Characterization of Partitioning in a Medium-Mn Third-Generation AHSS

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Medium-Mn steels are considered third generation advanced high strength steels (AHSS), which contain both ferrite (α) and austenite (γ) and aim to balance the low cost of first generation AHSS with the outstanding mechanical properties of second generation AHSS. These steels generally exhibit an α' martensitic microstructure after hot and cold rolling but after inter-critical annealing have a dual-phase microstructure of α with a volume fraction of reverted γ that ranges between 20 and 50%. Mechanical properties of a third generation AHSS ideally exhibit low strain hardening rates at low levels of strain and higher strain hardening rates as total strain accumulates [1]. Studies investigating deformation mechanisms and mechanical properties of medium-Mn steels have reported the effects of inter-critical annealing temperature and phase fractions/sizes [2-5]. Cold-rolling medium-Mn steels prior to inter-critical annealing is necessary to retain γ [6]. The partitioning of Al from γ to α and partitioning of Mn and C from α/α' to γ during inter-critical annealing in the $\gamma + \alpha$ phase field controls both the amount and stacking fault energy (SFE) of the reverted γ. The SFE is important for transformation and twinning-induced plasticity (TRIP/TWIP). Characterizing partitioning in this multi-phase alloy will aid in interpreting the mechanical properties of the constituent phases. This study utilizes transmission electron microscopy (TEM), convergent beam electron diffraction (CBED) and scanning TEM energy-dispersive X-ray spectroscopy (STEM-EDS) performed using an FEI Tecnai Osiris equipped with a quad Super-X detector to characterize partitioning among the ultra-fine-grained (UFG) phases in a Fe-12Mn-3Al-0.06C steel.

An ingot of Fe-12Mn-3Al-0.06C (wt%) was hot-rolled at 900°C to 3.4 mm thickness, homogenized in an Ar atmosphere at 1100°C for 2 h, water-quenched to room temperature (RT) and cold-rolled to 1.7 mm. Thermo-Calc software predicts an equilibrium γ volume fraction of 0.45, a γ composition of Fe-20Mn-2.2Al-0.12C and a RT SFE ~20 mJ/m^2 following inter-critical annealing at 585°C. To study the effect of annealing time on phase fraction, samples were annealed for 0.5 and 8 h. Punched 3-mm discs were electropolished to electron transparency with a Struers twin-jet system (5% HClO$_4$, 35% [CH$_3$(CH$_2$)$_3$]-O-C$_2$H$_4$OH, 60% CH$_3$OH by volume, -30°C, 15 V). Bruker Esprit software was used to record 200 kV STEM-EDS hypermaps with typically ~40 μs dwell, 2-3 nm pixels, ~2 × 2 μm scanned area and ~20 frames for ~600 s acquisition. Processing included background subtraction, deconvolution and normalized wt% mapping. Data were also acquired with FEI TIA software. As seen in Figure 1, a sample annealed for 0.5 h contained mostly α, but also a decoration of Mn-enriched γ. CBED analysis was successful for samples annealed for 8 h [Figure 2(a-c)], but was too challenging for material aged for 0.5 h. In Figure 2(d), a processed line scan supports the Thermo-Calc predictions of γ phase enriched in Mn and depleted in Al. As γ grains were preferentially electropolished, Mn/Fe K$_\alpha$ peak ratios from spot analyses are useful for phase identification [e.g. Figures 1(a) and 3(a,e)]. Quantitative STEM-EDS mapping of samples annealed for 8 h at 585°C [Figure 3(b-d, f-h)] revealed a γ phase enriched to ~20 wt% Mn and depleted to ~1.3 wt% Al, thus matching the equilibrium Thermo-Calc predictions. Since quantifying carbon by STEM-EDS is impossible for the levels of interest, a concurrent atom probe tomography study is underway, in part to measure C partitioning. FIB lift-out specimens that mitigate the limitations imposed by the selective electropolishing and the ferromagnetism of self-supporting discs should allow composition profiles at (edge-on) phase boundaries to be obtained [7].

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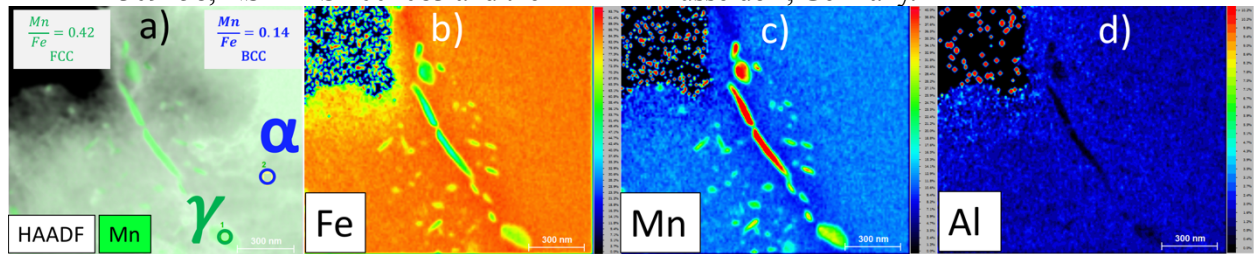[6] J Han et al., Acta Mater. 122 (2016) 199–206.

