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Atom Probe Tomographic Investigation of the Solute Segregation to Crystal Defects in γ-phase Co-35Ni-20Cr-10Mo Superalloy

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MP35N (Co-35Ni-20Cr-10Mo in wt.%) is a single γ-phase Co-based superalloy, which has a remarkable combination of high strength, ductility and fatigue resistance, coupled with good corrosion resistance. As such, it has found application from aerospace fastener material to the cables used for cardiac pacing and neurostimulation leads [1-2]. These leads are comprised of wires (~200 nm in diameter) that are typically manufactured through a cold drawing process, resulting in a submicron grained microstructure with a characteristically high amount of micro- and nano-twins. As these faults effectively obstruct the dislocation motion, the yield strength and ultimate tensile strength are significantly increased as compared to a fully recrystallized microstructure [2]. Subsequently, the wires undergo different processing steps, such as polymer insulation application, coiling, cable fabrication, etc. Interestingly, exposure to elevated temperature (such as during the coating process which lasts a few seconds to minutes) has been reported to also result in a secondary hardening [3], but with a significant sacrifice to ductility. The exact strengthening mechanism is still rather debated, with reports suggesting that it is due to either formation of secondary phases, martensitic reactions and solute partitioning thereafter, or segregation to crystal defects. Sorensen et al. [4] performed a detailed transmission electron microscopy (TEM) study of cold drawn and aged wires and did not observe any secondary phase formation or martensitic reactions. They stipulated that the secondary hardening is likely due to Mo segregation to stacking faults (SFs) and twin boundaries, based on STEM-EDS data. Plasticity assisted segregation has been a topic of interest in aerospace grade superalloys [5], mostly focusing on solute partitioning to faults of the $L1_2$ (γ′) phase lattice, with no reports on the fault segregation in the A1 γ matrix so far. Accurate partitioning data is very useful for alloy design as well as behavior modeling, as it can dictate the mechanical properties. In this study, we evaluate the partitioning behavior to lattice faults and the segregation-driven strengthening in a γ-phase superalloy via atom probe tomography (APT) with overarching goal of how to tune the composition to avoid embrittlement during aging.

A cold-drawn wire of MP35N was aged at 400ºC for 2 min and at 500ºC for 30 min. Several APT and TEM samples were prepared using an FEI Helios Nanolabs 600i focused ion beam (FIB). The APT microtips were polished with Ga^+ ions down to a ~50 nm radius using a final energy of 5 kV and a current of 16 pA. APT was conducted using a CAMECA LEAP® 5000 XR and running conditions set as: base temperature of -243°C (30 K), 40 pJ laser pulse energy, pulse repetition rate of 200 kHz, and evaporation rate of up to 1.0%. Transmission electron microscopy (TEM) was performed using a FEI Talos F200S microscope operating at 200 kV.

Fig. 1 (a) shows a TEM micrograph of a typical micro-twin observed in the MP35N material aged at 500ºC for 30 min, while Fig. 1 (b) is 3D reconstruction of a similar twin, where 12.55 #/nm^3 Cr isodensity surfaces are used to delineate the fault planes. A 2D Cr concentration map, Fig 1. (c), shows that in fact the micro-twin fault planes exhibit higher Cr content compared to the matrix. Interestingly, additional linear features (nano-twins) of high Cr concentration can be observed between the micro-twin fault planes, intersecting at ~53º. Some Cr depleted zones can be observed adjacent to the fault planes in both cases. A 1-D composition profile across these twinning planes shows that the Cr content at the fault plane is ~4 at.% higher compared to the matrix concentration in-lieu of Co and Ni, while the Mo content seems unchanged. A clear asymmetry of the segregation amount can be observed between the two micro-twin fault planes, which has been observed for twins in other systems [6]. Twin formation in face-centered-cubic materials involves shearing of Shockley partials on consecutive {111} planes, and similar segregation behavior observed in the γ′ phase of superalloys, has been proposed to be the remnants of the Co and Cr atmospheres of dislocation cores (Suzuki segregation) [7]. Interestingly, the fault planes of the γ phase lattice were devoid of any segregation in such γ′-containing superalloys. The segregation to the fault planes of single γ-phase MP35N even after short term temperature exposure (even 2 min at 400ºC) suggests that nearby Cr atoms diffuse to the fault planes, where they lower the fault energy. This study shows for the first time Cr segregation to fault planes in the γ phase of superalloys, and provides conclusive experimental evidence for the origin of the secondary strengthening observed in MP35N due to aging [8].

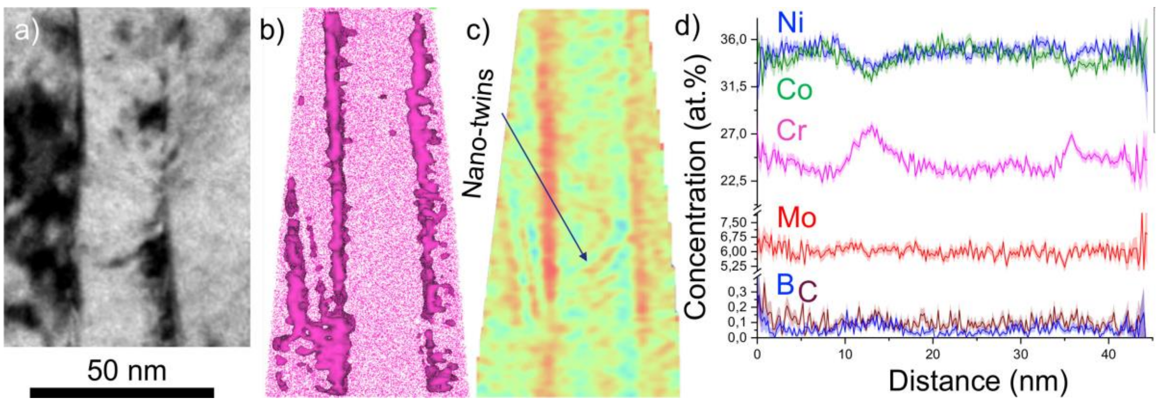

Figure 1. (a) Bright-field TEM micrograph showing a representative micro-twin, (b) 3D reconstruction of the Cr atoms along with 12.55 #/nm3 Cr isodensity surfaces representing the fault planes of a sample aged at 500ºC for 30 min, (c) 2D Cr concentration map showing the presence of nano-twins within the micro-twin, and (d) Concentration profile across the fault planes showing. Error bars are shown as lines filled with color and correspond to the 2σ counting error.

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