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STRUCTURAL STUDIES ON THE EVOLUTION OF TEXTURE IN HEAVILY WIRE DRAWN AND SUBSEQUENTLY ANNEALED PURE Al METAL

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Abstract

Conventional transmission electron microscopy and x-ray diffraction techniques have been applied to study the microstructure of heavily wire-drawn and annealed samples of pure Al metal. Samples wire drawn (60% of the original value) have been found to be comprised of columnar grains with heavily distorted crystal structure and to exhibit a strong <111> texture. Thus drawn samples after annealing appear free of columnar granular structure and show re-crystallized grains. Electron diffraction studies from thus re-crystallized grains are presented to evaluate the resulting annealing texture.

Introduction

Metals and dilute alloys produced by various methods such as casting usually are polycrystalline in the sense that the constituting grains in such materials assume random crystal orientation. If such metals and alloys are subjected to a metal forming process, the constituting grains of the materials undergo plastic deformation, and develop some degree of non-randomness in crystal orientation This non randomness of crystal orientations of grains in the deformed material is known as deformation texture. The type of deformation texture depends upon the type of material and metal forming, In cold-drawn wires of Al that has the f.c.c. structure, the observed deformation textures are essentially made up of an alliance in variable proportion of two components fibers <001> and <111> [1]. Upon annealing, new crystals form and grow preferentially on the textured grains of deformed materials. The recrystallized grains in the annealed samples also revealed some degrees of non-randomness in crystal orientation, and develop annealing texture. Heavily drawn Al wires with a single fiber texture tend to retain their deformation texture upon recrystallization [2].

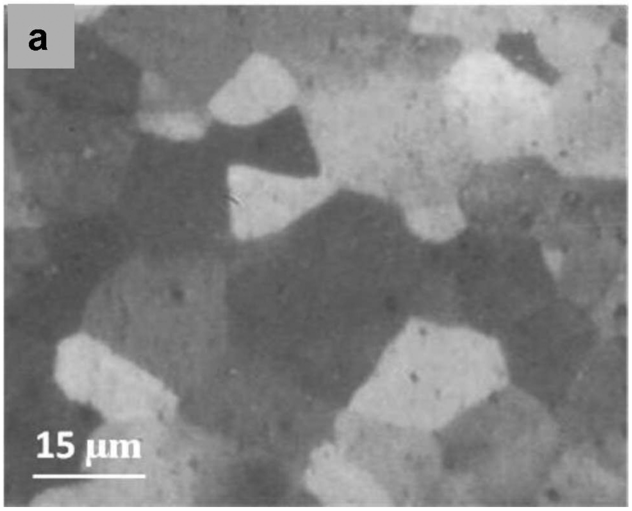
The <111>, <100> duplex fiber texture found in wire drawn aluminium has been originally explained mainly by two schools of thought. First of which is elaborated by Ahlborn and Wassermann [3], suggests that <111> is the true end texture resulted entirely by slip and that <100> results from recrystallization during deformation. Other school of thought is discussed by by Cullity [4], suggests that during wire drawing some portion of materials that is developed as (111) fiber texture is transformed to (115) by deformation twinning and this orientation presumably rotates to (100) with further deformation. However, no evidence is available to indicate preference for either school of thought.. The purpose of this work is to obtain more understanding in relation to structural information that support the development of the <111>, <100> duplex fiber texturing in cold drawn Al. This paper describes the various crystal and micro structural aspects of the formation fiber texture in pure Al during wire drawing, and subsequently annealing.

