Effect of Niobium/Molybdenum microalloying on SS316LN steel.

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***Abstract*— In recent years SS316LN microalloyed stainless steel is preferred for use as jacket material for Nb_3Sn superconductor strands/wires. In the present investigation, microalloyed SS316LN is prepared in a vacuum induction melting furnace; Niobium and Molybdenum in their ferroalloy stage are considered as alloying element. This microalloyed steels are cast in water cooled copper mould. The tensile strength and elongation are measured and the fracture surface is studied under scanning electron microscope. It is observed that, there is a reduction of tensile strength and decrease in hardness of the steels prepared with addition of either/both the alloying elements; however there is an increase in ductility, which is helpful for cold rolling operation. From the micrographs it is observed that nitride precipitates are formed along the grain boundary, but formation of chromium carbide precipitates is reduced.**

Keywords-Precipitation hardening; microalloying; jacket material; CICC superconductor.

I. INTRODUCTION

SS316LN is a nitrogen-strengthened version of Type 316L stainless steel. By means of solid solution strengthening, nitrogen provides significantly higher yield and tensile strength than Type 316L, without adversely affecting the ductility, corrosion resistance and non-magnetic properties. This grade stainless steel is being widely used as a structural material in nuclear reactors in view of its high temperatures mechanical properties viz. strength, ductility, toughness and corrosion resistance [1]. It also possesses good fabricability and weldability properties. Due to these properties SS316LN is used as jacket material for Nb_3Sn-type CICC superconductor strands. The reason being, J_c and T_c properties of Nb3Sn are strain sensitive and the thermal expansion of SS316LN between 700^0C to -269^0C which matches with that of Nb_3Sn superconductor [2, 4].

The jacket is put/covered on the strands which are flexible. Because, both the jacket and strands must pass through the reaction heat treatment required to form Nb_3Sn i.e. in range of 600-800 ^oC for 150-200 hours. But whenever the stainless steels are heated at this range of temperature,major microstructural degradation occurs due to precipitation of compounds like chromium carbide ($Cr_{23}C_6$) at the grain boundaries and later inside the grains [3]. This sensitized condition reduces corrosion resistance, ductility, toughness and the stability of the jacket material due to martensitic transformation. This leads to transgranular dimples and intergranular facets at cryogenic temperature, thus causing severe embrittlement [2, 6].

$$\gamma \rightarrow \alpha' + \varepsilon \qquad (1)$$

(The fcc austenitic (γ) partially transforms to bcc alpha (α') and hcp epsilon (ε) martensite phases)

Micro-alloying is one of the solutions for sensitization as it affects the diffusion rates of elements for formation of precipitates. Nb and Mo improves the performance of SS316LN by retarding the nucleation and growth of $M_{23}C_6$ intermetallic precipitates along the grain boundaries, due to formation of nitrides like CrNbN, Cr_2N and $CrMoN_x$ by micro-alloying.

II. EXPERIMENTAL

The alloys are prepared in the vacuum induction melting furnace at a vacuum level of 10^{-6} torr. The process is selected because high vacuum treatment of metals or alloys is susceptible to oxidation. Before starting of experiment, Nb or Mo was added from their ferroalloy stages. In this operation the atmospheric control (N_2) was maintained. The temperature was maintained up to maximum 1800^oC. Finally the materials were cooled up to very low temperature and then recovery process was done successfully. Table I shows the chemical composition of the starting material (i.e. SS316LN), detected by LeCO made GDS500A OES. The composition of the cast ingots with various percentages of additions of Mo/Nb is given in Table II.

Tensile tests are carried out using INSTRON 1195. Circular cross-section samples with gage length 20mm and diameter 4mm were prepared and each data point is average of tests of 4 samples.

gram. Five hardness measurements were made for each specimen.

