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PHASE STABILITY OF HIGH MANGANESE AUSTENITIC STEELS FOR CRYOGENIC APPLICATIONS

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Abstract

The aim of this work is to study the austenitic stability against α' martensitic transformation of three non-magnetic austenitic steels : a new stainless steel X2CrMnNiMoN 19-12-11-1 grade, a traditional X8CrMnNiN 19-11-6 grade and a high manganese X8MnCrNi 28-7-1 grade.

Measurements of relative magnetic susceptibility at room temperature are performed on strained tensile specimens at 4.2 K.

A special extensometer for high precision strain measurements at low temperature has been developed at CERN to test specimens up to various levels of plastic strain. Moreover, the high precision strain recording of the extensometer enables a detailed study of the serrated yield phenomena associated with 4.2 K tensile testing and their influence on the evolution of magnetic susceptibility.

The results show that high Mn contents increase the stability of the austenitic structure against α' martensitic transformation, while keeping high strength at cryogenic temperature. Moreover, proper elaboration through primary and possibly secondary melting maintains high levels of low temperature ductility.

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1. INTRODUCTION

High manganese austenitic steel grades are promising structural materials for cryogenic use down to liquid helium temperature (4.2 K). They are selected for different components of the superconducting magnets of the Large Hadron Collider (LHC) to be built at CERN. LHC, operating at 1.9 K, will provide 7 TeV proton-proton collisions. For these LHC components, low relative magnetic susceptibility at cryogenic temperature is required. At 4.2 K in the annealed state, high Mn steels retain high strength, ductility and a relative magnetic susceptibility generally lower than $5\cdot10^{-3}$. Due to possible magnet quenches (the conductive-to-resistive transition of superconducting magnets), structures may also suffer thermal cycling and local deformations at cryogenic temperature. For this reason, the stability of the austenite vs. spontaneous and strain-induced α' martensitic transformation is of major concern.

The aim of this work is to study the austenitic stability against α' martensitic transformation of three non-magnetic austenitic steels: a new stainless steel X2CrMnNiMoN 19-12-11-1, a traditional X8CrMnNiN 19-11-6 grade and a X8MnCrNi 28-7-1 grade.

The first grade was specially designed to produce the “beam screen” of LHC. The beam screen will shield the magnet cold bore from the synchrotron radiation emitted by the circulating proton beam and the power dissipated by the beam image currents. The screen will be cooled to 10-20 K by gaseous He circulating in cooling capillaries attached to its external wall. In the present project, the screen consists of a perforated 1 mm thick, 15 m long welded tube of 44 mm diameter, flattened top and bottom. The internal surface of the screen will be covered by a 50 μm layer of highly conductive colaminated OFE copper. Due to the proximity of the circulating beam, the screen should be totally non-magnetic (maximum acceptable relative magnetic susceptibility $\chi_r = 5\cdot10^{-3}$ at the working temperature) (1,2).

The remaining two grades are retained to produce non-magnetic laminations for the cold masses of the superconducting dipole magnets. This structure, made up of a pile of 3 mm thick laminations, confines and pre-stresses the superconducting coils. Moreover, it allows their geometry to be strictly maintained in spite of the very high electromagnetic forces occurring during magnet testing and operation. During the assembly procedure, the laminations are subjected to a room temperature mechanical stress up to 350 MPa, while during operation at 1.9 K a stress as high as 300 MPa is foreseen. For this reason, suitable mechanical properties are required for the raw material. In order not to impair the magnetic field quality, the selected materials shall also be fully non-magnetic at the working temperature (maximum acceptable $\chi_r = 3\cdot10^{-3}$) and keep non-magnetic behaviour under the cited stress and temperature.

2. MATERIALS

The following materials for cryogenic applications have been tested:

- the new stainless steel X2CrMnNiMoN 19-12-11-1 grade, supplied by Böhler Edelstahl GmbH (Austria) with internal denomination P506 (3),

- the traditional stainless steel X8CrMnNiN 19-11-6 grade, supplied by Nippon Steel Corporation (Japan) with internal denomination YUS130S (4),

- the high Mn steel X8MnCrNi 28-7-1 grade, supplied by Kawasaki Steel Corporation (Japan) with internal denomination KHMN30L (5),

- for a comparison purpose, a standard AISI 316 LN grade, supplied by Creusot-Loire Industrie (France), containing only 1.26% Mn.

Typical heat compositions are shown in Table 1.

Steels	**C**	**Mn**	**Ni**	**Cr**	**Mo**	**Si**	**N**	**P**	**S**	**B**
P506	0.012	**12.05**	10.90	19.18	0.86	0.23	**0.33**	0.005	0.001	<0.001
YUS130S	0.09	**10.70**	6.60	18.00	0.10	0.40	**0.32**	0.022	0.004	-
KHMN30L	0.10	**28.00**	0.82	6.70	-	0.60	**0.10**	0.022	0.004	-
316 LN	*0.021*	***1.26***	*13.03*	*17.33*	*2.61*	*0.61*	***0.16***	*0.025*	*0.0008*	*0.0011*

Table 1: Chemical composition (% in weight) of the three austenitic steels. For comparison, the typical composition of standard AISI 316 LN stainless steel is given.

P506, melted in a Vacuum Induction Degassing (VID) furnace, ingot cast and further processed in a Pressure Electro-Slag Refining (P-ESR) unit, was produced under the form of coiled solution-annealed cold-rolled strips (thickness 2 mm). In order to obtain the final 1 mm-thick beam screen, the cold-rolled strips are colaminated with 50 μm layer of highly conductive OFE copper and partially annealed at 940°C for 5 min. P506 steel has been tested under the two forms of solution annealed strips and partially annealed colaminated strips.