Experimental Procedures

Material used in the present study is a polycrystalline Al (with 99.999% stated purity) cylindrical bar 10 mm in diameter by 300 mm in length. The sample materials were reduced at a rate of 5mm/s by a single pass wire drawing to 3.99 mm to give close to 60 percent reduction. Some of the wire drawn samples were annealed in furnace at 450° C for six hours. The microstructure of as received rods of Al, drawn Al wires, and annealed drawn wires was examined by optical microscopy, scanning electron microscopy, transmission electron microscopy and by x-ray diffraction techniques. Scanning microscopy images were taken with a high resolution XMU FIB-FESEM operating at 5 keV. TEM micrographs and electron diffraction patterns were taken of the drawn and annealed samples using a 200 keV FEI Tecnai F-20 Transmission Electron Microscope. For preparation of cross-sectional and longitudinal section thin foil specimens, cylindrical discs of 3 mm were cut using an ultrasonic disc cutter. The discs thus prepared were sliced with a diamond saw to 0.5 mm thickness or less, then electro polished in a solution of 70% methanol and 30% nitric acid at a temperature of -23° C and at a voltage of 14 V. Polishing was halted just after perforation. The specimens were then cleaned in distilled water, ethanol, and acetone. Pinhole x-ray diffraction transmission technique was used for the determination of texture in the wire drawn samples.

Result and Discussion

The microstructure of as received high purity Al metal appeared polycrystalline constituting of reasonably large sized (500to 100 µm) close to equi-axed grains (Figure 1a).



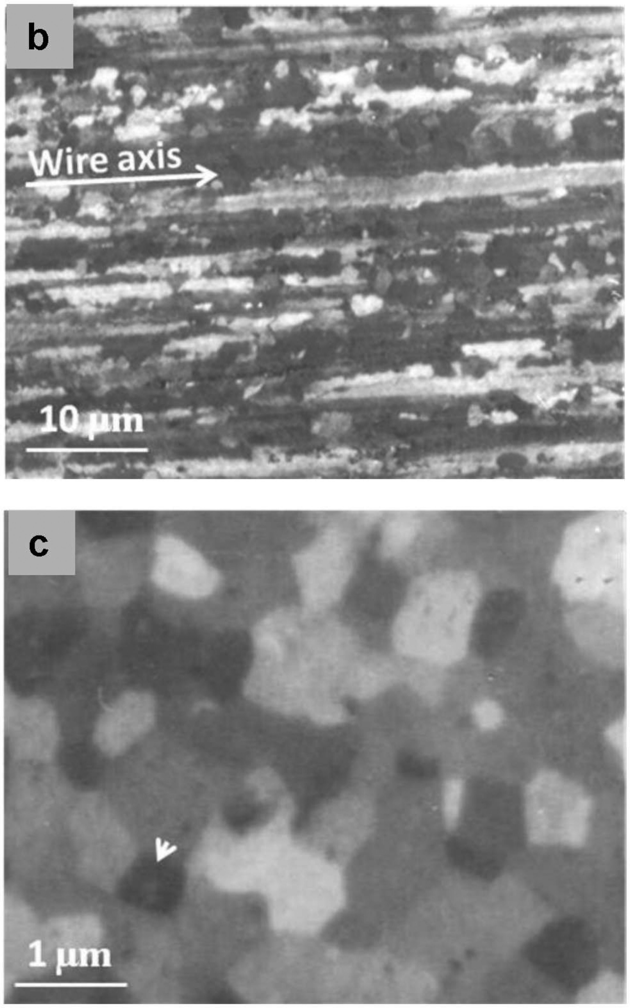


Figure 1. Optical micrographs: (a) as received Al sample, (b) , wire drawn sample of Al, (c) annealed wire drawn sample of Al.

Almost all the grains in the microstructure seldom exhibited any evidence of slip traces. In the wire drawn samples as is shown Figure 1b, every constituting grain were found elongated and oriented along the wire axis. The size of the oriented grains present in between center and edge of cylindrical wire drawn samples appears to show little difference. Grains found in the annealed Al wire also appears equi-axed but of censurably small n sizes than that found in the as received sample. Figure 1c is the optical micrographs of a transverse section of an annealed Al wire.. It exhibits cubical morphology for some of the grains. One of the cubic morphology grain is indicated by an arrow in the micrograph. This feature of the micrograph suggests that the recrystallization of the wire drawn sample probably resulted in the development of regions in the resultant sample that possess a <100> texture.

Pinhole x-ray diffraction pattern taken from wire drawn samples was found identical to that reported earlier [5] for the texture of wire drawn Al metal. It exhibited very faint and incomplete 111 and 200 Debye rings, each of which includes four intense equally spaced Debye arcs (elongated diffraction spots).Upon analyses the pattern also revealed that the wire has a strong <111> and a weak <100> fiber texture.

