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Preparation of [001]45ºtilt Quasi-periodic Grain Boundaries in Aluminium for HREM Studies by Cross Rolling and Annealing

M. Shamsuzzoha

School of Mines and Energy Development, University of Alabama, Tuscaloosa, AL 35487, USA

The information resolution limit of present-day easily accessible high resolution electron microscopes is about 0.14nm. This resolution limit allows most face centered cubic (FCC) crystals to expose a projected point pattern along <100>, <110>, <111> and <112>.directions. Hence, simultaneous imaging of the atomic columns across the interface between planes that are normal to these directions is possible, provided that a 1º or less misoriention between participating grains exists [1]. This allows for the opportunity to study the structure of some quasi-periodic grain boundaries, such as [001] 45º tilt grain boundary of such metals. Quasi-periodic bi-crystal meeting these requirements for HREM studies can be prepared by the Czochralsky techniques [2] or by the Bridgeman technique [3]. However, these crystal preparation techniques are very expensive and require modern state-of-art instrumentations. In the present study, as presented here it was found that the cross rolling and annealing of pure Aluminium crystals yield a duplex microstructure in which most of the constituting grains form (100)/(110) interface with each other.

Cylindrical rods 10 mm in diameter and 50 mm in length made of Aluminium with stated 99.999% purity were subjected to equal step cross rolling to obtain a total reduction of 99%. The pass step used was 5% of the initial thickness. Heavily deformed cross rolled samples were then given two cycle annealing. The first cycle annealing was primary recrystallization that was performed at temperatures ranging between 150 to 200ºC for 2 hours. The second cycle annealing was secondary recrystallization, which was performed at temperatures ranging between 300 to 400ºC for 12 hours. Thin foil samples from these annealed samples were prepared by electro polishing. Thin foil samples thus prepared were subjected to TEM and HREM studies.

All annealed samples showed a microstructure in which grains are equiaxed in morphology with sizes in the range of 100 nm to 1000 nm. This is evident in the microstructure of a typical annealed sample shown in Figure 1.TEM investigation of these grains revealed an in-plane duplex microstructe made of (100) and (110) grains. Elaborative analysis of many samples revealed that about 30% of such grains show (110) / (100) interface. However, a misoriention of 1º or less between the plane normal of such neighboring grains in the present cross rolling and annealing was found so far limited to about 1%. Figure 2a is a [001]45º tilt quasi-periodic grain boundary between neighboring (100) and (110) grains. A diffraction pattern with electron beam parallel to [001] and [110] of constituent grains across the interface is given in Figure 2b.