III. RESULTS AND DISCUSSION

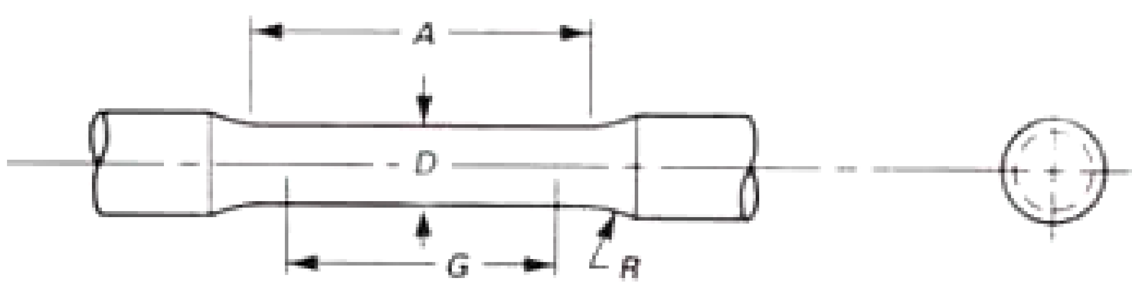
The results obtained from tensile test are shown in Table III. From the data it is found that the UTS of plain SS316LN is highest. When 0.07 % Nb is added the UTS and percentage elongation value decreases to a significant amount. However, the UTS and percentage elongation increases by increasing the percentage to 0.15 %. Also addition of 0.07% Mo, the UTS decrease but the elongation percentage increases.

TABLE I. CHEMICAL COMPOSITION (wt %) OF SS316LN BY OES

C	Mn	Si	P	Cr	Ni	Mo	Cu	V	Al	Nb
0.028	0.645	0.358	0.039	16.2	10.7	2.12	0.371	0.06	0.009	0.026

TABLE II. AMOUNTS (wt %) OF Nb/Mo ADDED DURING MELTING

Sl No	Wt of raw material (kg)	Alloying element (gm)	Wt % of Additive	Wt of Ingots (kg)
1	4.318	4.21 (Fe-Nb)	0.07	3.65
2	4.075	4.88 (Fe-Mo)	0.07	3.487
3	4.148	4.04(Fe-Nb) 4.96(Fe-Mo)	0.07 0.07	3.8
4	4.325	9.058 (Fe-Nb)	0.15	4.175
5	3.1	7.936(Fe-Mo)	0.15	2.07
6	4.3	9.05 (Fe-Nb) 11.001(Fe-Mo)	0.15 0.15	4.25



G-Gage Length = 20mm, D-Diameter = 4mm, A-Length of reduced section = 24mm, R-Radius of Fillet = 4mm

Figure 1. Tension test specimen with 20 mm gage length

Hardness test is conducted in a Vickers hardness tester, LeCO make. The Vickers hardness numbers (VHN) of the polished specimens were measured under a load of 100

TABLE III. TENSILE STRENGTH, YIELD STRENGTH AND ELONGATION

Material	0.2% Yield strength, σ^{YS}(MPa)	Tensile strength, σ^{UTS} (MPa)	Elongation (%)
SS 316 LN	307.6	658.1	56.4
SS 316 LN + 0.07 % Fe-Nb	248.9	494.9	26.61
SS 316 LN + 0.07 % Fe-Mo	240.7	536.4	61.31
SS 316 LN + 0.15 % Fe-Nb	260.7	571.6	62.25

From fig. 2, graph, it is observed that the tensile strength and yield strength decreases after micro-alloying but percentage of elongation mostly increases. This implies that the ductility increases after micro-alloying. From the SEM figure, (fig.3) it is observed that the fracture mode is ductile type evidenced form formation of dimples rather than inter-granular fracture which is observed in case of SS316LN.

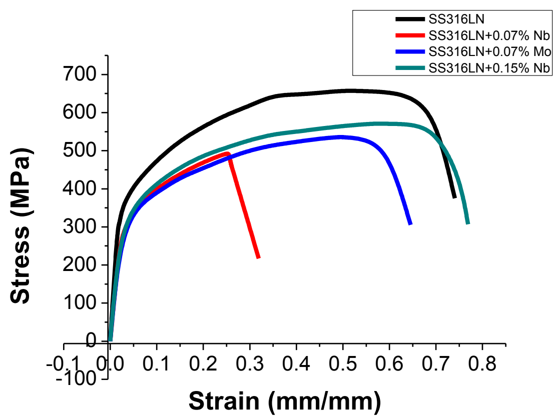


Figure 2. Stress - strain curve for different samples.

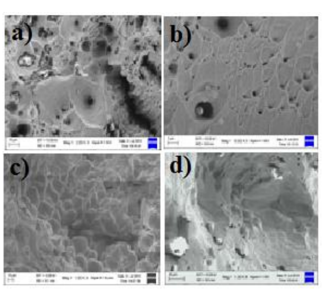


Figure 3. SEM of fracture surface of a) SS316LN, b) 0.07% Nb c) 0.07% Mo, d) 0.15% Nb% alloyed.

