Mechanical properties and microstructure evolution of aluminum alloy tubes with normal gradient grains under biaxial stress

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Abstract
Grain size gradient materials are a type of new structural material with the advantages of both coarse and fine grains. To study the effect of normal gradient grain on the mechanical properties and microstructure of aluminum alloy tubes during hydroforming, the normal gradient grain distribution of the outer fine and inner coarse grains was obtained using spinning and annealing methods, and the biaxial stress was determined using hydraulic bulging experiments. Gradient grain tubes with thicknesses of 300, 475, and 575 μm were obtained through spinning and annealing at different temperatures; the tensile strengths of the tubes were 79, 89, and 109 MPa; the maximum expansion rates were 18%, 17%, and 10%; and the work-hardening indices were 0.19, 0.20, and 0.17, respectively, under biaxial stress. With an increase in the refined grain area, the density of the low-angular grain boundaries (LAGBs) increased and the chance of stitching dislocation increased in the process of intragranular deformation, causing an increase in strength. However, the increase in the refined thickness weakened the compatible deformation due to a reduction in the number of large grains, leading to a decrease in plasticity. The obtained quantitative relationship between gradient grain and strength/ductility can be applied to guide production of aluminum alloy tubular parts in the aerospace and automotive industries.

Keywords Grain grain · Biaxial stress · Aluminum alloy · Microstructure

1 Introduction
To reduce the energy consumption of vehicles, conserve resources, and achieve higher energy efficiency in general, there is an urgent need for lightweight structural parts in the aerospace and automotive fields [1]. Hydroformed aluminum alloy tubular parts can simultaneously meet the needs of lightweight and structurally lightweight aerospace parts [2, 3], and are often used in densely arranged and structurally complex systems of tubes [4, 5]. However, the strength of aluminum alloy is relatively low compared with that of traditional steel parts [6]. Controlling plastic deformation by cold rolling, cold drawing, or thermo-mechanical treatment is an effective way to realize high-strength metal materials [7, 8]. However, work hardening occurs during the deformation process, which increases the strength of the material while reducing the ductility [9, 10], resulting in a decrease in the formability of the tube. Ultra-fine or even nano-sized grains are produced in the metal materials [11] when they are subjected to severe plastic deformation (SPD) [12], which yields limited ductility; it is difficult in this case to achieve good formability of the tube.

Simultaneously improving the strength and ductility of metal materials is a popular research subject for light alloys [13], and the gradient micron structure is generally considered to have the greatest potential for improving materials [14]. The microstructure of materials with gradient grains includes a gradual transition from coarse grains to fine grains or even nano-sized grains. Gradient grain materials can be obtained through gradient plastic deformation methods, such as surface mechanical attrition treatment (SMAT) [15], surface mechanical grinding treatment (SMGT) [16], or shot peening (SP) [17]. These methods are suitable for tubes, bars, and plates, and have been applied to titanium [18], magnesium [19], and aluminum alloy [20].
Many experiments have confirmed the excellent strength and ductility of the gradient grain materials. The tensile results at room temperature show that the yield strength of a copper bar with a nano-gradient grain structure is twice that of a coarse-grained copper bar, and the plasticity is the same as that of a coarse-grained copper bar [14]. In a study on gradient grain pure iron wire formed by cold drawing, the tensile strength increased from 607 to 808 MPa, and the elongation increased from 6.7 to 11.5% [21]. AZ31 magnesium alloy sheets with a nano-gradient obtained through SMAT have significantly improved the surface microhardness, tensile strength, and matrix plasticity. The orientation angle of the non-basal plane of the fine-grained layer is increased, which reduces the anisotropy of the mechanical properties of magnesium alloy [22]. A sample was fabricated by laser heat treating the surface of a cold-rolled TWIP steel plate, yielding an almost linear gradient in grain size; the sample had improved tensile strength (674 MPa), and a plasticity with approximately the same total elongation (60.7%) as that of the coarse grain sample (61.0%) [23].

Gradient grain materials have a special plastic deformation mechanism [24]. The increase in strength is mainly achieved by mechanically driven grain boundary migration; the grain size increases as the deformation increases [25]. The grain size increase in the fine-grained region is limited, which results in a limited decrease in strength and hardness [26], while the coarse-grained region gradually hardens to strengthen the gradient grain material as a whole [27]. The plasticity of gradient grain materials primarily depends on the proportion of coarse grains. The coarse grains of the matrix have excellent plastic deformability and contribute to increasing plastic strain and work-hardening capacity during the deformation process [28]. The fine-grained structure delays the strain localization and crack initiation of the material [29, 30]; thus, gradient grain materials exhibit better plastic deformation coordination.