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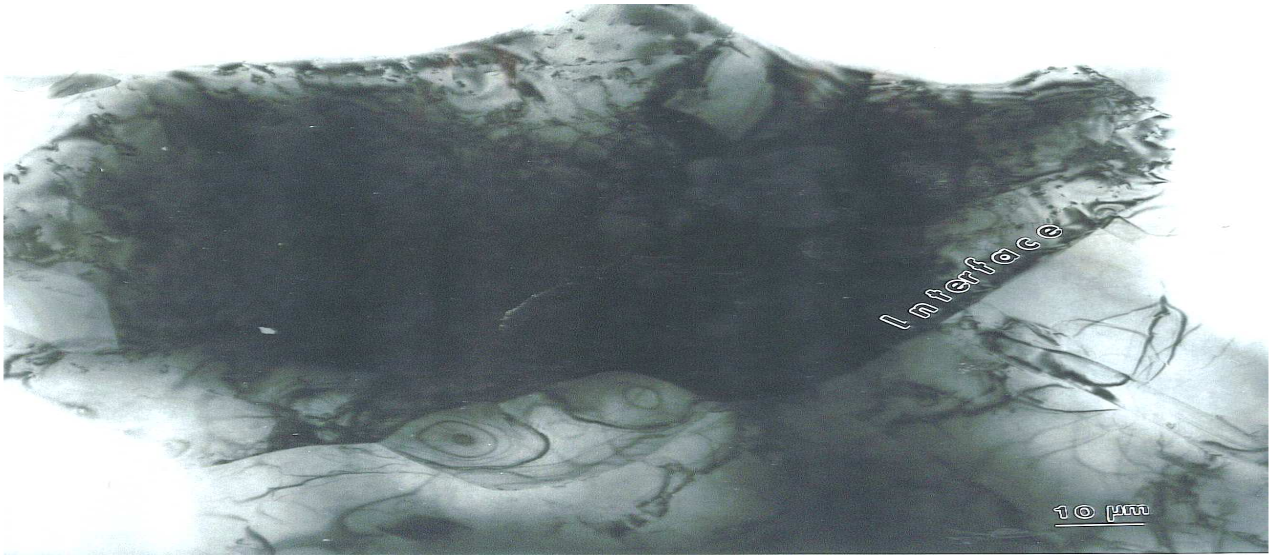
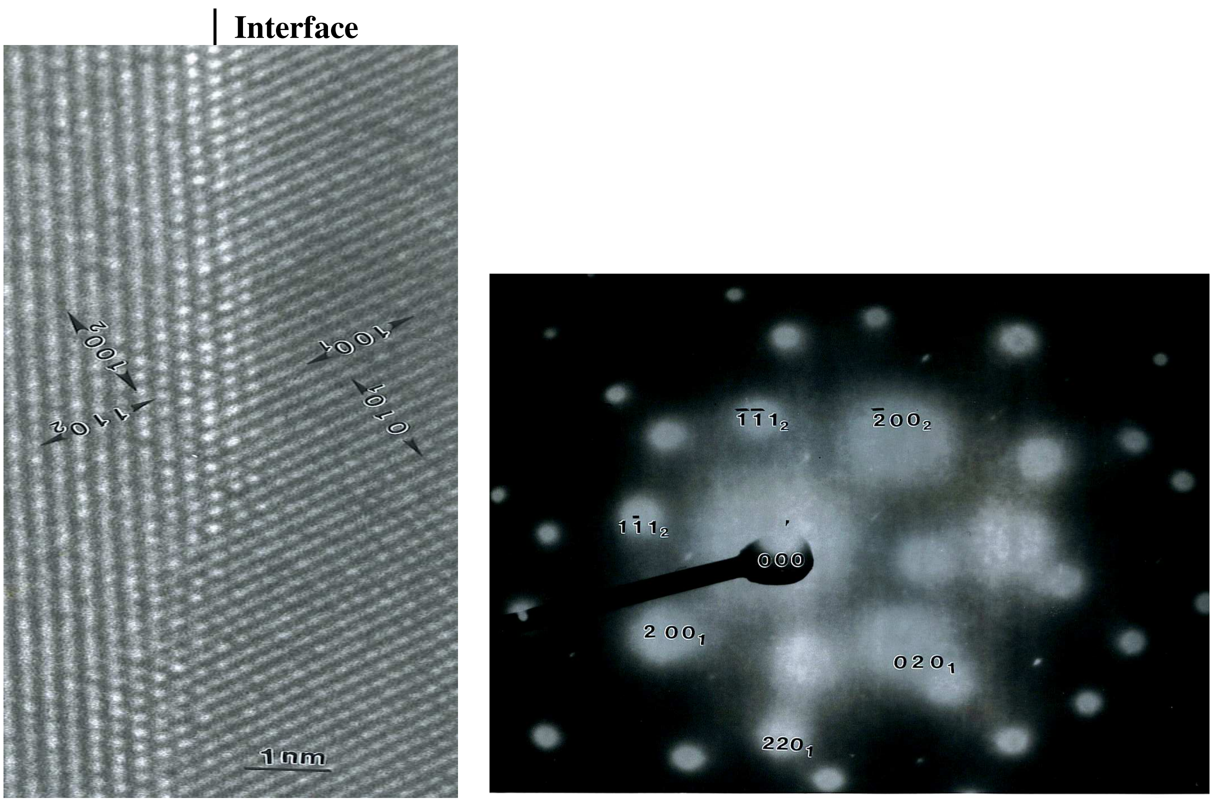


Figure1. TEM bright field micrograph taken of cross rolled-annealed sample. It shows typical grain morphology of the constituent microstructure.



(a) **(b)**

Figure2. (a) HREM image of [001]45º tilt quasi-periodic grain boundary in Al, (b) Corresponding selected area composite diffraction pattern.

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