doi: 10.1017/S1431927609093635
Microsc Microanal 15(Suppl 2), 2009


# The Time & Space of Pico-Deformation: Revealed Upon Unloading by 3D-FIB-EM

W. J. MoberlyChan, B. S. El-Dasher*, & G. H. Campbell*; *LLNL-MSTD, Livermore, CA.

The kinetic laws that govern material properties may be defined as "whatever happens, happens; and whatever happens first, happens first" [1]. Except, at extreme temporal and spatial limits, sometimes happenings may have to wait until what happens afterward. Classically, a metal structure is loaded and at some level activated dislocations lead to plastic deformation. Except when the stress and strain rate are too high and/or the mobility too low (e.g. due to temperature and/or other imposed limits such as fine grain size), then dislocations may not have the time to move and deformation twinning may occur instead [2]. These classic strain-rate sensitive modes of deformation happen "upon" loading. At the extreme, however, deformation may wait to occur during the unloading. One modern example is the "pop-out" as a Nanoindentor is unloaded from a single-crystal silicon surface. In this work, FIB/SEM serial sectioning enables 3D microstructural study of deformation twins that occur upon unloading from localized laser shock processing [3].

A single crystal of Fe has recently been loaded to the extremes [3,4]. With high enough pressure (>10GPa) and fast enough loading rate (sub-nanosecond), no dislocations can move, and no twins can form, but rather a high-pressure phase transformation occurs (alpha-to-epsilon) [3]. Furthermore, the compressive shock wave travels so fast that new ε-phase nuclei cannot grow beyond nanometer scale [4]. Both the metastable phase and metastable size ensure that upon unloading the Fe recovers its single crystal stable phase. However, the unloading path is sufficiently slow to enable the multiplicity of the reverse phase transformation to play a role, and the recovered single crystal exhibits extensive twinning. These twins possess the same crystallography as classic deformation twins having occurred during loading, except these twins formed upon unloading. Static microscopies of post-recovery samples can observe microstructural morphologies that distinguish deformation that occurs upon unloading versus that which occurs during loading.

Figure 1 presents a 3-D reconstruction of the recovered microstructure as observed by scanning electron microscopy when serial sectioned by FIB. (Supplemental data includes a movie of this data. Time-resolved observation of microstructural differences at the nanometer scale can enable an interpretation of sub-nanosecond happenings [5].) Two intriguing observations concern these twins: they have extensive regular dimensions (microns long but spaced a few hundred nanometers), and yet their interfaces atomically meander. Twin formation during fast loading (shock) typically results in smaller twins, as the growing twins are interrupted by time and space. Also the classic slow growth of a twin upon loading (albeit faster than dislocation multiplication and yet modeled via sequential passing of partial dislocations on each adjoining plane) ensures twin boundaries are atomically flat for long distances [2,6]. The present recovery from this extreme nanocrystalline ε-phase is sufficiently slow that a single growing twin can sequentially (and collectively) consume ε-phase over many microns. Yet the rate is sufficiently fast to disavow considerations of interfacial energy that ask for flat twin boundaries. Both high-resolution transmission electron microscopy and electron double diffraction have observed the extreme roughness of twin planes at the nanometer-scale for other recovered crystals [4].

Twins in a crystalline material typically begin and end at other boundaries, whether other twins or pre-existing grain boundaries. Since deformation twinning can be strain controlled, the twinning process can be sufficiently sequential that the concurrent work hardening can lead to a crystallographic rotation of twins the same as happens for grain orientations with dislocation multiplication [6]. However, inside this single crystal of Fe there are no pre-existing boundaries that start and stop twin growth. Furthermore, the multiplicative cross twins grow in amongst and around each other without trying to stop and start at other twin boundaries. Although this twin volume fraction in excess of 20% represents an even greater percentage of plastic hardening, the recovered twins are extremely straight and parallel at the micron scale. All of these twin inconsistencies: apparent random start-&-stop, very crystallographically straight and parallel yet high volume fraction, extreme meandering at the nanometer-scale yet very long twins that grow around cross-twins; all are a consequence of twins that grow during the unloading by consuming a sea of nanocrystalline ε-phase.

Characterization of the extreme (slow or fast, big or small) has many difficulties because of our naturally limited 30-frame-per-second observations. In essence, there are only two data points: one of what happens at the nanosecond of loading [3], and another of several analytical techniques but $>10^{13}$ nanoseconds later [4]. And continual improvement of ultrafast *in situ* characterization techniques (diffraction and microscopy) strive for the missing information of "what happens?" during all those picoseconds in between. By the time and spatial dimension that we observe, it is often likely that several things may happen at the nanoscale; and now when we view a deformation twin, we must consider whether it happened upon loading or unloading. An atomic-scale model of dislocation motion has an atom elastically rising up an energy barrier and then plastically dropping down the backside; thus at the nanoscale, deformation upon unloading is not so extraordinary. A nanosecond of load can now be judiciously probed by XRD [3] to ensure that nothing happened upon loading, and the post-recovery microstructure can be 3D reconstructed to establish how deformation occurred upon unloading. In the end, deformation happens; however, this recovered material exhibits ideally spaced and shaped twins with an "interlocking" [7] microstructure that bodes well for ultimate strengthening and site-specific work hardening.

