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Abstract. A commercial purity aluminum was highly deformed by the accumulative roll-bonding (ARB) process and subsequently annealed. The specimens having various grain size distributions were obtained. In case of the specimen ARB-processed with lubrication, the specimens with mean grain size larger than 3μm showed continuous yielding. On the other hand, in case of the specimen ARB-processed without lubrication, the specimens with mean grain size larger than 3μm showed discontinuous yielding. It suggests that appearance of the yield-drop phenomena can not be decided by the mean grain size. In order to consider effect of grain size distribution, the volume fraction of grains was summed from coarser grains, and the grain size when the summed volume fraction reached 70%, $d_{70\%}$ was estimated from the grain size distribution. it was found that $d_{70\%}$ of specimens which showed continuous yielding were larger than 8 μm while the specimens which showed discontinuous yielding were smaller than 6 μm, regardless of the lubrication condition in the ARB process. The result suggests that the appearance of the yield-drop phenomena depend on $d_{70\%}$.

1. Introduction

Ultrafine grained (UFG) materials having the mean grain size smaller than 1μm can be fabricated by severe plastic deformation (SPD) processes [1-4]. The UFG materials generally exhibit high strength. Additionally, the UFG materials sometimes show particular mechanical properties. For example, yield-drop phenomena similar to that conventionally observed in mild steels can appear in tensile tests of an UFG-Al [5]. The yield-drop phenomena appeared in the specimen having the mean grain size smaller than several micrometers. It is suggested that appearance of the yield-drop phenomena depend on the mean grain size of specimens.

By the way, polycrystalline materials sometimes show wide grain size distribution. The microscopic deformation of the materials having the wide grain size distribution might be inhomogeneous. Thus, it is expected that mechanical properties can not be decided by not only the mean grain size but also grain size distribution. However, effect of the grain size distribution on mechanical properties has not been clarified yet. Therefore, in the present paper, relationship between grain size distribution and yield drop phenomena in an UFG-Al was investigated.

2. Experimental procedures

A commercial pure aluminium (99% purity) was used in this study. The material was severely deformed by ARB process. Surfaces of two sheets of the materials with thickness of 1mm were degreased by acetone and wire-brushed. And then the sheets were stacked to be 2mm thick and roll-bonded by 50% reduction in one pass at ambient temperature. The roll peripheral speed was 2.0m/min using a two-high mill with 310 mm diameter rolls. This procedure defines one cycle of the ARB process. The roll-bonded sheet was cut in half-length, stacked and roll-bonded again.

It is known that microstructure of ARB-processed materials depends on the lubrication condition of the roll-bonding. In order to obtain the specimen with various grain size distributions, the ARB process was carried out with and without lubrication. To obtain same grain size, a number of the ARB cycle under unlubricated condition should be smaller than that under lubricated condition, because grain refinement with lubrication is faster than that without unlubrication. Thus, the ARB process was repeated up to 10 cycles with lubrication and up to 6 cycles without lubrication. Subsequently, the ARB-processed specimens were annealed in air at various temperatures ranging from 150°C to 300°C for 1.8ks.

The mechanical properties of the specimens were characterized by tensile test. Tensile specimens 5mm in gage width and 10 mm in gage length (1/5 of JIS-5 specimen) were tested at an initial strain rate of $8.3 \times 10^{-4}\ s^{-1}$. The tensile direction was parallel to the rolling direction (RD) of the sheets.

Microstructures of the ARB processed specimens and heat-treated specimens were characterized by EBSP measurement. The observed section was perpendicular to the transverse direction (TD) of the sheet. The observed plane was electro-polished in a 300ml HNO_3 + 700ml CH_3OH solution at -30°C and voltage of 15V. The EBSP measurements were carried out using the FE-SEM at 15kV. The step size of the EBSP scan was 50nm.

3. Results and discussion

The microstructure of the specimens ARB-processed and subsequently annealed were characterized by the EBSP measurement. The specimen ARB-processed by 10 cycles with lubrication (lubricated specimen) exhibited the lamellar boundary structure having the mean lamellar spacing of 220nm elongated to the RD. After annealing, the lamellar spacing increased with increasing the annealing temperature. The specimen showed comparatively equiaxed grain structure after annealing above 200°C. After annealing above 250°C, the specimen exhibited inhomogeneous microstructure which was composed of fine grains with grain size of several micrometers and coarse grains with several tens micrometers.

The specimen ARB-processed by 6cycles without lubrication (unlubricated specimen) also showed the lamellar boundary structure. The mean lamellar spacing was 290nm. Microstructure change of the specimen during the annealing was similar to that of the lubricated specimens.

