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Structural Stability of AA3003 with Ultra-fine Grain Size by Accumulative Roll Bonding Process

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Abstract

The microstructure and mechanical properties of AA3003 alloy produced by accumulative roll bonding (ARB) and following annealing processes have been investigated. The average grain sizes of 250°C-ARB samples were reduced greatly from about 10.2μm initially to 700-800nm. The tensile strength of the ARB processed 3003 alloy (after 6 cycles) is considerably higher than that of the initial material, and about 1.5 times higher than that of commercially available fully-hardened (H18) 3003 alloy. The continuous recrystallization took place in the ARBed 3003 alloy with increasing the annealing time at 250 °C and increasing the annealing temperature to 275°C. While, discontinuous recrystallization began in some regions after 300 °C annealing, and nearly finished after 400 °C annealing. The Hall-Petch dependence was observed in the plot of microhardness versus $d^{-1/2}$ of the ARBed 3003 alloy, but its dependence slope was changed,

Keywords: Aluminum; Accumulative roll bonding; Ultra-fine grain, Structural stability.

1. INTRODUCTION

One of novel intense straining processes for bulk materials using rolling deformation, named accumulative roll bonding (ARB), was developed recently[1-3]. The ARB process has been successfully applied to AA1100[1], AA 5083[2], AA8011[4] and Ti-added interstitial free steel[2,3]. Most of several cycle ARB processed materials have structures with sub-micron grains and show very high strength at ambient temperature[1-3]. However, ARBed materials have the low tensile ductility at ambient temperature. This is an inherent limit for their practical application as other intense plastic straining materials. After ARB process, the specimens accumulated considerable residual stress induced by high dislocation density inside materials. The heat treatment for relieving residual stress is necessary for their practical uses. In order to take the advantages of ultra fine grain (UFG) materials including the mechanical superiority and high formability, UFG structure should be maintained stable under either heat processing conditions or the service conditions[5]. AA3003 alloy has been widely used for moderate strength application requiring good workability. The purpose of the present study is to clarify the nature of microstructural stability in the ARBed 3003 alloy at different annealing condition.

2. EXPERIMENTAL PROCEDURES

A commercial AA3003 alloy (Al-0.286Si-0.560Fe-0.118Cu-1.039Mn-0.046Mg-0.006Cr-0.031Ti in mass %) was used in this study. Fully annealed 3003 sheets with the initial grain size of 10.2μm were prepared. Pieces were cut from the initial plate to dimension of $1\times30\times300$ mm^3. Then the interface between two sheets was degreased by acetone and wire-brushed. After that, the two sheets were layered to make brushed surface in contact and fixed each other closely by wires. They were held in an electrical furnace at 200°C or 250 °C for 5 minutes, and then rolled. ARB process was conducted under the conditions that the reduction in thickness per cycle was 50% (equivalent strain of 0.8). After the ARB process was repeated up to 8 cycles with an equivalent true plastic strain of 6.4[6], the deformed sheets were annealed in oil bath or salt bath at temperature of 250-400°C for 1hour, or at 250 °C for various times. The values of Hv represent the average of seven separate measurements taken at randomly selected points using a load of 200g for 15s. Tensile tests were conducted on the specimens having 25mm gauge length at room temperature on a standard universal testing machine at the strain rate of $8.3\times10^{-4}s^{-1}$. The optical examination of the samples was conducted under conditions of polarized light. Specimens were also examined using a JEM-2000 FX II transmission electron microscope (TEM) operating at 200 kV. Selected area electron diffraction (SAD) patterns were taken from regions having diameters of 3 μm. Measurements of the grain size were made directly from the TEM photomicrographs or optical micrographs using an Image & Microscopy Program. At least 100 different grains in every condition were chosen.

3. RESULTS AND DISCUSSION

3.1 Effect of ARB processing temperature on mechanical properties

Fig.1 shows that the Vickers microhardness of 200 °C-ARB samples increased with increasing the cycles up to the 4 cycles. Meanwhile, the Vickers microhardness of the 250 °C-ARB samples increased with increasing the cycles to the 5 cycles and then kept a constant value.

