STRENGTH OF NANOSTRUCTURED AUSTENITIC STEEL 316LN AT CRYOGENIC TEMPERATURES

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Abstract. The aim of this work was to investigate the effect of nano-refinement on the properties of austenitic steel. The material with the initial grain size of 40-50μm was subjected to hydrostatic extrusion at a room temperature to the total accumulated strain exceeding 1. The microstructure developed was investigated by Transmission Electron Microscopy (TEM) and Focus Ion Beam (FIB). The strength of the extruded samples was tested at 293K, 77K and 4.2K by means of cryostat for static tensile tests. The results show that the hydrostatically extruded steel 316LN has excellent strength in cryogenic conditions, which make this material interesting for applications in cryogenic devices.

1. Introduction

Austenitic stainless steels are common structural materials for cryogenic elements such as elements of fusion devices [1, 2]. Although strength of austenitic steels increases considerably with a decrease of temperature, it is still insufficient for many applications, and other alloys, such as Ni-Cr superalloys, are also used. Components made of dissimilar materials thermally deform in less uniform way what leads to disturbances of stress field. It justifies efforts to increase strength of austenitic steels which can be substantially improved by microstructure refinement down to the nanometre scale. Studies of nanocrystalline and nanostructured austenitic steels tested at room temperature [e.g. 3-5] have shown that its strength can achieve values higher than 1300 MPa. The aim of the present study was to investigate mechanical properties of nano-austenitic stainless steel at cryogenic conditions relevant for fusion applications, following the results obtained for pure metals in [6, 7].

2. Material and methods

The material studied was 1.4429 stainless steel with chemical composition given in Tab. 1. Billets with initial diameter of 12 mm, 9 mm and 7 mm were subjected to Hydrostatic Extrusion, HE, with total accumulated strain of 0.25, 0.84 and 1.36 (denoted as HE_0.25, HE_0.84 and HE_1.36). As has already been reported in [5] hydrostatic extrusion with the total accumulated strain exceeding 1.0 transforms standard austenitic stainless steel into nano-twinned with the size of twins in the range
below 100 nm. In the context of fusion applications it can be noted that the extrusion can be used to fabricate such parts as bolts, pins and shafts [8, 9] which in fusion devices transmit heavy loads [10].

| Ni  | Cr  | Mo  | Mn  | Cu  | N   | C   | Si  | P   | S   |
|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| 12.6| 17.2| 2.66| 1.15| 0.15| 0.15| 0.023| 0.53| 0.065| 0.013|

Table 1 Chemical composition (wt. %) of investigated steel 1.4429

The microstructures of the processed billets have been investigated by Transmission Electron Microscopy\(^1\) and Focus Ion Beam Microscopy\(^2\) using electrochemically polished samples. A representative TEM image of the material with total strain of 1.36 is shown in Figure 1. It can be noted that this microstructure consists of shear bands and deformation twins with thickness of tens of nanometers (Fig. 1). The volume fraction of twins was assessed by analysis of images obtained by FIB microscopy employing the channelling contrast. The results are plotted as an insert in Fig. 2 against microhardness of the HE processes samples in room temperature\(^3\). Assuming that the density of twins in the as-received samples is negligible, a nearly linear dependence of the resistance to the plastic flow on the density of twin is observed as already reported for fcc NiMn alloy in [11]. A profound increase in dislocations density is also observed, as recovery of processed material is limited due to very high strain rate (about 100 1/s).

Since some austenitic steels undergo partial phase transformation into ferromagnetic α′-martensite at room temperature during severe plastic deformation, it should be noted that in the current case no effects of γ→α′ transformation have been revealed by means of Hall sensor. The fact that HE material remains paramagnetic is important if the material is foreseen to work in the strong magnetic field.

Strength of hydrostatically extruded steel 1.4429 was measured using standard M6 specimens with gauge diameter of 3 mm and parallel length 20 mm, submerged in liquid helium inside a vacuum insulated cryostat adapted for tensile tests. The straining temperature was about 4.2 K. The initial strain rate was 10\(^{-3}\) s\(^{-1}\).

\(^1\) JEOL JEM 1200X microscope, acceleration voltage 120kV,
\(^2\) Hitachi NB5000 microscope, Ga ions, acceleration voltage 40kV
\(^3\) Vickers microhardness determined using an Anton Paar MHT10 unit under load of 200 g
3. Results and discussion

Representative engineering stress-strain curves obtained at 4.2 K are presented in Fig. 3. All batches reveal serration effect, therefore increase of strength can be assessed using discontinuous yield strength (DYS) value, which corresponds to the stress level at the top of first relaxation tooth. Comparing the curves obtained for HE_1.36 and as-received material, one can note 80% growth of DYS up to about 2.6 GPa.

As expected, the increase in the strength is accompanied by decrease in ductility caused by early localization of plastic deformation. However, scanning electron microscopy observations of the fracture surfaces (Fig. 4) reveal that both as-received and strengthened materials have similar pattern of fracture at 4.2 K, with the main difference between these two in the ratio of the surface of the outer shear lips to inner fibrous zone. As a general rule, a shear zone becomes larger with the increase in the level of cold work and with the decrease in temperature. It can be concluded that HE processed samples in cryogenic temperatures are not inherently brittle and exhibit small strain to failure because of the strong tendency for strain localization. A strong tendency for strain localization within the neck during deformation of the HE material is caused by the limited ability of the material for further propagation of the plastic flow due to saturation of deformation mechanisms.

Bearing in mind fusion applications, the current study addressed also thermal expansion of the HE processed billets, as this parameter is of prime importance for cryogenic devices. According to the literature, the difference in thermal expansion coefficient between micro and nano-crystalline materials might be observed, mostly due to an increased volume fraction of grain boundaries [12-14]. On the other hand, in the currently studied material the population of the grain boundaries is dominated by twin boundaries, which should not change the thermal expansion. In order to measure the thermal expansion coefficient of the HE processed billets an experimental set up with attached strain gauges circuit was assembled which is described in more detail in [15]. The set-up was cooled down by immersion in liquid nitrogen, so the temperature difference employed in the measurements was 216 K. The corresponding difference in the value of the thermal expansion coefficient between as-received state and HE_1.36 material was measured as 0.24e-6 [1/K] with an experimental error 0.12e-6 [1/K]. The literature

![Fig. 3 Engineering stress-strain curves of steel 1.4429 at 4.2K](image)

![Fig. 4 SEM images of fractures of HE_1.36 and as-received material after tests at liquid helium temperature](image)
data indicate that the thermal expansion coefficient for the investigated steel is ~12.8e-6 [1/K]. Therefore one can conclude, that hydrostatic extrusion does not change thermal expansion of steel considerably.

Summarizing, excellent strength and limited content of ferromagnetic martensite phase are the main advantages of hydroextruded steel 1.4429, which makes this material competitive to high strength alloys used at cryogenic conditions.

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