Figure 1: STEM-EDS hypermap of Fe-12Mn-3Al-0.06C annealed for 0.5 h at 585°C indicates a γ phase enriched in Mn and depleted in Fe and Al (39 μs dwell, 2 nm pixels, 1.6 × 1.3 μm scanned area, 19 frames). (a) Spot-EDS from marked locations distinguishes between α and γ phases via Mn/Fe K_α peak ratios. Quantitative maps for (b) 0-95 wt% Fe, (c) 0-40 wt% Mn, and (d) 0-10 wt% Al.
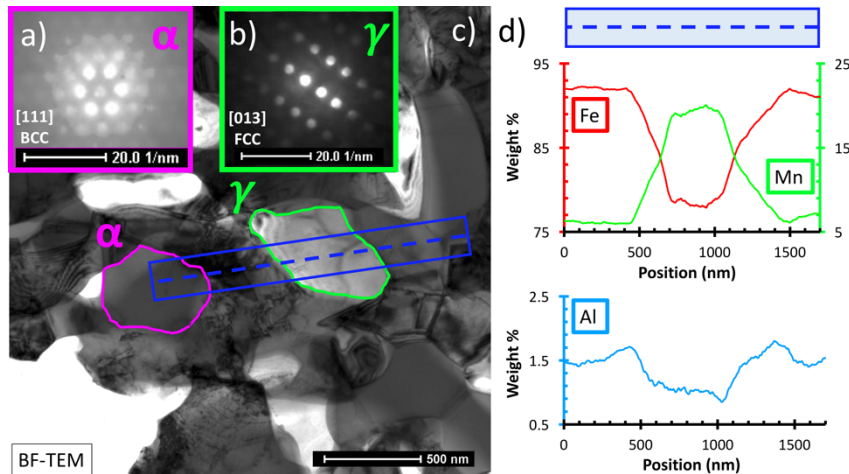

Figure 2: Fe-12Mn-3Al-0.06C annealed for 8 h at 585°C. 200 kV CBED patterns used to identify (a) ferritic (α) and (b) austenitic (γ) grains shown in (c) the bright-field TEM image. (d) Line scan [position marked on (c)], extracted from hypermap, across α and γ grains, highlights normalized differences in Fe, Mn & Al (wt%).
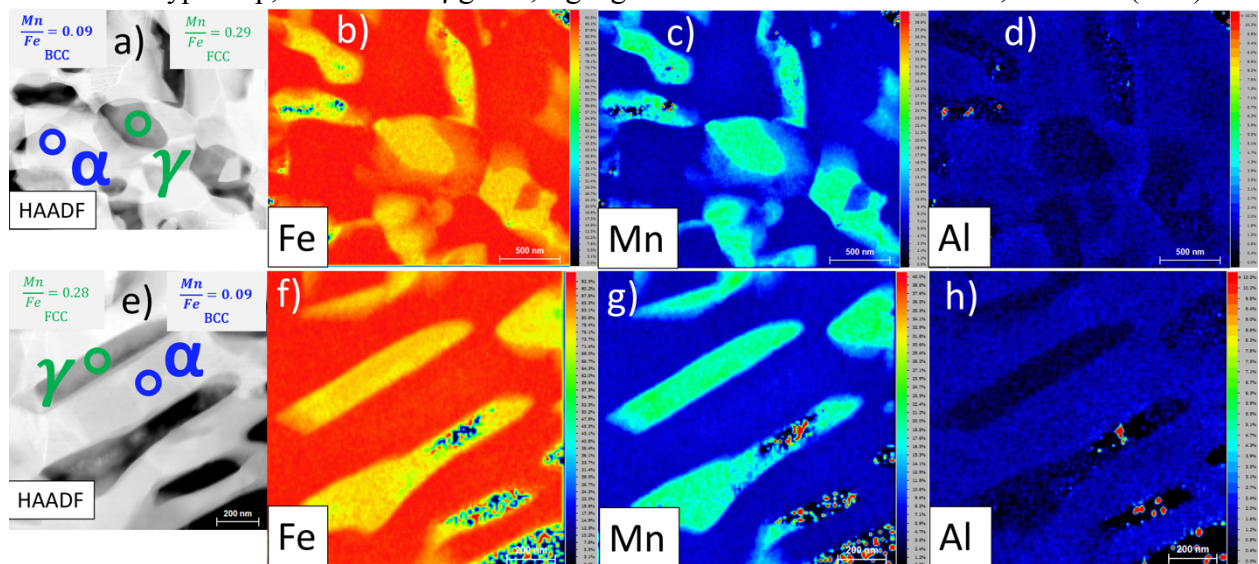

Figure 3: STEM-EDS hypermaps of Fe-12Mn-3Al-0.06C annealed for 8 h at 585°C indicates a γ phase enriched in Mn and depleted in Fe and Al. (a-d) 39 μs dwell, 3-nm pixels, 2.5 x 2.1 μm scanned area, 35 frames, (e-h) 34 μs dwell, 2-nm pixels, 1.2 x 1.2 μm scanned area,19 frames. (a,e) Spot-EDS from marked locations identifies γ and α phases via Mn/Fe K_α peak ratios. Quantitative maps for (b,f) 0-95 wt% Fe, (c,g) 0-40 wt% Mn, (d,h) 0-10 wt% Al.