TEM specimens prepared from longitudinal and cross sectional sections of the drawn wire upon investigation by the TEM yielded microstructure in which columnar deformation bands are present in majority of the TEM investigated areas.

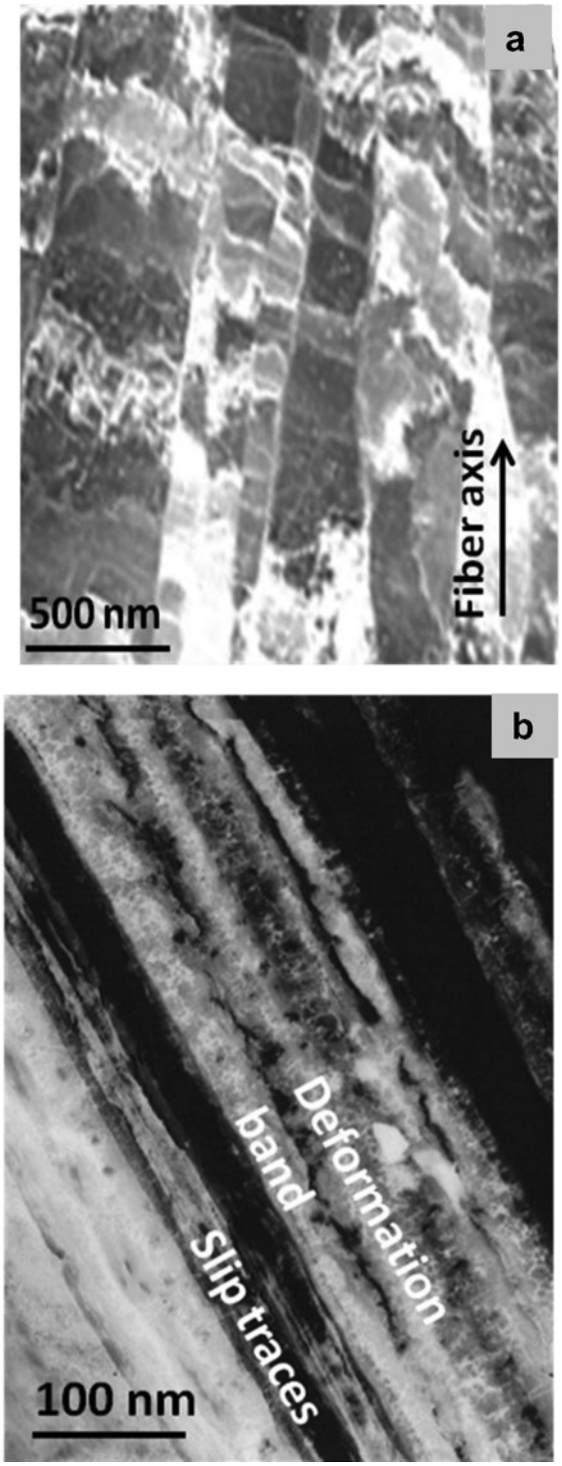
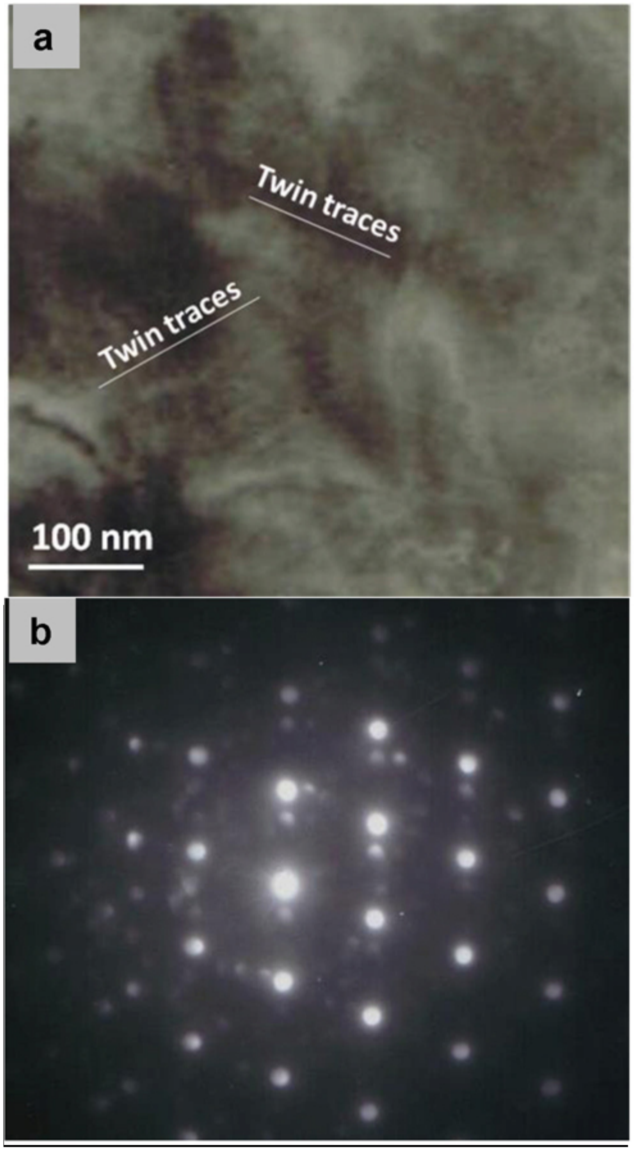