Ref. 1 Tiller, W.A. The Science of Crystallization: Vol II (in compiled class notes), Cambridge Univ. Press, (1991).
Ref. 2 Cahn, R.W., "Plastic deformation of alpha-uranium: twinning and slip." Acta Met., V1, 49-62, (1953).
Ref. 3 Hawrekiak, J., et al. "X-Ray diffraction of α-ε transition in shocked iron." Phys. Rev. V74, 184107-1-16 (2006).
Ref. 4 El-Dasher, B., et al. "Crystal recovery of iron shocked beyond the kinetic bound of plasticity." in submission.
Ref. 5 MoberlyChan, W.J., A.E. Gash, "Capturing Sub-nanosecond Quenching in FIB/SEM", EMC#14 643-644 (2008)
Ref. 6 Moberly, W.J. et al, "Deformation, Twins, & Strengthening", Acta Metall. Mater., V38(12) 2601-2611 (1990).
Ref. 7 Cao, JJ., et al. "In situ toughened SiC with Al-B-C additions." J. Am. Ceram. V79(2) 461-469 (1996).

Acknowledgements: A. Bliss, H. Lorenzana* for shock samples; DOE#DE-AC52-07NA27344. UCRL-JRNL-401867.

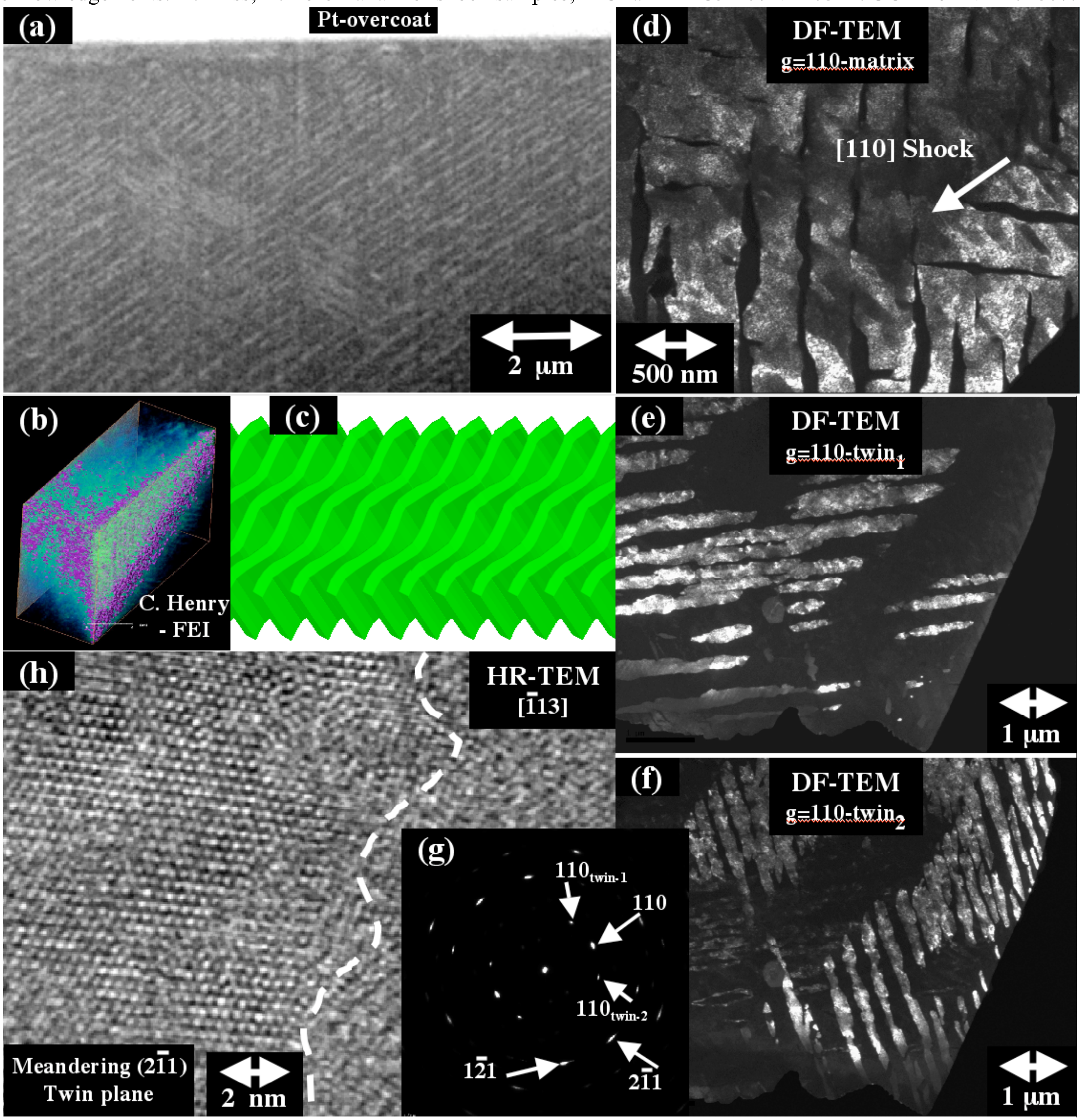


FIG. 1 (a) SEM snapshot from FIB-slice-&-view movie of Fe-[110] crystal shocked at ~11GPa [3,4]; slice normal ~[$\bar{1}$12]. (b) Voltex surface-view reconstruction (courtesy of C. Henry of FEI and Amira software). (c) Model of 1 set of parallel, but meandering twins. (2, 4 or 6 sets of intermingling twins are observed depending on shock orientation.) (d, e, f) DF-TEM (g=<110>) for matrix, tilted to [111]; and each set of twins, tilted to [$\bar{1}$13]. (g) SADP acquired along [$\bar{1}$13]. {2$\bar{1}$1} twin boundaries are also non-straight through the TEM-foil thickness Thus overlapping twins and matrix make widths appear different in DF images of same area, and lead to 1/3 reflections from double (and triple) diffraction. (h) At HR-TEM scale, even for thinnest prepared samples, twin boundaries meander and Moiré fringes appear [4].

https://doi.org/10.1017/S1431927609093635 Published online by Cambridge University Press