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Original Research

On the contribution of boundaries towards strength in oxygen free high conductivity copper subjected to repetitive upsetting – extrusion $\dot{\phi}$

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ABSTRACT

Oxygen free high conductivity (OFHC) copper was subjected to 1, 5, 10, 15 and 20 cycles of repetitive upsetting and extrusion (RUE) process at room temperature. Microstructure and microhardness in the RUE processed material were evaluated at specific locations and correlated with the equivalent plastic strain. The microhardness of the material was found to be independent of strain or number of RUE cycles at certain locations whereas it exhibited strain softening behaviour at certain locations even though significant grain refinement was achieved. This difference in behaviour is attributed to the varied strength contribution from different types of boundaries present in the material after deformation.

1. Introduction

Among the various severe plastic deformation (SPD) processes that are in vogue $[1-3]$ $[1-3]$, Repetitive Upsetting and Extrusion (RUE) is a relatively new process. The process was originally proposed by Aizawa and Tokumitu [\[4\]](#page--1-1) to process powder materials for bulk mechanical alloying. Hu et al.[\[5\]](#page--1-2) used the process to impart severe plastic deformation on aluminum alloy and achieved significant grain refinement. By carrying out experiments on various materials and finite element analysis, Balasundar et al. [6–[10\]](#page--1-3) established a modified die design that can be used to successfully impart severe plastic deformation in bulk material without forming defects such as fold and axial hole or funnel. Pardis et al.^[11,12] later introduced cyclic expansion extrusion (CEE), a variant of the RUE process by which double the volume of material can be processed. Though significant grain refinement can be achieved in a variety of materials by subjecting them to RUE process, a clear understanding on the structure-property correlations has not been reported so far. A more commonly observed phenomenon reported in Cu subjected to various SPD processes is that the strength of the material increases initially with number of cycles and thereafter reaches a saturation level [\[12](#page--1-5)–17]. Such a steady state or strain independent behaviour has been attributed to the saturation in grain refinement where grain boundary rotation was cited as the main grain refinement mechanism. However a less commonly observed behaviour is a decrease in strength with increasing deformation [\[18](#page--1-6)–20]. In most of the studies, the decrease in strength

has been attributed to recrystallization and grain growth [\[18\]](#page--1-6) or decrease in the dislocation density [\[19\].](#page--1-7) In a more recent work, Pardis et al.^[12] reported that oxygen free high conductivity (OFHC) Cu when subjected to CEE, exhibits strain softening till an equivalent strain of 6 and thereafter attains a steady state or strain independent behaviour. They attributed such a behaviour to the formation of strong <110> fiber texture. As there are contradicting reports available in the literatures [11–[20\],](#page--1-4) it is essential to ascertain the reason for two different types of variation in strength or hardness viz., strain independent and strain dependent softening behaviour exhibited by Cu when subjected to SPD processes. As OFHC Cu subjected to RUE process exhibits both these behaviours, a detailed investigation has been carried out in this study to establish microstructure-mechanical property correlation.

2. Experimental procedure

2.1. RUE process

A cycle of RUE comprises upsetting and extrusion as shown in [Fig. 1](#page-1-0). A cylindrical work piece of known dimension is first subjected to upsetting, wherein, the length is reduced and the cross-sectional area is increased. The upset work piece is subsequently subjected to extrusion, wherein the length is increased and the cross-sectional area is reduced. The upsetting and extrusion processes are repeated to the desired number of cycles. The RUE die can be divided into three parts or

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Fig. 1. Schematic illustration of RUE processing: (a) Initiation of Upsetting; (b) End of upsetting; (c) extrusion; (d) End of extrusion [\[10\]](#page--1-9).

regions each having a volume V_1 , V_2 , and V_3 respectively. These volumes are designed in such a way that $V_1+V_2=V_2+V_3$. During the upsetting stage the work piece fills the volume $V_u=V_1+V_2$ and during the extrusion stage it fills the volume $V_e=V_2+V_3$. The volume of the work piece (V_w) therefore is given by $V_w=V_e=V_u$, i.e., $V_w = V_1 + V_2 = V_2 + V_3.$

2.2. Material

Oxygen free high conductivity copper (OFHC) Cu was procured from M/s. Nonferrous materials technology and development centre (NFTDC), Hyderabad in the form of 50 mm diameter annealed bars. The microstructure of the as-received material is shown in [Fig. 2.](#page-1-1) The material was found to have a microhardness of 60.2 ± 5.3 VHN with an average grain size of 40.2 ± 10.16 µm.

2.3. RUE experiments

Five cylindrical samples of 20 mm diameter and 30 mm height were electrode discharge machined (EDM) from the as-received bar. The

321 R in order to reduce friction. The coated samples were then subjected to RUE experiments at room temperature using a 250 MT servo-hydraulic press. A ram or punch speed of 1 mm/sec was used to deform the material during both upsetting and extrusion process. The details on die design and other tools used are described in detail elsewhere [\[6](#page--1-3)–8]. The RUE die used for the study here imparts a true strain of ε =2 ln (D₂/D₁) =0.693 (D₁ and D₂ are defined in [Fig. 1](#page-1-0)) during either upsetting or extrusion operation. The strain at the end of a RUE cycle i.e., after completion of an upsetting and extrusion operation would be 1.38, which corresponds to a Von-Mises equivalent strain of 1.13.

OFHC Cu samples and the RUE die were coated with Molykote® D-

Samples after 1, 5, 10, 15 and 20 RUE cycles were vertically cut into two halves. One half of each sample was then hot mounted and subjected to mechanical polishing. In order to evaluate the microstructure, the polished samples were etched using a mixture of 80 ml water, 10 g of potassium dichromate, 2 ml of sulphuric acid. The microstructures at various locations of the samples were recorded using an optical and scanning electron microscope (SEM). Based on finite element analysis of RUE process, specific locations or regions were identified in the deformed material to establish structure-property correlation as shown in [Fig. 3](#page--1-8). A detailed description of the finite element analysis can be found elsewhere [\[6,9\]](#page--1-3). Only those regions (A-D) that correspond to a certain average equivalent strain which can be identified without any ambiguity on the deformed sample were selected to establish the structure-property correlations. The microhardness of the material at these selected regions (A-D) was evaluated using Vickers microhardness tester. A load of 50 g with a dwell time of 10 s was used for microhardness measurement. A minimum of 5 readings were taken at each location in order to obtain the average microhardness at that location. To obtain the required microstructural information, first a coarse scan on an area of $627 \times 461 \mu m^2$ with a step size of 1.5 µm was used to get an overview of the selected region using SUPRA 55 field emission scanning electron microscope (FE-SEM) equipped with Nordlys EBSD detector. A second refined scan of $585 \times 574 \mu m^2$ with a step size of 0.9 μ m was then used to acquire the relevant data. TSL software from TexSEM laboratories (TSL) was used to quantify the relevant information such as misorientation angle (θ), boundary fraction, boundary spacing etc.

Fig. 2. Inverse pole figure (IPF) of OFHC Cu in the as-received (annealed) condition.

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