Figure 2: TEM micrographs: (a) Columnar deformation bands present in a longitudinal section TEM specimen of the wire drawn Al , (b) detailed structure of the constituents of a couple of deformation bands.

Figure 2a shows a low magnification TEM micrograph of a group of columnar bands present in a longitudinal section TEM specimen. Most of these deformation bands make an average angle of 15 to 20° with the drawn axis. A high magnification microstructure of a couple typical columnar bands is given in Figure 2b. It exhibits constituting slip or twin traces that also run parallel to the propagation direction of the band. The micrograph also exhibits a very detailed cellular structure (at the top portion of the central band) .that is probably formed by a network of dislocations formed during drawing operation. Similar cellular structure of dislocations was also found [6] in Cu that was subjected to cyclic deformation. Each of the individual bands in the micrograph of Figure 3b exhibits slip or twin traces that also run parallel to the propagation direction of the band. Such slip//twin bands were also found to be present in cross sectional section EM specimens. These slip bands as found in all favorable deformation directions of the present study collectively provide the opportunity to develop a <111> fiber texture to the wire as suggested by Ahlborn and Wassermann [4].

The TEM studies on the drawn Al wire samples also revealed that many neighboring deformation bands contains entrapped materials that possess a group of zigzag co-zonal {111{/<112> twins. Figure 3a is the TEM image of such a region containing such a group of zigzag co-zonal. Its corresponding diffraction pattern is given in Figure 3b. Analyses of the diffraction pattern (Figure 3cand d) revealed that the twin traces extend along <211> and twin planes are {111}. The analyses conform well with the zigzag configuration of co-zonal twin traces present in the micrograph of Figure 3a. This phenomenon of zigzag twinning is likely to exist in all other {111}. This suggests that the entrapped region experienced deformation twinning on multiple {111} planes. In a {110} plane, the resultant direction of co-zonal zigzag twins for a f,c.c. crystal can be <100>. This is shown in Figure 3d for a schematic diagram for an assembly of equal number of zigzag twins The configuration of the zigzag twins present in the micrograph of Figure 3a can be