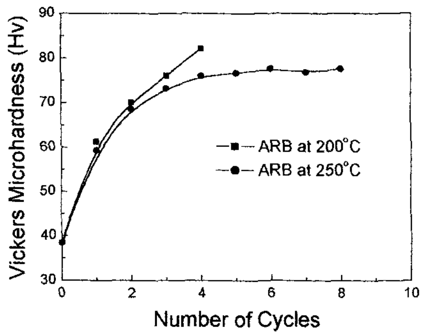

Fig. 1. Variation of Vickers microhardness of 3003 alloy with increasing the cycles of ARB at 200 °C and 250 °C.

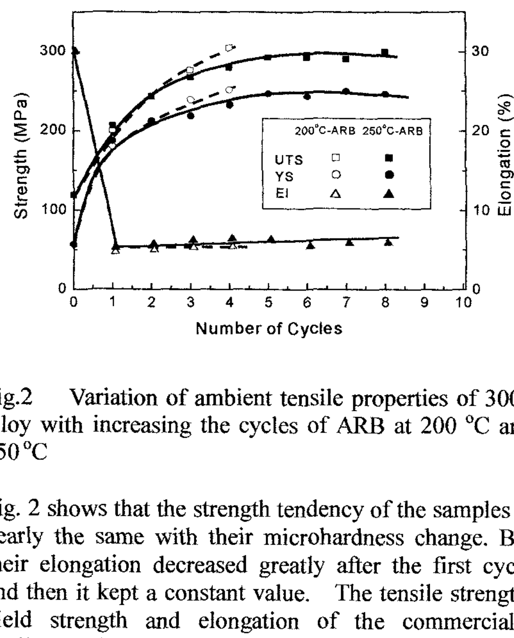

Fig.2 Variation of ambient tensile properties of 3003 alloy with increasing the cycles of ARB at 200 °C and 250 °C

Fig. 2 shows that the strength tendency of the samples is nearly the same with their microhardness change. But their elongation decreased greatly after the first cycle and then it kept a constant value. The tensile strength, yield strength and elongation of the commercially available full-hardened 3003 alloy (H18 temper) at room temperature were reported as 200MPa, 185MPa and 10%, respectively. So the tensile strength of the ARB processed 3003 alloy (after 6 cycles) is about 1.5 times higher than that of the H18 processed 3003 alloy.

3.2 Microstructure evolution of ARBed 3003 alloy

The average grain (or subgrain) sizes of 250°C-ARB samples were reduced greatly from about 10.2μm initially to about 770nm after 1 cycle of ARB. After 6 cycles, all the SAD patterns show the presence of high angle grain boundaries due to the distribution of diffraction spots around circles. The average grain (or subgrain) size of 200 °C-ARB samples after 200 °C/5min annealing was about 760nm, nearly the same with that of as-ARB samples. However, the average grain (or subgrain) size of 250 °C-ARB samples after 250 °C/5min annealing was about 900nm, slightly bigger than that of as-ARB samples. In order to proceed ARB process continually, the static recovery which results in grains (or subgrains) growing should take place in some extent during annealing period. For 3003 alloy, the annealing condition 250°C/5min is suitable for the ARB process.

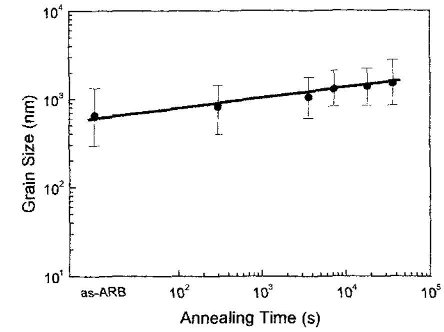

Fig. 3. Variation of grain sizes of the ARBed 3003 alloy with increasing the annealing time at 250 °C

Fig.3 gives the correspondent changing tendency of the grain size measured in the TEM micrographs of the ARBed samples after annealing at 250 °C for various times. It shows that the average grain size of the ARBed 3003 alloy increased homogeneously and gradually with increasing the annealing times. Fig. 4 shows the variation of grain sizes of the ARBed samples after annealing at different temperature for 1hour. The fine grains grew gradually from as-fabricated state (~0.65 μm) to 275 °C-annealing state (1.69 μm). After that, bimodal distribution of the grain size appeared due to the coexistence of fine grains and coarse grains. After 400°C annealing, all the fine grains were transferred to coarse grains with the average grain size of 20.5 μm.