The result of hardness test in $HV_{0.1}$ is given in table IV and a graph was plotted between percentages of alloying element vs hardness given in fig. 4.

TABLE IV. Hardness value in $HV_{0.1}$ of different alloys

Specimen	Hardness value ($HV_{0.1}$)
SS316LN	216
SS316LN+0.07%Nb	180
SS316LN+0.07%Mo	173
SS316LN+0.07%Nb+0.07%Mo	168
SS316LN+0.15%Nb	161
SS316LN+0.15%Mo	186
SS316LN+0.15%Nb+0.15%Mo	188

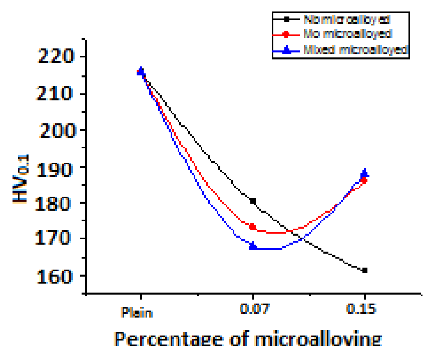


Figure 4. Variation of hardness with alloying addition.

From the result it is clear that, hardness decreases with addition of microalloying elements such as Nb, Mo and their mixed composition. This indicates that the matrix becomes more ductile by introduction of Nb and Mo. The role of Nb and Mo is known to prevent chromium carbide precipitation i.e. $Cr_{23}C_6$ in the grain boundary and inside the grains.

Microstructures of the as cast specimens are shown in fig. 5. Fig. 5(a) shows the optical microscopy of pure SS 316 LN as cast sample. Figure b, c and d show the as cast Fe-Nb(0.15%), Fe-Mo(0.15%) and Fe-Nb(0.15%)+ Fe-Mo(0.15%) added SS316LN sample respectively. Microstructural evolution of Nb and Mo microalloyed as cast SS316LN is quite different from original SS 316 LN i.e. second phase precipitation/segregation is observed for micro-alloyed samples. It is observed that, in case of a plain SS 316 LN, the grains are mostly equiaxed, whereas after Mo/Nb addition, rectangular grains with prominent grain boundary are formed. It is observed that in Nb added sample the second phase precipitates are cellular Cr_2N/CrNbN or inter-dendritic CrN. For Mo added sample $CrMoN_x$ precipitates are formed. Such observations are also been made by previous workers[6]. But the formation of $Cr_{23}C_6$ is reduced, because the properties of Cr are more similar to Mo.

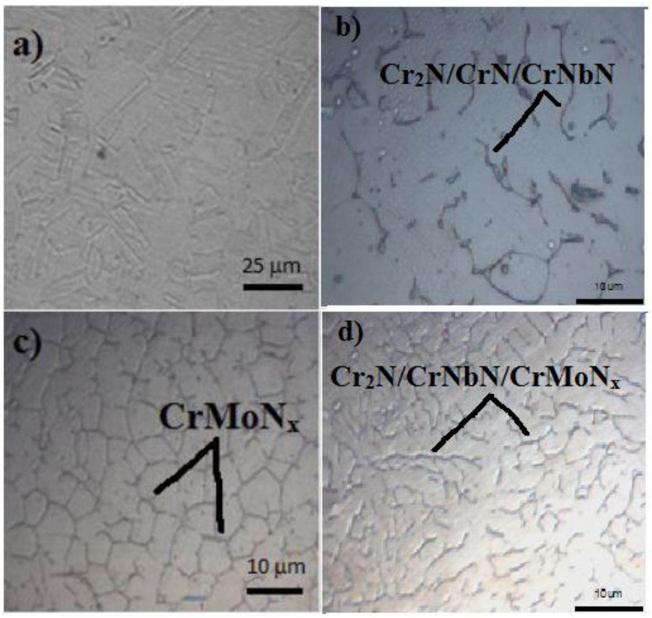


Figure 5. Optical micrograph of (a) SS316LN, (b) Nb micro-alloyed, (c) Mo micro-alloyed (d) Both Nb & Mo mocro-alloyed.

IV CONCLUSIONS

Microalloying of SS 316 LN by addition of Nb and Mo, though vacuum induction melting lowers the tensile strength (in case of as-cast samples); whereas elongation percentage is mostly found to improve. Similarly hardness also decreases with addition of Nb and Mo micro-alloying. This implies that, the alloyed samples become more ductile which is helpful for cold rolling, hence advantageous in the production of superconductor jacket strips.

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