Figure 1 shows the stress-strain curves for the specimen ARB-processed and subsequently annealed ((a) the specimen ARB-processed by 10 cycles with lubrication and (b) the specimen ARB-processed

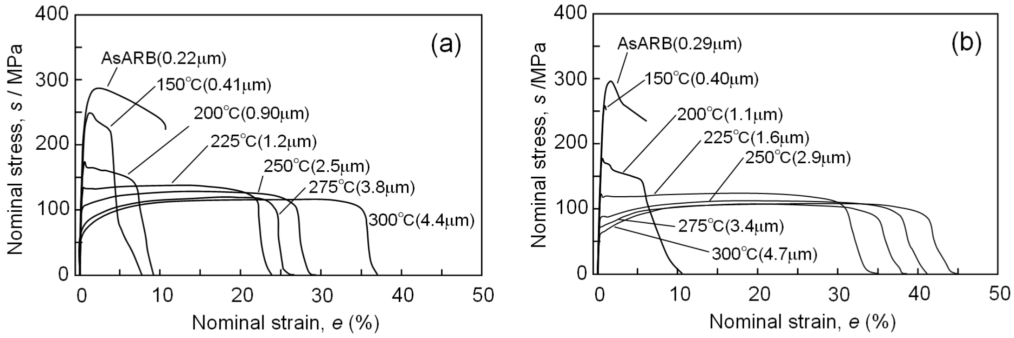

Figure 1. Results of the tensile test of the specimens ARB-processed and subsequently annealed. (a) The specimens ARB processed by 10 cycles with lubrication and (b) the specimens ARB-processed without lubrication

by 6 cycles without lubrication). The annealing temperatures and the mean grain thickness of each specimen are also shown in Fig. 1.

In the lubricated specimen (Fig.1(a)), the flow stress and total elongation decreased after annealing at 150°C. After annealing above 200°C, the total elongation increased and the flow stress decreased with increasing the annealing temperature. The specimens ARB-processed by 6 cycles without lubrication showed the same tendency of the specimen ARB-processed by 10 cycles with lubrication. Both specimens annealed at 200°C showed the yield-drop phenomena.

Figure 2 shows the stress-strain curves of (a) the lubricated specimens and (b) the unlubricated specimens annealed above 225°C in low tensile strain region. In Fig. 2(a), the lubricated specimens annealed at 225°C and 250°C show discontinuous yielding. The specimens annealed above 275°C show continuous yielding. On the other hand, in Fig. 2(b), the unlubricated specimens annealed between 225°C and 300°C show discontinuous yielding. In case of the lubricated specimen, the specimen having the mean grain size larger than 3μm show continuous yielding. However, in case of the unlubricated specimens, the specimens having the mean grain size larger 3μm show discontinuous yielding. The result suggests that the yielding behavior not only depend on the mean grain size.

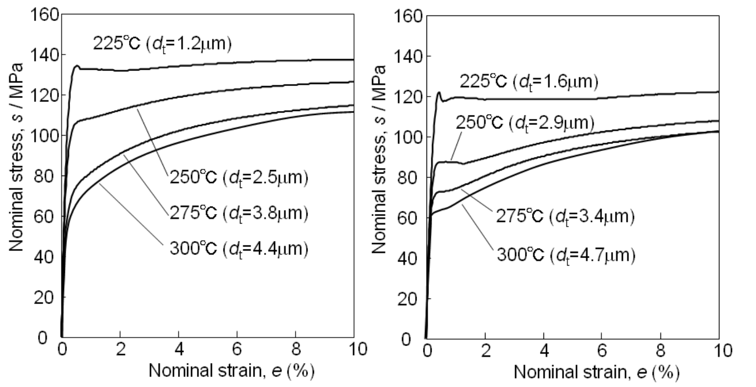

Figure 2. Stress-strain curves of (a) the lubricated specimens and (b) the unlubricated specimens annealed above 225°C in low tensile strain region.

Figure 3 shows the distribution of the grain size (in area) as semi-logarithmic graph for (a) the lubricated specimen annealed at 250°C, (b) lubricated specimen annealed at 275°C, (c) unlubricated specimen annealed at 275°C and (d) unlubricated specimen annealed at 300°C. In the figures, bar chats indicate the measured values obtained from the EBSP data and black lines indicated fitted lognormal distribution curves. The shapes of the grain size distribution for these specimens are similar. However, it seems that the fraction of grain size in area larger than 100 μm^2 of the lubricated specimen annealed at 275°C (Fig.3(b)) which showed continuous yielding is larger than that of other specimens (Fig.3 (a), (c) and (d)) which showed discontinuous yielding. It suggests that the yielding behavior depend on the grain size distribution.