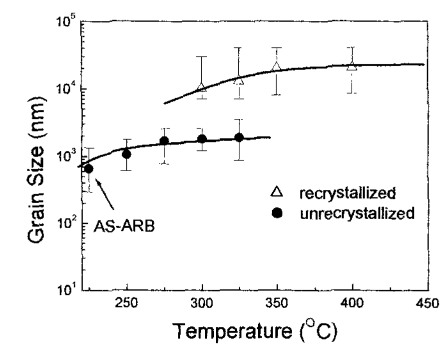

Fig.4. Variation of grain sizes of the ARBed 3003 alloy with increasing the annealing temperature for 1hour.

3.3 Structure stability of ARBed 3003 alloy

Fig. 5 presents the stress-strain curves of the samples at different conditions. For the as-ARBed material, strain hardening behavior also happened, but it was in a narrow strain range (~2%), then the material tended to break. So it possessed a high strength and low ductility

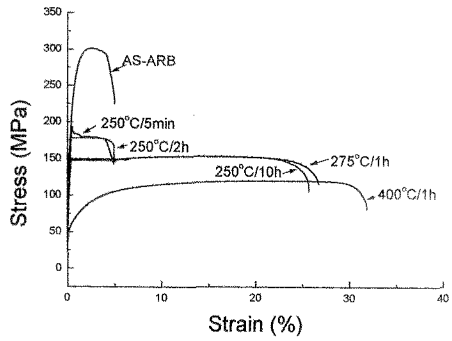

Fig.5 Stress-strain curves of ARBed 3003 alloy at different conditions.

mainly due to high dislocation density and small grain size. An additional annealing (250-275°C) changed the GB condition of this material from deformed state to a stable state, and grains grew gradually and continuously. Therefore, the mechanical behaviors of 250-275 °C annealing samples were very different with those of conventional materials. They exhibited a narrow (or no) strain-hardening region followed by stable flow at a very low strain hardening rate. Especially for the 250°C/10h and 275°C/1h sample, this stable process is very long, and led to a high ductility (>20%). But their yield strength were about three times of fully recrystallized material (400°C/1hour). It is recognized in this study that ultra fine grain structure can evolve homogeneously and gradually during the annealing process below the annealing temperature of 275 °C of severely deformed metals. This phenomenon was referred variously to as "extended recovery", " in-situ recrystallization", and "continuous recrystallization" (the most widely used) [7,8]. From above study, it can be seen that the UFG ($\leq$1 μ m) formed in the highly deformed 3003 alloy can be stable until annealing at 250 °C for 1h, and the fine grains (<2 μ m) can be stable until annealing at 275°C for 1hour by a large number of fine dispersoids such as Al_6Mn. Therefore, grain structure in the ARBed 3003 alloys after intense plastic strain is reasonably stable.

3.4 The change of microhardness versus grain size

Fig. 6 presents the microhardness versus $d^{-1/2}$ (grain size) dependence characteristic of the ARBed 3003 alloy. The Hall-Petch dependence was observed after annealing,
but the slope of this dependence was changed. The curve has been divided into three parts, marked by A, B, C zone, respectively. The microhardness decreased significantly with increasing grain size before annealing for 5 min at 250°C (A zone), and annealing after 275 °C for 1 hour (C zone). With further increasing the annealing time up to 10h at 250 °C, and increasing annealing temperature to 275°C (B zone), the grains grew continuously in this zone (continuous recrystallization process). So the Hall-Petch dependence kept a constant value during this period.

