examined in the two polycrystals investigated by EBSD-assisted slip trace analysis [39,40]. This approach makes use of SEM images and EBSD orientation data input into a MATLAB code [41]. The basic steps of this methodology are the following. First, one surface of each compression sample must be perfectly polished in order to facilitate the post-test imaging of a vast number of slip traces. Large areas within the gage length are mapped by EBSD before and after each compression test, with the aim of analyzing the evolution of the orientations of the grains in which slip traces become apparent during straining. The assignment of each slip trace detected by SEM examination to a specific slip system is carried out by inputting the Euler angles of the corresponding grain (measured from the post-mortem EBSD maps) into a MATLAB code, which provides as output a visual representation of all the possible plane traces corresponding to that particular orientation [41]. Comparison of the slip trace under study with those simulated by the code allows selecting the actual active slip system. In general only one slip trace (or one set of parallel slip traces) was detected for each grain and no noticeable trace rotation was apparent until the macroscopic strain at which the slip trace analysis was carried out (~10%). The activation of twinning during deformation at the different temperatures and strain rates investigated was tracked using conventional EBSD examination. The twin fraction has been estimated by calculating the fraction of grains that exhibit at least one twin within a given representative area. The Schmid factors (SF) for the slip and twin systems found to be active in each grain were calculated taking into account the corresponding pre-test EBSD-determined Euler angles and under the assumption of uniaxial stress along RD.

3. Results and discussion

3.1. Twinning to slip transition with decreasing grain size

The true stress–true strain response during compression along RD at 50 °C and 10^{-3} s^{-1} of the two pure Mg polycrystals under study is represented in Fig. 1. The characteristic concave-up nature of the flow curve of the polycrystal with $d = 19$ μm, which is regarded as a manifestation of twinning-dominated plastic deformation following yielding, has been widely reported in the literature [13,16,22,31,42–44]. The plateau of constant stress apparent at low strains was attributed earlier to the formation of Lüders bands containing high amount of twinned grains, which progressively extend across the gage length of the compression sample [45]. The high degree of work hardening has been associated to crystal reorientation due to twinning and to strong dislocation-twin boundary interactions [7,46]. The stress–strain curve corresponding to the polycrystal with $d = 5$ μm (Fig. 1) exhibits, on the contrary, a concave-down shape typical of slip-dominated flow. As expected, the average yield strength ($\sigma_{0.2}$) increases with decreasing grain size. In summary, in agreement with earlier works in the literature [7,19–21], the change in the shape of the true stress–true strain curves with decreasing grain size seems consistent with a twinning to slip dominated flow following yielding.

Estimating the dominant deformation mechanism solely from the shape of the macroscopic stress–strain curves is, however, a very rough approach. Thus, the microstructure and the microtexture of the two pure Mg polycrystals

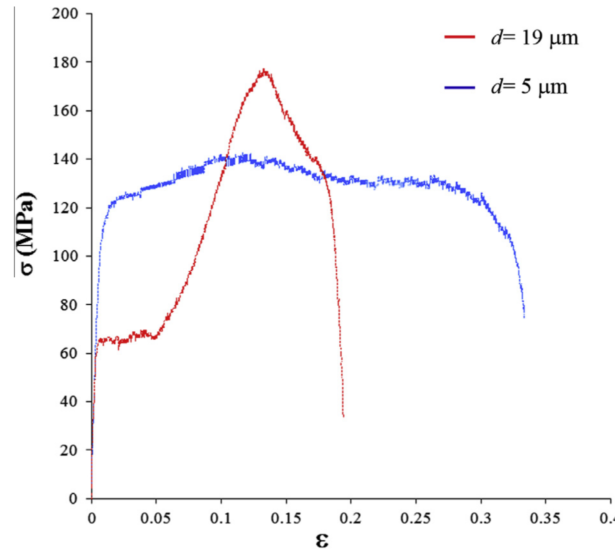

Fig. 1. Compressive true stress–true strain curves corresponding to pure Mg polycrystals with d values of 19 and 5 μm deformed at 50 °C and at an initial strain rate of 10^{-3} s^{-1}.

investigated were characterized before and after straining in order to provide further insights on the operation of different deformation mechanisms at a microscopic scale. Fig. 2a and b illustrate the EBSD inverse pole figure (IPF) maps in the ND as well as the {0001} pole figures corresponding to the deformed gage of the polycrystal with $d = 19$ μm before testing as well after compression at 50 °C and 10^{-3} s^{-1}. An SEM micrograph of the same area is plotted next to the EBSD maps (Fig. 2c). In the as-processed condition, this polycrystal exhibits a strong basal texture, with a maximum intensity of ~12 times random [38]. After straining, however, a new intensity maximum close to RD appears in the {0001} pole figure, in full agreement with previous experimental and modeling studies [5], due to lattice reorientation (~86°) by tensile twinning. Indeed, it can be clearly seen in both the EBSD maps and the SEM micrograph of Fig. 2a–c that parallel bands containing a large fraction of twinned grains develop. The fraction of twinned grains is 60% (Fig. 2b). The formation of these so called Lüders bands was attributed to enhanced twin nucleation and propagation in neighboring grains with a high degree of connectivity in order to relax the local boundary stresses [23,45]. Fig. 2d, e and g are additional SEM micrographs at different magnifications after straining. In agreement with the mentioned previous study [45], Fig. 2e illustrates how twins (black arrows) inside the Lüders bands are connected at grain boundaries. Furthermore, this figure reveals the presence of slip traces dispersed within the bands (red arrows). An exhaustive EBSD-assisted analysis of 158 slip traces revealed that 79% corresponded to basal slip, 10% to prismatic ⟨a⟩ slip, and 11% to pyramidal ⟨c+a⟩ slip. Fig. 2g depicts incipient twin nucleation and propagation in the areas between two Lüders bands. No slip traces were detected in these regions. The SF distributions with respect to the global external stress corresponding to the twins detected both inside and outside the Lüders bands, together with the corresponding {0001} discrete pole figure showing the orientation of the c-axes of the twinned grains are plotted, respectively, in Fig. 2f and h. The SF histogram