Microstructural Characterization of 321 austenitic stainless steel below ambient temperatures

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Modern engineering applications demand material solutions with enhanced properties to operate at extreme temperatures and environments. From simple everyday utensils to challenging storage and transportation systems for liquid nitrogen and liquefied natural gas and even space exploration applications require tailored-made materials solutions. Austenitic stainless steels in the 300 series combine excellent corrosion resistance, weldability, formability, high strength and therefore are widely used in demanding sectors and industries. Moreover, they exhibit high structural integrity in a range of cryogenic to elevated temperatures. Below the ambient temperature, these attributes lay on microstructural mechanisms’ characteristics, the effect of plastic deformation on the microstructure and finally phase transformations of metastability related austenite γ (fcc) phase to deformation (strain or stress) induced ε (hcp) and α’ (bcc) martensite phases [1], [2]. The metastability-related martensitic phase transformations are complex mechanisms, which are governed by temperature, stress, strain, chemical composition, crystallographic and texture features, as well as stacking fault energy and significantly affect the mechanical properties [3]. Therefore, it is well established that, in order to utilize such steels to their full potential (up to 2GPa) and design new generation alloys, it is vital to have profound knowledge on metastable martensitic phase transformations. The purpose of this research is to perform a full characterization of attained microstructures, leading to the understanding of phase transformations that occurred, owing to the plastic deformation and to investigate the metastability threshold of 321 stainless steel grade.

An AISI 321 cold rolled annealed stainless steel plate of 3mm thickness was studied in the present work. Uniaxial tensile tests of standard dog bone specimens were performed on an Instron 4482 electromechanical tensile machine, with a specially designed experimental set up of a controlled environmental chamber under 3 different temperatures of 20oC (ambient), -50oC (liquid, a mixture of ethanol - liquid nitrogen) and -190oC (liquid nitrogen only). A constant strain rate of 0.02 s-1 and a constant temperature was employed, using immersion thermocouples. Microstructural characterization was carried out, following a standardized metallographic procedure of preparing cross-sectioned samples within the uniform plastic deformation area adjacent to the fracture point, employing Scanning and Transmission electron microscopy, using a Jeol 6380LV SEM and a Jeol 2100HR TEM, operating at 200kV.

The obtained engineering stress-strain curves (Fig 1.a) show a major increase in the ultimate tensile strength with a minor drop in elongation and a total increase of tensile fracture toughness. Of great importance is the higher strain hardening starting at 10% and 5% elongation thresholds, as a function of the temperature decrease at -50oC and -190oC respectively, due to the different nucleation mechanism of ε and α' martensite from strain-induced to stress assisted respectively. Backscatter electron micrographs (Figs 1.b-d) reveal the plastic deformation characteristics of the austenitic matrix such as elongated grains, sites of ε martensite nucleation, such as deformation twins and slips bands, and submicron irregular blocky
martensite nucleation in a manner of crystallographic order and stress-strain oriented direction. A major formation of α’ martensite is exhibited at the lowest deformation temperature (-190oC) and is justified by the higher tensile strength (1331 MPa), as well as, the minor loss of ductility (44%). TEM bright and dark field electron micrographs (Figs 2.a-b) of the sample tested at -190oC confirms the existence of irregular blocky α’ martensite nucleation from dislocation pile-ups intersections on active slip planes of banded microstructure. This effect stems from deformation slip lines, twins and ε martensite, while ε martensite is promoted at the early stage of transformation by shear displacements of twinning and extensive stacking faults formation, that is even challenging to trace and distinguish it even with TEM [4].

This research gives valuable insights into the martensitic transformation mechanisms of austenitic stainless steels, through electron microscopy characterization and the confirmation of TRIP effect mechanism accompanied by high strain hardening capacity due to deformation twins and ε martensite with lowering the deformation temperature, but prolonged uniform elongation with resist necking and fracture.

![Figure 1.](image)

**Figure 1.** (a) Engineering Stress-strain of 321 stainless steel at different temperatures. (b-d) Backscatter electron micrographs of microstructure following tensile testing at 20oC, -50oC and -190oC respectively, showing (marked by white arrows) elongated austenitic grains with some large slip bands (b), additional and thinner deformation and slips bands, sites of ε martensite nucleation (c) and irregular blocky α’ martensite nucleation between deformation band intersections (d).
Figure 2. (a-b) A pair of bright and dark field (weak beam of $\alpha'$ martensite twin reflection) electron micrograph of tensile tested specimen at -190°C, showing mostly irregular blocky $\alpha'$ martensite nucleation between deformation bands of austenitic matrix.

References