Fig. 7a shows a typical feature of deformed GBs with the occurrence of extinction contours inside grains, which are linked with elastic stresses originated from

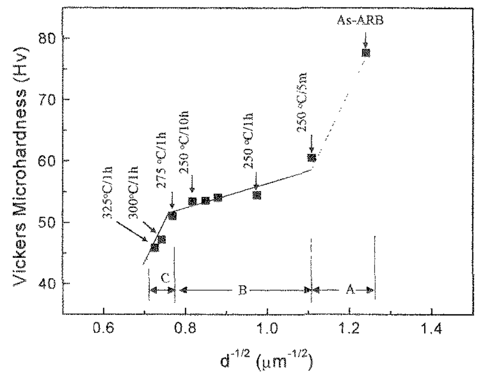

Fig.6 Microhardness versus the mean grain size in the ARBed 3003 alloy

GBs. In this state, the hardness is very high because of high dislocation density and large strain field. After annealing at 250 °C for 5 minutes, the usual equilibrium GBs with a typical banded contrast and relatively few dislocations in the grains formed in most grains of the materials, as shown in Fig.7b. After annealing at 250 °C for 1hour, the whole volume of the material was filled with stable straightened GBs and low dislocation density in grain[Fig.7c]. Therefore, the significant decrease of the microhardness during the A zone in Fig.6, is mainly attributed to the transformation from deformed GBs to the stable straightened GBs by annihilation of stress field, and the grain growth has only a little effect on it.

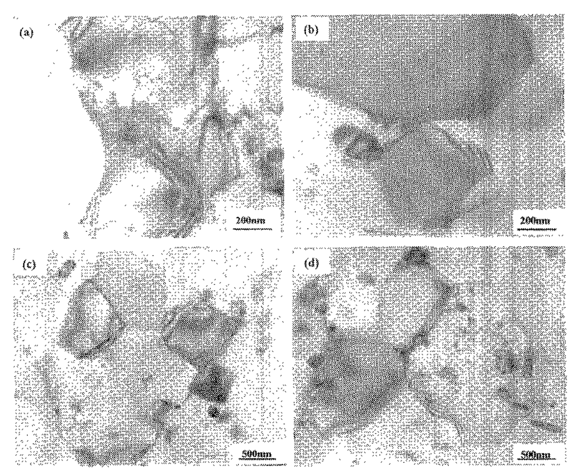

Fig.7 Higher magnification of TEM micrographs of the ARBed 3003 alloy after annealing at different conditions. (a) as-ARB, (b) 250 °C/5m, (c) 250 °C/10h, (d) 325 °C/1h.

4. CONCLUSIONS

(1) The ultra-fine grained 3003 alloy having mean grain size of 700-800 nm was successfully produced by the ARB process. The annealing condition 250°C/5min is suitable for the ARB process.

(2).The strength of 250 °C-ARB samples increased with increasing the cycles up to 5 cycles and then kept a constant value. And their elongation decreased greatly after the first cycle and then it kept nearly no change. The tensile strength of the ARB processed 3003 alloy

(after 6 cycles) is about 1.5 times higher than that of commercially available fully-hardened (H18) 3003 alloy.
(3) The grain size of the ARBed 3003 alloy increased homogeneously and gradually with increasing the annealing time at 250 °C and increasing the annealing temperature up to 275 °C for 1hour. These reactions were referred to as continuous recrystallization reactions. While, the process that coarse grains grew suddenly in some regions after 300 °C annealing for 1 hour, was referred as discontinuous recrystallization reactions.
(4) The Hall-Petch dependence was observed in the plot of microhardness versus $d^{-1/2}$ of the ARBed 3003 alloy, but its dependence slope of this alloy was changed due to the internal stress field and grain size.
(5) The UFG ($\leq$ 1μm) formed in the ARBed 3003 alloy can be stable until annealing at 250 °C for 1hour, and the fine grains (<2μm) can be stable until annealing at 275°C for 1hour. Therefore, grain structure formed in the ARBed 3003 alloys after intense plastic strain is reasonably stable.

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