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On the nature of twin boundary-associated strengthening in Fe-Mn-C steel

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ABSTRACT

We unravel the nature of twin boundary-associated strengthening in Fe-Mn-C twinning-induced plasticity steel (TWIPs) by micro-pillar compression tests. Dislocation interactions with a coherent twin boundary and their role on strain hardening were investigated. The results indicate that twin-matrix bundles dynamically introduced by deformation twinning and their interaction with dislocations are required for strengthening Fe-Mn-C TWIPs, while single coherent twin boundaries enable dislocation transmission. Correlative studies on orientation dependent deformation mechanisms, detailed dislocation-twin boundary interactions, and the resulting local stress-strain responses suggest that twin boundary-associated strengthening is primarily caused by the reduction of the mean free dislocation path in nano-twinned microstructures.

Plastic deformation of metals is mediated by the motion of structural defects, so-called dislocations [1]. Interfaces such as grain boundaries (GBs) act as obstacles against dislocation motion, causing strengthening of the material, an effect known as Hall-Petch hardening [2,3]. Hence, the strength of polycrystalline materials increases with decreasing grain size while maintaining much of their ductility. Among all GBs specifically twin boundaries (TBs) are very attractive interfaces as they can either be inherited from alloy synthesis and processing or form during plastic deformation through strain-induced formation of twins. Thus, TBs dynamically refine the microstructure of the material upon mechanical loading impeding the motion of dislocations, a mechanism referred to as dynamic Hall-Petch effect [4]. TBs are highly symmetric GBs and are often assumed to contribute in a similar way to the strengthening of metals as conventional GBs, particularly in twinning induced plasticity (TWIP) steels [5–9]. They have also been reported to help maintaining an acceptable level of ductility [10–13]. Specifically, the high strain hardening capacity of twinning-induced plasticity steels with face-centered cubic (fcc; austenitic) lattice have been rationalized by a dynamic reduction in grain size by the formation of TBs [5,6]. With continued plastic deformation, these coherent TBs might transform into incoherent TBs caused by the interaction with dislocations providing further strengthening of the material [14].

Recent simulations show that coherent TBs impede dislocation flux kinematically [15]. This would imply that TB strengthening is due to a reduced dislocation mean free path rather than to an inherent obstacle character of the interface itself. Other works suggest that imperfections due to defects in coherent TBs can also substantially affect the mechanical behavior of nano-twinned metals [16]. This view is further supported by recent experiments on the dislocation slip transmission through coherent TBs in copper [17–19] using *in situ* nanomechanical testing of single interfaces [20]. They have indicated that the TB-containing crystals showed a comparable mechanical behavior as equally sized single crystals, whereas an impenetrable high angle GB exhibited a higher flow stresses and significantly higher apparent work-hardening than the TB. In the present study, we have characterized the detailed dislocation-TB interactions by means of micro-pillar compression tests. Specifically, this study focuses on the mechanical impact of a single coherent TB. The measured crystallite orientation and pillar fabrication enable to isolate a unique interface in a limited deformation volume with a specific orientation relationship. The dislocation interactions with the coherent TB analyzed by post-mortem TEM provide further insights into the nature of TB-associated strengthening mechanisms in TWIP steels.

A bulk oligo-crystal of Fe-22wt%Mn-0.6wt%C steel was grown by the Bridgman technique [21]. The sample was cold-rolled and subsequently annealed (PS + SA) at 950 °C for 12 h in Ar atmosphere in order to introduce TBs into the material. From this material we prepared single crystalline micro-pillars as well as bicrystal micro-pillars containing an

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inclined Σ3{111} TB using focused ion beam (FIB). The two adjacent grains sharing a coherent TB are referred to here as Grain 1 and Grain 2. The diameter of the micro-pillars investigated was 4 μm. Detailed information on the micromechanical experiments is given in [21].

Fig. 1 shows the micro-compression engineering stress-strain curves revealing comparable yield and flow stresses of the single and the bi-crystalline micro-pillars. As the highest Schmid factors for Grains 1 and 2 are very similar (0.487 and 0.486, respectively), the stress levels are comparable as evident in Fig. 1. Three micro-pillars were compressed for each crystal orientation yielding reproducible results (Fig. 1b). The measured crystallographic loading directions of Grains 1 and 2 are presented as insets in Fig. 1b and correspond to [15 6 25] and [7 2 26], respectively.

A coherent TB was created by pre-straining and subsequent annealing (PS + SA), and therefore the effects of PS + SA on the mechanical response and activated deformation mechanisms in comparison to micro-pillars produced from the as-grown single crystal [21] were first investigated. The as-grown single crystals showed a low dislocation density of 7×10^{12} m^{-2} [22]. A higher initial dislocation density due to pre-straining is expected to induce no additional size effect on the yield and flow stresses but more bulk-like stress-strain responses [23,24]. The micro-pillars produced from as-grown [21] and PS + SA (Grain 2) materials are both favorably oriented for deformation twinning, and their orientations are only 4° apart from each other. The micro-pillars of the PS + SA material exhibit a slightly higher yield stress (304 ± 6 MPa) than the as-grown single crystal micro-pillars (282 ± 44 MPa). Moreover, the standard deviation of PS + SA micro-pillars is smaller than for micro-pillars on the as-grown single crystal pointing to lower stochastic impact on material strength. Thus, higher stresses of PS + SA material are assumed to be due to the higher initial dislocation density. It has been reported that the initial dislocation density of annealed polycrystalline TWIP steels is around 5×10^{13} m^{-2}, which is higher than the dislocation density of 7×10^{12} m^{-2} of the current as-grown single crystal [22,25]. This difference in dislocation density induces an increase in the yield stress by 23 MPa in Fe-22wt%Mn-0.6wt%C TWIP steel according to the Bailey-Hirsch equation [22,26] which is in good agreement to the difference of the measured average yield stresses of 22 MPa. Differences in the stress-strain response between both types of micro-pillars are also evident in the plastic deformation regime. The as-grown single crystal micro-pillars oriented for deformation twinning showed higher flow stresses and a more intermittent stress-strain response than the as-grown single crystal micro-pillars oriented for dislocation glide, which exhibited continuous flow with stable apparent work-hardening [21]. Higher flow stresses and unstable stress-strain response can be attributed to the activation of deformation twinning. The micro-pillars prepared from Grain 2 in the present study behave similar to the pillars oriented for dislocation glide prepared from an as-grown single crystal, although the orientation of Grain 2 is almost identical to that of the pillar oriented for deformation twinning in [21].

