TEM characterization of martensite formation in nitrogen-stabilized precipitation hardenable steel

Martensite formation at sub-zero Celsius temperatures in nitrogen-added 17-4 PH stainless steel was studied. Two transformation conditions, namely athermal and isothermal, were tested. Samples transformed athermally showed plate-type martensite, whereas samples transformed isothermally exhibited typical lath-type martensite. The results reveal a connection between microstructure and the kinetics of martensite formation.

Precipitation-hardenable 17-4 PH martensitic stainless steel, belonging to a class of structural metastable stainless steels, is favored in the additive manufacturing (AM) industry [1]. Literature indicates that samples produced from nitrogen-atomized 17-4 PH powders exhibit a high amount of retained austenite in their microstructure [2], which influences strength by regulating the kinetics of martensite transformation. Consequently, there is a growing interest in understanding the effect of nitrogen on austenite stability and martensite formation in this steel. Research has demonstrated that martensite can form isothermally at sub-zero Celsius temperatures, challenging the conventional belief that martensite solely undergoes athermal transformation. Furthermore, suggestions have been made regarding the distinct morphologies of athermal and isothermal martensite [3].

The current study investigated this hypothesis by altering the martensite start temperature, Ms, of commercial 17-4 PH through control of interstitial nitrogen content via a high-temperature solution nitriding (HTSN) process, bringing Ms into the sub-zero Celsius range. For this investigation, the nitrogen content was fixed at 0.12 wt%. Two samples were prepared: one sample was quenched in liquid nitrogen (athermal), while the second sample underwent isothermal transformation at 210 K for 2 days. In-situ vibrating sample magnetometry (VSM) was employed to monitor the isothermal martensite formation, revealing a martensite fraction of approximately 5 vol%. The microstructure of the transformed samples was examined using electron backscatter diffraction (EBSD) and transmission electron microscopy (TEM). TEM specimens were prepared through focused ion beam (FIB) lift-out, highlighting areas in the EBSD maps. Additionally, TEM analysis included bright and dark-field imaging, along with diffraction. Results are depicted in Fig. 1.

A clear difference in microstructure between the athermal and isothermal samples was observed, with the athermal sample exhibiting a plate-type morphology, as evident from the dark-field (DF) images of TEM, whereas the sample transformed isothermally displayed a typical lath-type morphology with thin layers of austenite sandwiched between martensite laths (cf. TEM results). These results indicate that martensite formation at sub-zero Celsius temperatures is thermally activated. The current investigation highlights a clear connection between the kinetics of martensite formation and the resulting martensite morphology, suggesting that isothermal or thermally activated martensite formation in steels is more of a rule than an exception.

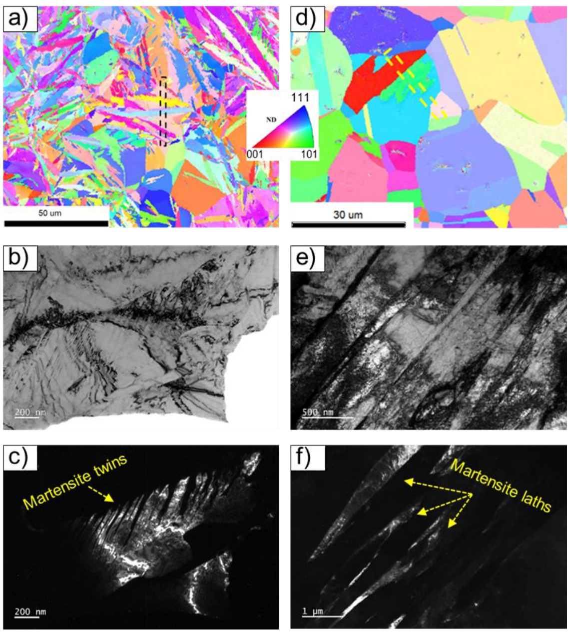

Fig 1: a-c) EBSD and TEM results of martensite formed in sample athermally transformed by quenching in liquid nitrogen, and d-f) for sample transformed isothermally at 210 K for 2 days.

References

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