The above conclusions were obtained under uniaxial stress conditions, whereas hydroformed tubular parts are formed under complex stress conditions. Therefore, it is more urgent to study the influence of biaxial stress on gradient grain tubes. The stress state of the tube during hydroforming is complicated, as most areas are in a biaxial stress state. Conventional tube mechanical property test methods—such as uniaxial tensile or circumferential tensile tests—can only reveal the mechanical properties of the tube in the axial or circumferential directions but cannot show the comprehensive mechanical properties of the tube during hydroforming. Consequently, the tube bulging test method, which involves passing a high-pressure liquid into the tube to make the tube bulge under the action of biaxial stress, is used. The obtained stress–strain relationship directly describes the hydroforming ability of the tube [31]. In this study, aluminum alloy tubes with a normal gradient grain are obtained via spinning heat treatment. The mechanical properties under biaxial stress are measured by a hydraulic bulging test. The effect of the normal gradient grain on the mechanical properties under biaxial stress is studied, and the microscopic evolution of the normal gradient grain structure under biaxial stress is revealed. These findings provide a theoretical basis for the application of aluminum alloy tubes in the hydroforming of tubular parts in the aerospace field.

2 Experimental procedure

2.1 Materials and bulging process

The initial material was a 6063-T4 aluminum alloy extruded tube. Figure 1a shows the spinning diagram, where a large plastic deformation was carried out on the outer surface of the tube through a double roller horizontal spinning machine at room temperature. The wall thickness of the tube was reduced from 3.5 to 2.0 mm with a 42.86% reduction in the thickness; the outer diameter was reduced from 78 to 75 mm through six-pass spinning. The axial direction of the tube is denoted as AD; the tangential direction is denoted as TD, and the normal direction is denoted as ND. Figure 1b shows a schematic diagram of the annealing process. To obtain tubes with different normal gradient grains of outer fine grains and inner coarse grains along the radial direction, spinning tubes were annealed at 350, 400, and 450 °C for 1 h; taken out of the furnace; cooled to 260 °C; and then air-cooled. Figure 1c shows a diagram of the tube bulging process. The tube was fixed using an annular fixing block and sealed with a rigid punch at each end. A high-pressure fluid was then passed through the block to bulge the tube until it bursts.

2.2 Measurement of mechanical properties

During the bulging process, the instantaneous internal pressure p and the bulging height h were measured using a sensor, and the equivalent stress–strain curve of the tube bulging could be obtained (by measuring the wall thickness t0 and tp before and after bulging) [32]. The yield strength, tensile strength, expansion, K value, and n value of the tube could be obtained from the stress–strain curve of the tube bulging process. We set the length–diameter ratio L/D of the bulged length to 1.5, and calculated the bulged length L to be 117 mm.

2.3 Microstructural characterization

The microstructure was acquired through electron backscatter diffraction (EBSD) using a Quanta 200 FEG operated at 20 kV with a 5.5-μm step size. Specimens for the EBSD
analysis were prepared by mechanical grinding and electropolishing in an electrolyte containing 20% perchloric acid in alcohol at a temperature of approximately $-20^\circ$C and a voltage of 25 V for 50 s.

3 Results and discussion

3.1 Microstructure characterization

Figure 2 shows the EBSD results at the ND–AD section of the initial tube (6063-T4). The distribution of grain orientation and grain boundaries are shown in Fig. 1a; different grain orientations are denoted by different surface colors, and the low-angular grain boundaries (LAGBs, 2–15°) and high-angular grain boundaries (HAGBs, >15°) are represented by white and black lines, respectively. The inverse pole figure (IPF), which expresses the grain orientation in another form, is shown in Fig. 2b. This shows the grain orientations: most are close to (001), fewer are close to (111), and the least are close to (101). The grains indexed by EBSD are the regions enclosed by HAGBs. Typically, HAGBs represent grain boundaries, while LAGBs represent unstable dislocations and substructures [33]. The grain boundaries
(GBs) of the initial tube are mostly HAGBs, accounting for about 78% of all boundaries. The morphology is nearly equiaxed, the grains are nearly uniformly distributed in the ND direction, and the average grain size is approximately 150 μm.

