Al-Ti-Mn , (b): Al-Ti-Cr.

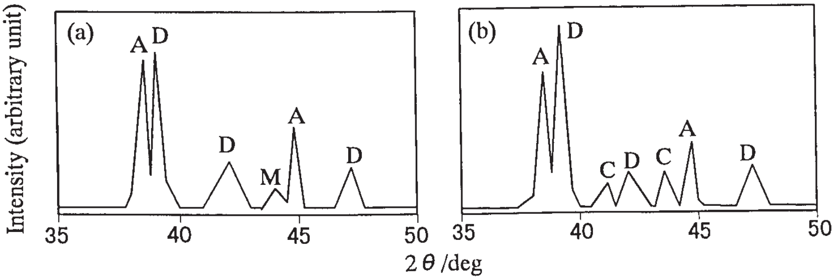


Fig.9 X-ray diffraction patterns from the specimens uniaxially compressed by 88% and then annealed at 650°C for 24h. (a): Al-Ti-Mn , (b): Al-Ti-Cr.

Figure 7(a)~(d) shows transmission electron micrographs of the incompletely side extruded Al-Ti-Mn alloy specimen corresponding to Fig.6(b). Microstructures consisting of high density of dislocations or of fine sub-grains remain in the matrix as shown in Fig.7(a) and (b), in spite of the extrusion temperature as high as 600°C. In contrast, generally no dislocation was found in the intermetallic phase as shown in Fig.7(c), although some intermetallic particles contain super lattice dislocations as shown in Fig.7(d).

To identify the second phase in the two alloys X-ray diffraction was carried out on the as-cast ingots. The results obtained are shown in Fig.8, indicating that Al_6Mn or $Al_{13}Cr_2$ and Al_3Ti (DO_{22}) phases are present together with aluminum matrix. In other words, both alloys contain two intermetallic phases but no $L1_2$ phase out of accord with the expectation mentioned earlier. Since the equilibrium is not always attained in as-cast conditions where metastable phases are frequently contained, $L1_2$ phase might be present in the equilibrium state. To examine this possibility, the specimens uniaxially compressed by 88% were annealed at 650°C for 24h and then subjected to X-ray diffraction. The resultant patterns are presented in Fig.9, showing that Al_6Mn or $Al_{13}Cr_2$ and Al_3Ti phases exist in addition to aluminum, which is qualitatively the same as those for the as-cast ingots shown in Fig.8. Comparing the peak height ratio of the intermetallic compound phases to the matrix phase in Fig.9 with that in Fig.8, relative amount of the intermetallic phases, especially of Al_3Ti phase, was found to be increased by the annealing. To produce $(Al,Mn)_3Ti$ or $(Al,Cr)_3Ti$ $L1_2$ phase, the amount of Al_6Mn or $Al_{13}Cr_2$ and Al_3Ti (DO_{22}) phases must decrease. Therefore, formation of the $L1_2$ phase can not be expected by further annealing. Hence, in spite of the initial expectation, it is concluded that addition of Mn or Cr to Al-10mol%Ti alloy does not bring about the occurrence of $L1_2$ phase.

4. Summary

In uniaxial compression, the second phase particles were refined more markedly as the reduction amount increased. Although repeated working became impossible since the bolts which fixed the dies were seriously deformed because of lack in high-temperature strength, significant refinement was attained in the side extrusion at 600℃. It was shown by means of X-ray diffraction that there is no $L1_2$ phase in either alloy, and that Al_6Mn or $Al_{13}Cr_2$ and Al_3Ti (DO_{22}) phases are present in addition to the matrix phase both in the as-cast and annealed states.

Acknowledgments

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