The Mechanical and material properties of 316LN austenitic stainless steel for the fusion application in cryogenic temperatures

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Abstract. Due to the constant increase of claims for all materials used in superconducting magnets in „magnetic fusion reactors“, the article deals with the possibilities of increasing the mechanical properties of austenitic stainless steel tested at cryogenic conditions that ensure the transport of Helium to magnets. The aim of the experimental plan was to increase the mechanical properties of the steel grade 316LN tested at 4.2K from the original value Steel A: YS = 1045 MPa, UTS = 1528 MPa, A = 33 % to the value of YS = 1204 MPa, UTS = 1642 MPa, A = 34 % and Steel B: YS = 1173 MPa, UTS = 1541 MPa, A = 28 % to the value of YS = 1351 MPa, UTS = 1645 MPa, A = 17 %. The increase in mechanical properties of the steel grade under examination has been made by means of heat processing in the conditions of annealing: Texists = 625 °C / texists = 696 h. The mechanical properties of steel were evaluated using static tension tests at 4,2 K. The samples were placed in a cryostat filled with liquid helium. Except for the mechanical properties, there were also evaluated structural changes depending on the conditions of heat processing by light optical microscopy and EBSD (Electron Backscatter Diffraction). The increase of steel properties used in low temperatures was achieved by heat processing.

1. Introduction

For large-scale superconducting magnets the so-called cable in conduit conductor (CICC) allows a structural reinforcement of the bare superconducting NbTi or Nb₃Sn strands by austenitic stainless steel pipes [1]. In relation to this specific material and its characteristics, 316LN austenitic stainless steel is a prospective material for fusion reactor due to its resistance to neutron irradiation and corrosion as well as excellent mechanical properties, exemplified by high temperature strength, toughness and ductility [2].

Nitrogen addition resulted in the improvement of mechanical properties, related to short range order SRO, retardation of dynamic strain aging DSA, microstructure change from cellular dislocation to planar dislocation structure, decrease of grain size and carbide precipitation at grain boundaries. Importance of these factors is dependent on stress as well as temperature which was applied during the testing period [2].

316 LN Stainless steel was used as a jacket material for the Nb₃Sn conductors [3]. In relation to this fact, cold working effect as well as annealing on jacket’s mechanical properties have to be investigated due to conductor compaction, bending and annealing, after the superconducting cable insertion into
the jacket. Moreover, since the CICC operates at about 4.5K, it is inevitable to evaluate mechanical strength at low temperatures. Mechanical properties of a stainless jacket with 316LN differs greatly from the 316L stainless steel [4], having lower elongation at failure but simultaneously higher mechanical strength. During the cryogenic test, the primary minimum requirement to be achieved on elongation for TF jacket was 30%. However, it has been proven by the experiments on different jacket materials that high elongation at failure can be reached with certain difficulties. As a result, the minimum value to be achieved was reduced to 20% and the requirement for elongation was modified as well.

However, the measurements were performed on the sub-size samples. Therefore, this study deals with the comparison of results from tensile tests as well as with the evaluation of mechanical properties and material properties in the cryogenic temperatures [5].

2. Experimental material and procedure

2.1. Experimental material

For the purposes of this study, we used the seamless tube, which is made of the modified 316LN austenitic stainless steel. The carbon content was reduced to less than 0.02 (compared to C value which is the norm - less than 0.03%) to reduce the degradation in ductility after the reaction of the Nb3Sn superconductor to heat treatment. The chemical composition of the modified 316LN austenitic stainless steel is mentioned in the Table 1. In order to simulate the magnet manufacturing process, the experimental tubes (original outer diameter of 48 mm) were compacted and tensile stretched 2.5% in steps at room temperature. After the magnet manufacturing process simulations, the final dimensions of the jacket were 43.5 mm outer diameter and 2.0 mm wall thickness, as mentioned in the Figure 1. After these procedures, experimental tubes were put into the furnace for aging with argon atmosphere. To specify the parameters of the heat treatment (aging), it is necessary to state that the temperature was 625°C and the duration was 696 hours. The reason for this aging procedure was to simulate the Nb3Sn formation process.

The sub-size specimens were cut by electron discharge machining (EDM) according to ASTM E8M standard, as it can be seen in the Fig.2. The dimensions of the specimens were 12.5 mm x 2 mm in the reduced section and the gauge length was 50 mm. Every test series consisted of at least three specimens in longitudinal direction.

| Spec. ID | C    | Si   | Mn   | P    | S    | Cr   | Ni   | Mo   | N    | Fe   |
|----------|------|------|------|------|------|------|------|------|------|------|
| B        | 0.013| 0.152| 1.668| 0.019| <0.005| 17.25| 13.71| 2.537| 0.174| Bal. |
| A        | 0.011| 0.332| 1.79 | 0.006| <0.005| 17.36| 13.58| 2.671| 0.131| Bal. |
2.2. Test conditions
The sub-sized specimens were tested by an ATLAS universal testing machine with the load capacity of 650 kN, including the cryostat system inbuilt in the machine. Working temperature of the machine is down to 4.2 K. The sub-sized samples were immersed into the liquid helium in order to reach the given temperature 4.2 K. Tensile tests were conducted in displacement control with a strain rate of below $5 \times 10^{-4}$ s$^{-1}$ according to ASTM E8M. The material properties of the tested samples were examined using the scanning electron microscope with the EBSD camera (ZEISS LEO 1530) and the optical microscopy (ZEISS AXIO Vert. A1). For the analysis of chemical elements, we used the machine Bruker Q4 Tasman (optical emission spectrometer) and for the analysis of nitrogen content, we used the Bruker G8 Galileo (inert gas fusion analyser).