Figure 3 shows the EBSD results at the ND–AD section of the grain gradient tubes obtained by spinning and annealing at different temperatures. Figure 3a–c show the distribution of grain orientation and grain boundaries, and Fig. 3d–f show the IPF to express the grain orientation in another form. The proportions of HAGBs at the gradient microstructure were 91.3%, 81.5%, and 91.8% for annealing temperatures of 450, 400, and 350 °C, respectively; both exceed the initial tube proportion of 78%. The microstructure of the tubes obtained by spinning and annealing is nearly an isometric grain structure, and exhibits a gradient distribution of grain size at the ND–AD section from the surface to the core of the tube. The distribution of grain orientation of the spinning-annealing tube is dispersed compared with that of the initial tube (6063-T4), and the microstructure obtained through annealing at 350 °C is more dispersed than the other two, in terms of both grain orientation distribution and IPFs. Thus, a lower annealing temperature may make it easier to achieve dispersed grain orientations.
The ND direction was defined as the positive x-axis, the a point on the outer surface of the tube was defined as the origin, and the mean grain size was calculated by the number of grains on each equidiameter plane, as shown on the y-axis of Fig. 3. Figure 4 shows the grain size distributions along the thickness direction at the ND–AD section of the initial and grain gradient tubes. The mean grain size of the initial tube was between 55 and 10 μm, and the distribution was relatively uniform. The fine-grained region was designated as the area in which the mean grain size was less than that of the initial tube along the ND. The refined thicknesses of the grain gradient tubes obtained by spinning and annealing at 450 °C, 400 °C, and 350 °C were 300 μm, 475 μm, and 575 μm, respectively. Furthermore, we observed the grain size outside the fine-grained region of the microstructure after spinning and annealed at different temperatures. The grain size for the tube obtained by annealing at 450 °C is shown in Fig. 4 as a red curve with circles; after a distance of approximately 600 μm to the outer surface, it is higher than that for the initial tube. The grain size for the tube obtained by annealing at 400 °C is shown as a blue curve with triangles; after a distance of approximately 550 μm to the outer surface, it is approximately equal to the initial tube. The grain size for the tube obtained by annealing at 350 °C is shown as a green curve with five-pointed stars, which is significantly below that of the initial tube. Thus, fine grains were more easily obtained using lower annealing temperatures on the microstructure after the same spinning deformation. Because there was high distortion energy caused by the appearance of unstable dislocations and substructures in the deformed microstructure, especially in the form of SPD such as spinning [34], the equiaxed grains formed at a lower temperature. As the temperature or holding time increased during heat treatment, grain growth occurred, and the larger grains grew even larger with the merging of smaller grains [35].

3.2 Mechanical properties under biaxial stress

Bulging experiments were performed using tubes with normal gradient grain. Figure 5 shows a photograph of the bulged tubes. The maximum expansion rate of the bulging tube was measured—the gradient grain tubes with refined thicknesses of 300, 475, and 575 μm had maximum expansion rates of 10%, 17%, and 18%, respectively. The larger the
refinement thickness of the gradient grain tube, the poorer the plasticity and the lower the expansion rate. The true stress–strain curves and mechanical properties under the biaxial stress state were obtained through tube bulging experiments. Figure 6a shows the true stress–strain curves, and Fig. 6b shows the mechanical properties of the tubes with different normal gradient grains. The gradient grain tubes with refined thicknesses of 300, 475, and 575 µm were compared in terms of their mechanical properties. Figure 7 shows the kernel average misorientation (KAM) at the ND–AD sections of the tubes with different normal gradient grains. The KAM images illustrate the distribution of misorientations in the tubes, which can be used to evaluate the grain refinement and its impact on the mechanical properties. The KAM images were obtained using X-ray diffraction and electron backscatter diffraction techniques. The KAM values were calculated using the Otsuka algorithm, which is commonly used to quantitatively analyze the degree of misorientation in materials.
575 μm exhibited yield strengths of 60, 68, and 86 MPa; tensile strengths of 79, 89, and 109 MPa; and work-hardening indexes of 0.19, 0.20, and 0.17, respectively. For a normal gradient grain tube with fine outside and coarse inside grains, as the refined thickness increased, the strength of the tube increased, the work-hardening index decreased, and the ductility decreased. The strength of the tube with a refined thickness of 300 μm was too low, and the maximum expansion rate of the tube with a refined thickness of 575 μm was only 10%, which was too poor in terms of plasticity. However, the tube with a refined thickness of 475 μm exhibited better strength and plasticity.