Fig. 2 shows the SEM and TEM micrographs of the microstructure of a deformed micro-pillar prepared from Grain 2. The micro-pillar contains a number of sharp slip traces on several slip systems (Fig. 2a). In contrast, the morphology of slip steps on the micro-pillar surface of the as-grown single crystal [21] was wide with shear-type steps. A TEM dark field (DF) image of the compressed micro-pillar prepared from Grain 2 shows that the activity of deformation twinning is low (Fig. 2b) even at an engineering strain level of 10% although the micro-pillar is oriented for preferential activation of deformation twinning. Only a few narrow twins are observed as indicated by arrows in Fig. 2a and b. Hence, it is concluded that the formation of large surface steps does not necessarily involve deformation twinning as confirmed by the DF image in Fig. 2b.

The TEM micrograph in Fig. 2c shows the region marked by the rectangle in Fig. 2b using the reflection condition $\boldsymbol{g} = [-1-11]$. The area corresponds to the large slip step on the left side of the deformed micro-pillar in Fig. 2a. Extended stacking faults, which are formed by the glide of partial dislocations on the primary slip system, have caused the formation of a huge slip step (see SEM micrograph in Fig. 2). No deformation twins are observed in this area. We conclude that pre-straining provides a higher number of dislocation sources, which mediate plastic deformation by emitting dislocations (Fig. 2). Only when these sources are exhausted deformation twinning may occur at higher stresses.

The effect of the activation of multiple slip systems on the deformation behavior in micro-compression tests is discussed based on the present observation as follows: A higher initial dislocation density is reported to induce a bulk-like deformation behavior without large strain bursts for example in Mo-alloy single crystalline micro-pillars [23]. Fig. 1, however, indicates that the number and size of strain bursts of micro-pillars prepared from the PS + SA Fe-Mn-C TWIP steel are similar to those observed in as-grown single crystalline micro-pillars [21]. Therefore, we suggest that the compression curve shows relatively stable and apparent work-hardening behavior with a continuous flow curve for Fe-Mn-C TWIP steel micro-pillars regardless of the activation of secondary slip system, when deformation twinning is not activated. This observation also indicates that twin-matrix bundles dynamically introduced by deformation twinning and their interaction with dislocations are a precondition for the observed strain hardening of TWIP steels.

Fig. 3 shows SEM and bright-field (BF) TEM micrographs of a deformed bi-crystalline micro-pillar containing a coherent TB. The activated slip systems are indicated by arrows and they correspond to the expected slip systems. The primary slip system of Grain 2 (P_2) formed slip traces visible in the SEM only in the lower part of the micro-pillar,

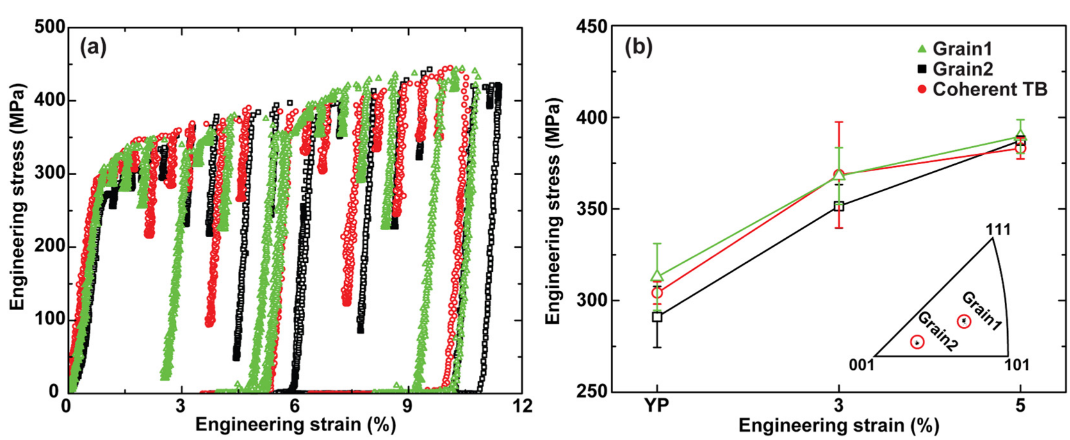

Fig. 1. (a) Engineering stress-strain curves of single crystalline and bi-crystalline micro-pillars containing a coherent TB. (b) Statistical comparison of stresses at the yield point (YP) and at engineering strain levels of 3% and 5%. The error bars give the standard deviations and the orientations of Grains 1 and 2 are presented as inset.