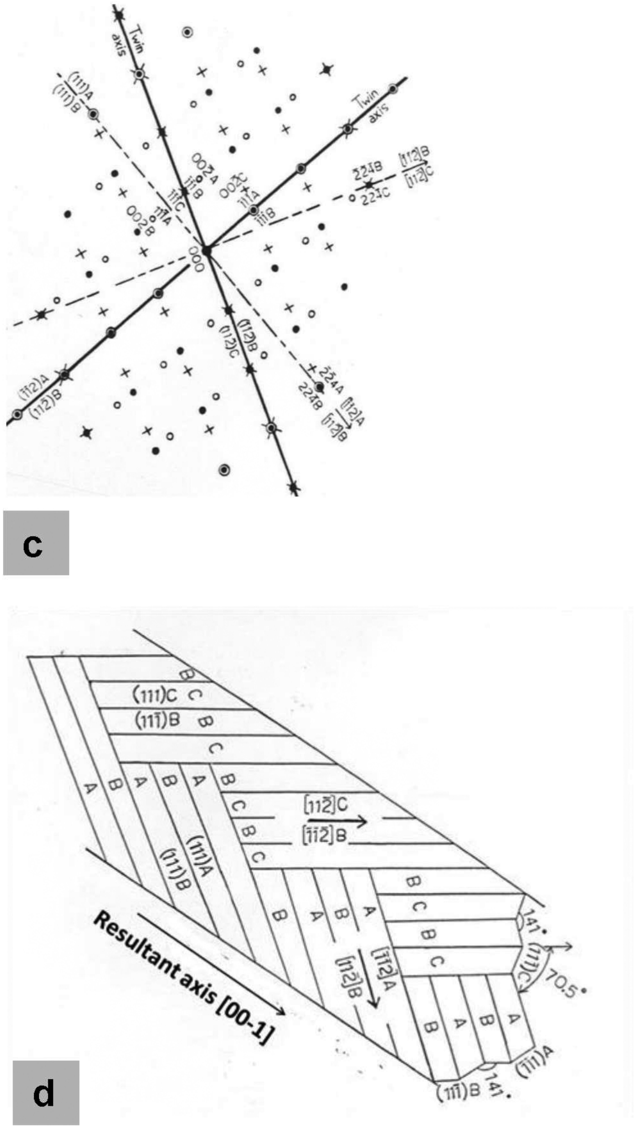
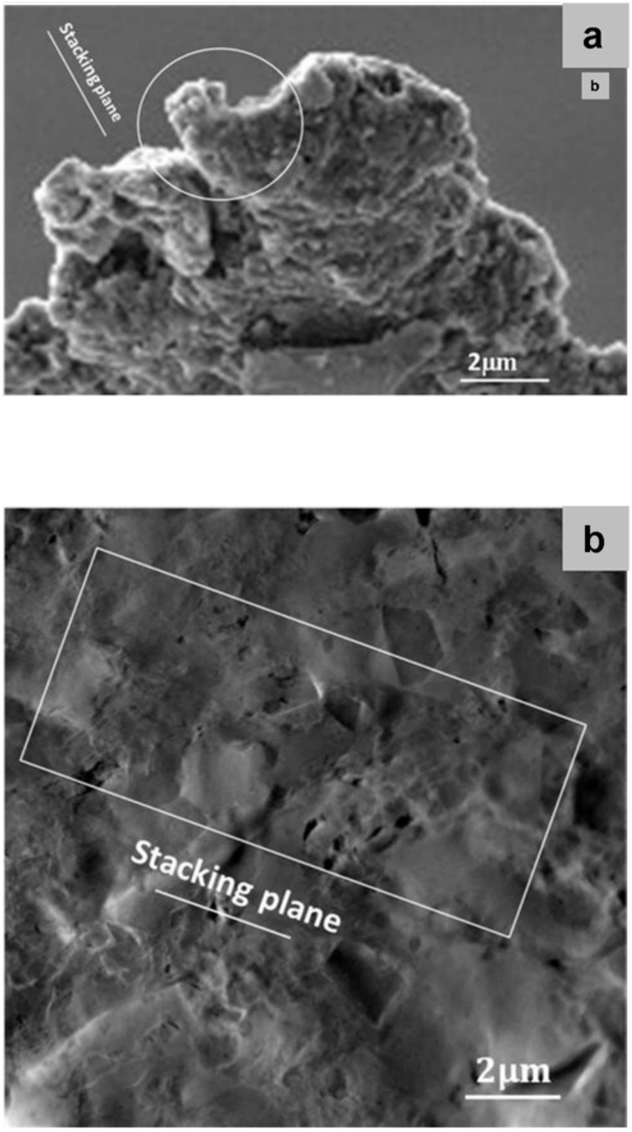


Figure 1: (a) TEM micrograph showing traces of zig-zag twins, (b) diffraction pattern taken with electron beam parallel to a <110> of the investigated crystal, (c) indexing for the crystal diffraction pattern, (d) orientation relationships between twinned crystals in (a).

conceived to be representative of a pattern that follows the resultant <100> direction of twinned crystals just discussed..