### 3.3 Microstructure evolution

Figure 7 shows the kernel average misorientation (KAM) of tubes with different normal gradient grains. Figure 7a–c show the KAM distributions of the microstructure before bulging (ε = 0). Figure 7d–f show the KAM distributions at the area with an equivalent strain of 12% (ε = 0.12). Different colors represent the different KAM values. The larger the KAM value, the greater the degree of local deformation. The blue area represents the smallest degree of plastic deformation, where no plastic deformation occurs. The green area represents less plastic deformation, the yellow area represents a greater degree of plastic deformation, and the red area represents the greatest degree of plastic deformation. The blue areas of the KAM exceeded 90%, indicating that there was little plastic deformation in the microstructure before the bulging shown in Fig. 7a–c. At the area that bulged to an equivalent strain of 12%, the KAM fractions are listed in Table 1, while the KAM value at the deformed microstructure (ε = 0.12) at the ND–AD section of tubes with different normal grain gradients are shown in Fig. 7d–f. In each figure, the KAM values of the refined grain areas were higher than those of the other areas. Comparing Fig. 7d–f, higher refined thicknesses corresponded to higher KAM values. This shows better uniform deformability in larger grains, and more concentrated deformation in small grains in the refined grain areas. The degree of deformation was most uniform for the tube with a refined thickness of 300 μm during bulging. However, the degree of deformation of the tube with a refined thickness of 475 μm was relatively uniform. The degree of deformation was concentrated for the tube with a refined thickness of 575 μm, and the compatible deformability was poor—that is, the greater the refined thickness, the worse the plasticity.

Observing the change in the number of grain boundaries in Fig. 7, the black lines represent the HAGBs (15°–180°)—that is, the grain boundaries—and the white lines represent LAGBs (2°–15°)—that is, the intragranular dislocations. Figure 8 shows the misorientation at the region where the strain is 12% for tubes with different normal gradient grains through a statistical

| Area           | KAM             | Refined thickness |
|----------------|-----------------|-------------------|
|                |                 | 300 μm | 475 μm | 575 μm |
| Refined grain area | 2–3 (in yellow) | 0.143 | 0.207 | 0.211 |
|                 | 3–4 (in orange) | 0.029 | 0.047 | 0.086 |
|                 | 4–5 (in red)    | 0.007 | 0.012 | 0.083 |
| Other area      | 2–3 (in yellow) | 0.133 | 0.123 | 0.143 |
|                 | 3–4 (in orange) | 0.028 | 0.025 | 0.037 |
|                 | 4–5 (in red)    | 0.009 | 0.005 | 0.011 |

Fig. 8  Misorientation of tubes with different normal grain gradients (ε = 0.12)
4 Conclusion

Different normal gradient grain distributions were obtained through spinning heat treatment; furthermore, the mechanical properties under biaxial stress were measured using a hydraulic bulging test, and the microstructure was investigated. The following conclusions were reached:

1) The microstructure was nearly an isometric grain structure, and exhibited a gradient distribution of grain size at the ND–AD section from the surface to the core of the tube, when tubes were processed by spinning to achieve a 42.86% reduction in the thickness and annealing at 450, 400, and 350 °C for 1 h. The refined thicknesses of the tube with the normal gradient grain were 300, 475, and 575 μm, respectively. The grain orientation changed from a concentrated (001) orientation in the initial tube to a dispersed distribution after spinning and annealing. Lower annealing temperatures may be more conducive to obtaining dispersed grain orientations.

2) The tensile strengths were 79, 89, and 109 MPa, maximum expansion rates were 18%, 17%, and 10%, and work-hardening indices were 0.19, 0.20, and 0.17 for gradient tubes with the refined thicknesses of 300, 475, and 575 μm, respectively. An increase in the refined thickness of the gradient grain is beneficial for the strength of the tube, but results in lower plasticity.

3) The KAM diagram shows that the refined thickness increased and the concentrated deformation increased, leading to poor plasticity. The misorientation diagram shows that a larger refinement thickness increases the proportion of LAGBs in the deformed microstructure; the more opportunities for pinning dislocations during intragranular deformation, the higher the strength of the tube.

Author contribution Yang Cai conceptualized, planned, and carried out the experiments, and prepared and edited the original draft. Xiaosong Wang contributed to the analysis and interpretation of results, supervised, and critically reviewed the research and manuscript.

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Declarations

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Consent to participate Not applicable. The article involves no studies on humans.

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Competing interests The authors declare no competing interests.

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