3. Experimental results and discussion

3.1. Mechanical properties
Mechanical properties of 316LN austenitic steel for RT measured at work [5] were compared with experimental materials A and B, which were tested at 4.2 K, shown in the FIG. 3. The measurements indicate that the value of yield and ultimate tensile strength measured at 4.2 K approximately doubled as compared to similar materials tested at RT and vice versa, total elongation demonstrated the opposite trend. By decreasing the temperature to 4.2 K, the values of total elongation were decreased for both tested materials of approximately 11 %. It was also observed that the material B has higher values of yield strength than the material A - of 128 MPa after the measurement at 4.2 K. For ultimate tensile strength and total elongation were not measured significant differences in both tested materials. Better mechanical properties (YS and UTS) of B versus A material can be attributed to the higher content of nitrogen in the chemical composition at the level of 0.17 % (material B) compared to the
material A, with the proportion of nitrogen 0.13 %. FIG. 4 shows the mechanical properties of both tested materials at 4.2 K, depending on the impact of compaction (CO) and 2.5 % tensile stretching (TS) carried out at RT (cold working), followed by aging (AG). The major influence on the value increase of yield (up to approximately 15%) and tensile strength (up to approximately 7 %) was from CO + TS. It is possible to observe the influence of aging on the values of total elongation at which the material B has its value decreased by 10 % [6].

3.2. Structural analysis
In Fig. 5 is shown the microstructure of tested austenitic steel for the initial testing materials (Fig 5 a,b) as well as for selected testing material compacted and tensile stretched and aged (Fig 5 c,d). The microstructure is shown in the longitudinal direction of the pipe, and it is formed by polyedric austenite grains (when twin boundaries are not considered as twin boundaries) and high fraction of Σ3 twin boundaries. The average size of austenite grains (determined from the optical microscopy) was at the initial basic material A and ranged from $d_\gamma = 50-140 \mu m$, wherein after CO + ST + AG, their size does not change significantly ($d_\gamma^{TS+CO+AG} = 60-160 \mu m$) which means the austenitic structure was well stabilized by precipitates. The cover Size B is observed a heterogeneous particles size, exhibiting patterns of bimodal division ($d_\gamma = 20 - 250 \mu m$), but the material does not exhibit a change in the size of austenite grain size after the heat treatment.
The presence of the second phase was observed on both materials in the form of unidentified particles of white and black colour which were located evenly on the borders of austenite grains (Fig. 6). It can be stated (from works of various authors [6,7]) that the found particles are precipitates M\textsubscript{23}C\textsubscript{6} carbide (white elements) and M\textsubscript{6}C\textsubscript{6} (black elements). The reduced content of M\textsubscript{6}C\textsubscript{6} in the microstructure, as opposed to M\textsubscript{23}C\textsubscript{6}, can be attributed to the large consumption of carbon, necessary for its creation.

**Figure 5** Band contrast map and Σ3 - Twin Boundaries (white lines), longitudinal direction of the tube = horizontal: (a) A; (b) B; (c) A after CO+TS+AG; (d) B after CO+TS+AG

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**Figure 6.** SEM picture of the material A after CO+TS+AG
4. Conclusion

This work dealt with the assessment of the mechanical properties of two chemical concepts 316LN austenitic stainless steel in the cryogenic temperatures; down to 4.2 K. In relation to measurements, it can be stated that the increase in yield and ultimate tensile strength (in the cryogenic temperatures) results from the impact of the increasing nitrogen content, without significant impact on the total elongation. Further increase of mechanical properties was achieved after the application of compaction, tensile stretching and aging. Testing steel microstructure was composed of polyedric austenitic grains with twins and precipitates. These materials achieved very good results, when considering the work in cryogenic temperatures, and therefore could be possibly applied in the area of fusion in the future.

Reference

[1] Mitchell N, Devred A, Libeyre P, Lim B, Savary F 2012 IEEE TASC 22 4200809
[2] Kim D W, 2012 J. of Nuc. Mat. 420 473-478
[3] ITER, Procurement specification for the jacketing of the cables for ITER Toroidal Field (TF) coil conductors. ITER design document. Revision V-01, April, 2005
[4] Liu H et al 2011 Mechanical tests on the ITER PF 316L jacket after compaction Cryogenic 51 234–236
[5] Wu Z et al 2013 Fusion Engineering and Design 88 2810-2813
[6] Sgobba S et al 2013 Fusion Engineering and Design 88 2484-2487
[7] Liang Z et al 2015 High. Temp. Mar. Proc. aop