From the view point of the development of the <100> fiber texture in drawn Al wire, the presence of zigzag twins in this trapped region of the wire sample is interesting. This suggests that during wire drawing typical deformation band regions does not entirely comprised of a large number of solely slip/twin bands responsible for the development of the <111>fiber texture [4]. It also contains included regions of zigzag twins caused by mechanical twinning that probably develops <100> fiber texture in according to suggestion put forward by Cullity [5]. TEM observation of the thin foil samples of annealed wire drawn samples revealed that the microstructure is free of any cellular structure form by dislocation network. However, constituting grains in annealed sample exhibit an arrangement that closely resembles to that are found for mosaic tilting of closely stacked materials along certain plane. The tiling arrangement of grains just discussed can be found present both in the SEM and TEM micrographs shown in Figure 4a and 4b respectively.



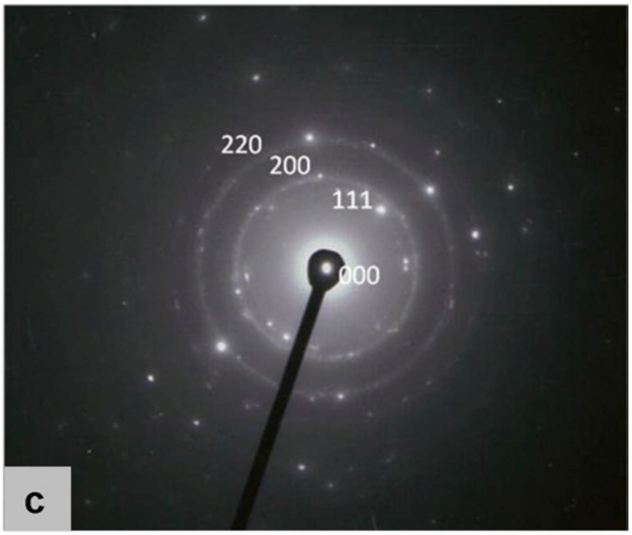


Figure 4: (a) SEM micrograph of transverse section of an annealed sample of drawn Al wire, showing preferential stacking of constituting grains at the circled area, (b) TEM micrograph of the sample in (a), showing traces of stacked grains (enclosed in a rectangular area) along certain crystal plane, (c) electron powder diffraction pattern from a thinned area in (b).

Selected area diffraction pattern covering a large number of such tilling grains (Figure 4c) of transverse section TEM specimens of annealed wires revealed a very weak (almost absent) 111 and strong 200 and 220 diffraction rings. The almost absences of the 111 ring, and presence of 220 ring in the diffraction pattern does indicate the presence of large number of grains that have their (111) normal to the electron. These features of diffraction pattern found in transverse section samples of annealed wires strongly suggest that the annealed wire drawn sample has a strong <111> fiber texture. Furthermore, the presence of strong 200 and 220 diffraction rings in the pattern indicates some grains do have their {100} plane perpendicular to the electron beam and contribute to the weak <100> texture of the annealed wire.

Conclusion

Determination of structural status of pure Al metal following a single pass heavy drawing and subsequent annealing obtained by the present study reveals some important conclusion about the deformation\behavior of drawing operation. The presence of deformation bands constituting slip bands in majority portion of the drawn samples reveals that the strong <111> fiber texture in wire drawn sample is accomplished by a combined action of the slip bands. The zigzag twinned regions found in a minority portion of the deformed wire sample is suggested to be related to the presence of weak <100> fiber texture. Annealed wire drawn samples retain the texture of the drawn metal,

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