By the way, when a polycrystalline material macroscopically yields, individual grains naturally deform. It is considered that individual grains microscopically yield before the material macroscopically yield. The yielding of the individual grains is called "micro-yielding". It is expected that the materials having the wide grain size distribution such as Fig.3 may deform non-uniformly and micro-yielding occur from coarser grains to finer ones. Regarding the relationship between micro-yielding and macroscopic yielding, yielding behavior of an IF steels having various grain size distributions has been reported [6]. In the report, it was suggested that polycrystalline materials including duplex-grained structure materials cause the macroscopic yielding when the grains of 70%-80% vol% are micro-yielded.

Assuming the micro-yielding occur form coarser grains to finer grains, yielding behavior may dependents on the mechanical properties of the grains of 70-80 vol% from coarse-sized grain.

Thus, the volume fraction of grains was summed from coarser grains, and the grain size when the summed volume fraction reached 70%, $d_{70\%}$ was estimated from the grain size distribution as a grain size parameter including an effect of the grain size distribution. $d_{70\%}$ estimated from Fig.3(a), (b), (c) and (d) were 5.1μm, 8.1μm, 4.8μm and 5.5μm, respectively. $d_{70\%}$ of Fig.3(b) which showed continuous yielding is larger than that of other specimens (Fig.3 (a), (c) and (d)) which showed discontinuous yielding. $d_{70\%}$ of the other specimens were also estimated from their grain size distributions. From the results, it was found that $d_{70\%}$ of specimens which showed continuous yielding were larger than 8 μm while the specimens which showed discontinuous yielding were smaller than 6 μm, regardless of the lubrication condition in the ARB process. The result suggests that the appearance of the yield-drop phenomena depend on $d_{70\%}$..

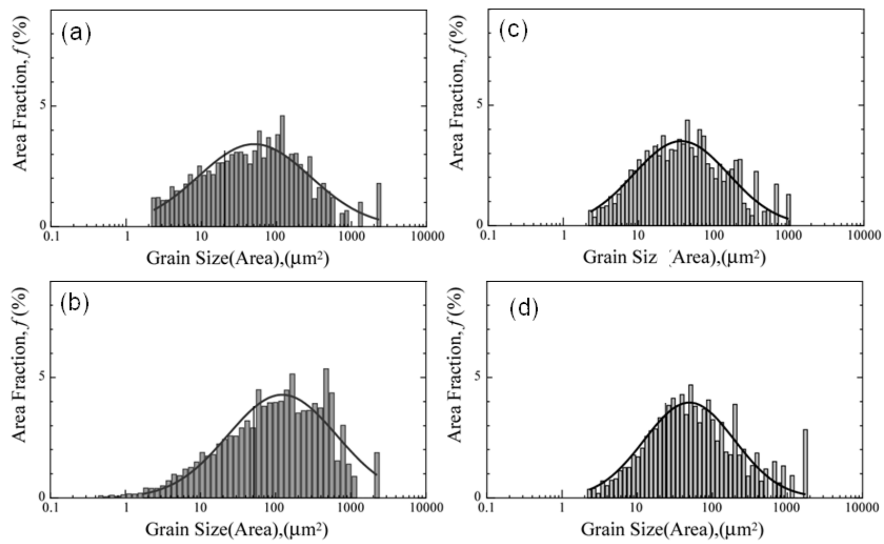

Figure 3. Distribution of the grain size (in area) as semi-logarithmic graph for (a) the lubricated specimen annealed at 250°C, (b) lubricated specimen annealed at 275°C, (c) unlubricated specimen annealed at 275°C and (d) unlubricated specimen annealed at 300°C. The bar chats show the measured value obtained from the EBSP data and black lines indicate fitted lognormal distribution curves.

4. Conclusions

In this paper, the mechanical properties of a commercial purity aluminum highly deformed by the accumulative roll-bonding (ARB) process and subsequently annealed were investigated. The main results are summarized below:

(1)From results of tensile tests, it was found that In case of the specimen ARB-processed by 10 cycles with lubrication, the specimen having the mean grain size larger than 3μm show continuous yielding. However, in case of the unlubricated specimens, the specimens having the mean grain size larger 3μm show discontinuous yielding. The result suggests that the appearance of the yield-drop phenomena can not be decided by the mean grain size.

(2) It was found that the grain size when the summed volume fraction from large grains reached 70%, $d_{70\%}$ of specimens which showed continuous yielding were larger than 8 μm while the specimens which showed discontinuous yielding were smaller than 6 μm, regardless of the lubrication condition in the ARB process. The result suggests that the appearance of the yield-drop phenomena depend on $d_{70\%}$.

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