pressing (ECAP), Choi et al. [20] measured a transition grain size of 1 μm in pure Mg samples fabricated by hot extrusion of ball-milled powders, and Chino et al. [21] found negligible twinning activity in an extruded AZ31 Mg rod when $d = 8$ μm. Other works, on the contrary, observe no variation in the twinning activity with grain refinement [22,23]. Ghaderi and Barnett [22] reported that the twin volume fraction during room temperature compression of an extruded AZ31 alloy along the extrusion axis (EA) remains invariant for grain sizes ranging from 5 to 55 μm. Muránsky et al. [23] also measured the same twinned fraction in an extruded ZM20 alloy with d values comprised between 17 and 114 μm under similar testing conditions. Finally, Wu et al. [24] reported the presence of twins in a ball-milled nanocrystalline Mg-10 at.% Ti with an average grain size as small as 33 nm. The effect of grain size on twinning is, clearly, still an open question that needs further clarification.

Testing conditions such as strain rate and temperature are also known to alter dramatically the twinning activity [1,5,7,25–35]. In particular, decreasing the strain rate and/or increasing the deformation temperature hinder twin activation to such an extent that, at a constant strain rate, twinning might be suppressed at sufficiently low temperatures and conversely, at a constant temperature, twin activation ceases below a critical strain rate [34]. The rationale behind these observations is that the CRSS for twinning is less sensitive to strain rate and temperature than that of slip [5] and this is reportedly consistent with the large core width (w) predicted for $\{10\bar{1}2\}$ zonal twinning dislocations ($w = 6a$) [1]. Indeed, the CRSS of tensile twinning has been shown to remain basically constant at strain rates ranging from 10^{-4} to $10^{3}\ s^{-1}$ and at temperatures comprised between room temperature and 300 °C [3,6,7,27,34–36]. It is generally believed that, with increasing temperature, twinning is gradually replaced by non-basal slip, as it is known that the CRSSs of prismatic and pyramidal systems decrease rapidly with temperature [3,36]. However, the nature of the actual slip mechanism that replaces twinning at high temperatures or, for that matter, at low strain rates, has never been measured directly. The reason for this is that transmission electron microscopy (TEM) which is, to date, the most widely used experimental characterization technique to obtain *direct* evidence of the relative activity of different slip systems [37], does not yield sufficient statistics in coarse-grained polycrystals. This knowledge gap precludes a good understanding of the fundamental basis behind the observed dependence of twinning on strain rate and temperature.

The aim of this work is to clarify the origin of the twinning- to slip-dominated flow transition in pure Mg with decreasing grain size, decreasing strain rate and increasing temperature. With that purpose, several hot rolled pure Mg polycrystals with different grain sizes are compressed in-situ along the rolling direction (RD) at temperatures ranging from 50 to 250 °C and at strain rates comprised between 10^{-5} and $10^{-3}\ s^{-1}$. The slip and twin activities are estimated by electron-backscattered diffraction (EBSD)-assisted trace analysis. Denuded efforts were devoted to measure a large number of traces, in order to ensure the reliability of the study. Twin and slip activities are related to the corresponding microstructure and to the testing conditions, and are discussed in the frame of the existing literature.

2. Experimental procedure

The material employed in the current work was commercial high quality pure magnesium (99.95%), which was received as a 10 cm diameter ingot in the as-cast condition. Slabs of the as-received material with ~10 mm in thickness and ~30 mm in width were processed by hot rolling at 200 °C using three passes, each of 50% reduction, to a final thickness of ~3 mm. Post-processing heat treatments at 300 °C for 5 min and at 150 °C for 10 min were then carried out in order to generate two microstructures with $d = 19$ μm and $d = 5$ μm, respectively. The dominant basal textures as well as the GB misorientation distributions are very similar in both polycrystals, as demonstrated in a previous publication [38].

The microstructure and the crystallographic texture of the two rolled and annealed samples were examined by scanning electron microscopy (SEM) and EBSD using a field emission gun SEM (Helios NanoLab 600i, FEI) equipped with an HKL EBSD system, a CCD camera and the Channel 5.0 data acquisition and analysis software package. EBSD measurements were conducted at an accelerating voltage of 15 kV and 2.7 nA, using different step sizes depending on the sample grain size (0.2–1.5 μm). The average grain size values were calculated by the linear intercept method from EBSD maps in the normal direction to the rolling plane (ND) using only grain boundaries with misorientation angles greater than 15°. Sample preparation for EBSD included mechanical mirror-polish using first diamond pastes of increasingly finer particle sizes and then a colloidal silica slurry finishing. Finally, a chemical polish was performed for 5 s using a solution comprising 12 ml HCl, 8 ml HNO_3 and 100 ml methanol.

A series of dog-bone compressive samples with 10 mm gage length and transversal section of $2 \times 2.5\ mm^2$ were electrodischarge-machined out of the two rolled and annealed sheets with the compression axis parallel to the rolling direction (RD). Compression tests were then carried out in the two polycrystals at 50 °C and 10^{-3} using a screw-driven tensile stage (Kammrath and Weiss, Dortmund, Germany) in order to analyze the effect of grain size on their macro and micromechanical response. The testing temperature was reached and maintained using a tungsten-based heater located just below the gage section of the sample. With the aim of investigating the effect of strain rate on the dominant deformation mechanisms additional compression tests were carried out in the same samples at 50 °C and at initial strain rates of 10^{-4} and $10^{-5}\ s^{-1}$. A second set of prism-shaped compression samples with 4 mm gage length along RD and $2 \times 2\ mm^2$ section were also machined out of the rolled and annealed sheets in order to evaluate the influence of temperature. With that purpose, tests were performed at 50, 150, and 250 °C and at an initial strain rate of $\sim 10^{-3}\ s^{-1}$. These compression tests were carried out in a *Servosis* universal testing machine equipped with a four-lamp ellipsoidal furnace. Under all the testing conditions mentioned above, some tests (two per sample) were performed to failure in order to characterize the full mechanical response and others were stopped at a strain of ~10%, at which the operating deformation mechanisms were carefully evaluated by the methodology described below.

The activation of different slip systems during compression testing at different temperatures and strain rates was