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Scaling up Segal's principle of Equal-Channel Angular Pressing

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ABSTRACT

This paper focuses on scaling of Equal-Channel Angular Pressing (ECAP) from conventional, laboratory scale (billet cross section $15 \times 15 \text{ mm}^2$) to large scale ($50 \times 50 \text{ mm}^2$). We study pure copper billets produced by ECAP in two identical ECAP-dies (but with different cross-sections) that have been optimized to provide reduced contact friction. In order to characterize processing parameters and the resulting properties, the billets are processed by 4 and 8 passes on both scales. Mechanical and microstructural characterization is performed by hardness testing and EBSD measurements. The materials produced in the different scales show very similar properties. A slight top to bottom hardness gradient (<6%) is detected in the billets on both scales. After 4 passes, his gradient is also reflected in grain size distributions. The higher cumulated strain after 8 passes leads to a more homogenized microstructure, again with similar grain sizes for both scales. Our results show that there are no scaling effects regarding the mechanical properties and the microstructures when comparing laboratory and large scale ECAP. This study clearly highlights the potential for scaling ECAP (using a suitable die-design) for a commercial implementation of ultrafine-grained materials.

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1. Introduction

Cold plastic deformation processes such as forging, rolling or drawing are very effective for altering the microstructure (e.g. in terms of strain hardening and manipulation of texture) and mechanical properties (e.g. elevated strength) of many metallic materials [1]. Recent research shows that the application of cold severe plastic deformation (SPD) with superimposed hydrostatic pressure allows for the homogenization and refinement of microstructures into the sub-micrometer regime [2]. One of the most promising SPD processes is Equal-Channel Angular Pressing (ECAP), which was invented about 35 years ago by Segal [3]. In his seminal papers [3,4], Segal applied the ECAP technique to introduce very high strains (as e.g. typical for cold wire drawing) into bulk billets of pure copper and pure iron. This allowed the characterization of severely cold worked materials (i.e., ultra-fine grained (UFG) materials) with standard tensile samples for the first time.

The fundamental principle of ECAP is that a billet (typically with square or round cross-section) is pressed through a rigid die, and forced to flow through an angled channel (see Fig. 1). A shear strain is introduced into the material in a thin zone at the intersection plane of the in- and outgoing channels. For the typical case of an angle of 90°, the equivalent von Mises strain (per pass) is 1.15 [5]. One of the key features of the ECAP technique is that the outer dimensions of the billet remain practically unchanged so that the pressing can be repeated several

times with the same billet in order to reach very high cumulative strains [6]. Today this basic concept for ECAP is well established. It is widely used in a laboratory scale for the improvement of different metallic materials [7–16]. But despite the large number of publications focusing on ECAP and the corresponding gain in scientific knowledge in the field of severe plastic deformation over the last two decades, practical applications of UFG materials produced by ECAP are still rare. Only a few attempts to scale up [9,17-23] and to commercialize [21,24-28] the process have been reported so far. In part because of the relatively small, laboratory scale billet sizes, and because of the fairly long processing times, ECAPed materials still are mainly interesting for the rare kinds of applications where the costs of the materials themselves only play a minor role. This strongly limits the potential fields of applications for UFG materials; there clearly is a need for further detailed work on up-scaling and commercialization of the ECAP process that takes different technological and processing parameters into account.

Recent work [6,29–32] has shown that the contact friction between the billet and the ECAP die plays an important role with respect to homogeneous deformation: friction needs to be reduced in order to achieve optimum processing conditions, i.e., an intensive and uniform simple shear deformation and a good surface quality of the billets. Another crucial detail affecting the uniformity of shear straining during ECAP, and thus also the uniformity of the resulting microstructures and macroscopic properties, is the geometry of the shear zone, which can be characterized by an opening angle ψ (see also Fig. 1). Very small opening angles correspond to an ideal, pure shear deformation with a fully homogeneous strain distribution. Homogeneity is, however, significantly reduced when the shear zone exhibits a fan-shape ($\psi \gg 0^{\circ}$), [11, 33–36] and gradients can be observed in the post-ECAP strain

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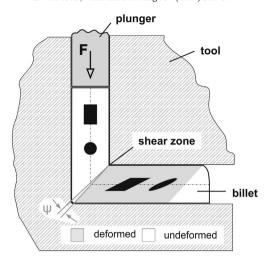


Fig. 1. Schematic illustration of the principle of ECAP for a conventional die with an angle of 90° . When the material passes through the corner of the die, a simple shear deformation is introduced in a thin, fan-shaped shear zone (characterized by an angle ψ) while the macroscopic dimensions of the billet remain unchanged. The cross section of the channel is typically round or square. A volume element in the billet is subjected to rotation and shear, as indicated by the black rectangular/circular shapes before and after shearing.

distributions. It should be noted that materials that show pronounced strain hardening and/or increased strain rate sensitivity tend to form such fan-shaped shear zones even when dies with sharp corners are used [33]. As shown by experimental [31,32] and numerical [37] analyses, this strong issue, which appears to be intrinsic for the ECAP process of conventional materials, can be overcome by the application of a backpressure (BP). Using a second plunger to apply a force against the billet's front end in the exit channel restricts material flow, leads to a superimposed hydrostatic pressure in the shear zone and thus considerably reduces the fan angle. While in principle well documented, these important engineering aspects of ECAP processing have largely been ignored by the majority of the scientific community so far.

In the present study, we use two unique ECAP dies with different channel-cross-sections for the investigation of the influence of scaling effects on the microstructural refinement and the corresponding mechanical behavior. The friction and shear zone geometry are carefully controlled by the use of sliding parts and the application of a well-defined BP.

From the point of a simple dimensionless scaling up of the process, one may not expected that significant scaling effects can occur during ECAP. However, real engineering materials are characterized by intrinsic length scales that can typically be directly related to microstructural features like the grain size. Even when the ECAP die itself, and the corresponding stress and strain fields (including size and fan-shape of the shear zone), are properly scaled up, and friction conditions are kept similar, the ratio of the intrinsic microstructural length scale to the characteristic dimensions of the ECAP process will change during up-scaling. And because the shear zone geometry is not (as is often assumed in simple models) a single plane, the deformation history experienced by individual grains while passing through a fan-shaped shear zone are likely to be different when using differently sized ECAP dies. Scaling effects may therefore well be observed under realistic processing conditions. There clearly is a need for an experimental study on scaling up ECAP that is focused on the resulting microstructures (and the corresponding mechanical properties) after processing. To our knowledge, this is the first report that systematically combines and compares results from experiments with two identical ECAP dies of different sizes. The main focus of this work is on an in-depth analysis of the resulting microstructures produced on laboratory vs. large scale.

2. Experimental

For this study on scaling up of the ECAP process we used oxygen free high conductivity copper (OFHC-Cu) with a purity of more than

99.99 wt.% as a model material that is well understood in terms of physical metallurgy and mechanical behavior. Square-shaped billets with cross sections of $15 \times 15 \text{ mm}^2$ (laboratory scale) and $50 \times 50 \text{ mm}^2$ (large scale) and lengths of 120 mm and 300 mm were used, respectively. Prior to ECAP all billets were annealed at 600 °C in a convection furnace; this allows for static recrystallization which results in an initial condition that is free of any microstructural features associated with cold work. Multi-pass ECAP was performed at room temperature (RT) for 4 and 8 passes, respectively, using the two different dies introduced in the next section. In-between consecutive pressings, the billets were rotated along their lengthwise axes, alternating between rotations by 180° and 90°. This rotation scheme is generally referred to as route E. When compared to the well-known routes C and Bc, route E provides an optimum of fully worked billet volume and rapid grain refinement [38]. 4 passes are often seen as good compromise for achieving a reasonable grain refinement with a moderate effort for processing. The more time consuming processing for 8 passes is most frequently applied because it is known to ensure a fully homogeneous UFG microstructure for most materials.

A very low processing speed of 20 mm/min was chosen so that the temperature rise in the shear zone is negligible [39]. Maintaining quasi-isothermal conditions is particularly important for thermally unstable materials such as OFHC-Cu in order to prevent dynamic recovery or recrystallization [40]. The formation of a pronounced fan-shaped shear zone was suppressed by using a BP of 75 MPa, applied via the hydraulic cylinders in front of the outgoing channels. A high viscosity, mineral oil-based grease, containing a mixture of molybdenum disulfide and graphite, was used for lubrication of the moving parts of the dies as well as for the billets. These experimental parameters ensure a very homogeneous plastic shear deformation during ECAP both in laboratory and large scale [31,32]. The resulting opening angles of the shear zones (ψ – measured from as-processed billets) were less than 2°, both after 4 and 8 passes (corresponding to accumulated equivalent plastic strains of ~4.6 and ~9.1, respectively, [5]) and for both billet sizes.

In order to obtain information about the global homogeneity after ECAP, Brinell hardness (HBW 2.5/62.5) was measured in the longitudinal planes (x–z-plane, see also Fig. 2 for a definition of the coordinate system used throughout this paper). Indents were placed in rectangular grids consisting of 200 and 550 sites (corresponding to indent spacings of 3 and 5 mm) for the laboratory and large scale billets, respectively. Hardness distributions across the billets were carefully analyzed, and two characteristic locations (representing the sites of minimum and maximum hardness) were identified for subsequent microstructural investigations.

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