YUS130S was melted in an Argon Oxygen Decarburisation (AOD) furnace, continuous cast and, after a final skin-pass rolling process, produced under the form of coiled solution-annealed cold-rolled strips (thickness 3 mm). The non-magnetic steel laminations will be machined by high precision fine-blanking.

KHMN30L followed the same elaboration plan as YUS130S but was produced under the form of solution-annealed cold-rolled sheets (thickness 3 mm).

316 LN, melted in a VID furnace, was ingot cast and produced under the form of hot-rolled solution annealed plates (thickness 11 mm).

Microstructural observations have shown a fully austenitic microstructure for the three high Mn steels as well as for AISI 316 LN.

3. EXPERIMENTAL TECHNIQUES

The following characterisation techniques were applied:

1) An apparatus for high precision tensile tests at 4.2 K has been developed at CERN and described in a previous paper (6). Flat specimens up to 9 mm^2 square section with a calibrated length of 25 mm have been tensile tested in a liquid He cryostat. Tensile tests on YUS130S stainless steel have been carried out at Air Liquide (France).

2) Room temperature relative magnetic susceptibility (χ_r) measurements have been performed on a magnetic balance under a static magnetic field of 1000 Oe. Cylindrical samples of 2 mm diameter and 3 mm height have been mechanically machined. For P506 stainless steel, samples for susceptibility measurements have been machined in the thinner copper-coated strips. For this material, three samples of 1 mm thickness each have been piled up in order to perform measurements with a sufficient volume of material. As surface oxides are known to perturb susceptibility measurements (7), the surface of all samples was cleaned and deoxidised by a chemical surface treatment (5 min in a 10% HCl aqueous solution).

4. EXPERIMENTAL RESULTS

4.1 Tensile tests

Typical stress-strain curves of the three austenitic steels at 4.2 K are shown in Figure 1. As expected, the so called "serrated yield" (characteristic spikes on the tensile curves) occurs at 4.2 K for some steels. Three tensile curves have been measured for each steel in the rolling and transversal directions (RD and TD, respectively).

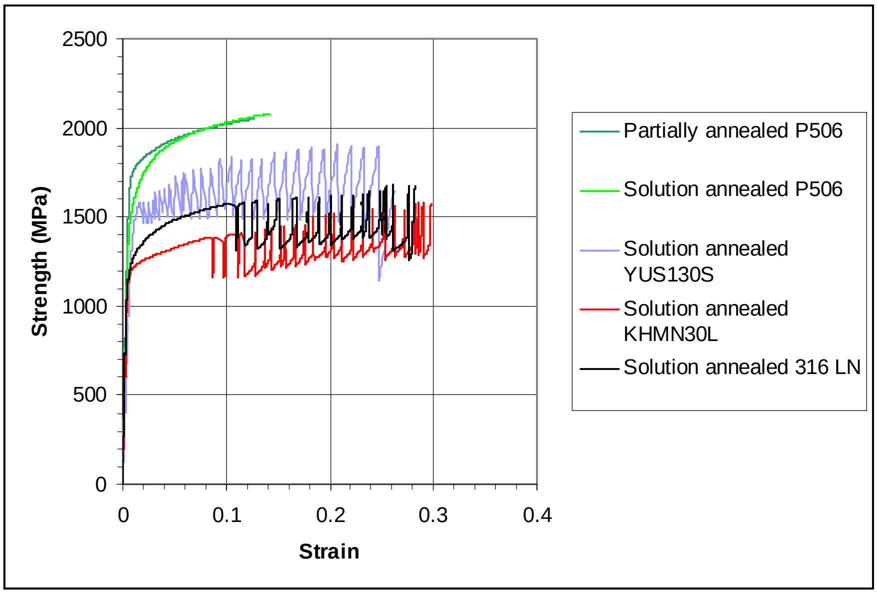

Figure 1: Typical tensile curves at 4.2 K of the three austenitic steels. For comparison, the stress-strain curve of standard 316 LN steel is also shown. Tensile tests have been performed under a strain rate of about 3.5 10^{-4} s^{-1}.

Table 2 shows average values for yield strength at 0.2% offset, Rp0.2, tensile strength, Rm and elongation at breakdown, A.

Due to the homogeneous fully austenitic microstructure and the absence of δ-ferrite oriented bands, only small anisotropy between rolling and transversal tensile properties is observed on the three high Mn steels. All the grades present high strength at 4.2 K, accompanied by an elongation at breakdown larger than 10%. Note the comfortably high strength properties of P506 and YUS130S steels compared to 316 LN.

Steels	Direction	Rp0.2 / MPa	Rm / MPa	A / %
P506	RD TD	1520±35 1633±22	2105±28 2086±43	17.3±1 15.3±3
YUS130S	RD TD	1360±35 1581±9	1881±8 1902±8	25.3±0.4 24.8±2.4
KHMN30L	RD TD	1168±16 1155±14	1557±20 1559±8	42.8±4.3 40.6±1.3
316 LN	*TD*	*1202±20*	*1693±34*	*39.8±5*

Table 2: Tensile properties of the three high Mn austenitic steels. Both RD and TD directions have been tested. For comparison, typical tensile properties of standard 316 LN stainless steel (TD only) are reported. Elongation at breakdown has been measured on broken specimens.

Finally, no change of curvature of the stress-strain curves is detected. On the basis of tensile tests, it is not possible to assess whether the formation of α' martensite occurs associated with a typical change of curvature on the tensile curve (8).

4.2 Magnetic susceptibility measurements

Figure 2 shows the dependence of the room temperature relative susceptibility on plastic strain at 4.2 K for the three austenitic steels. Samples were machined from the deformed region of tensile specimens tested at 4.2 K up to different plastic strain levels and from the closest-to-fracture region of broken tensile specimens.

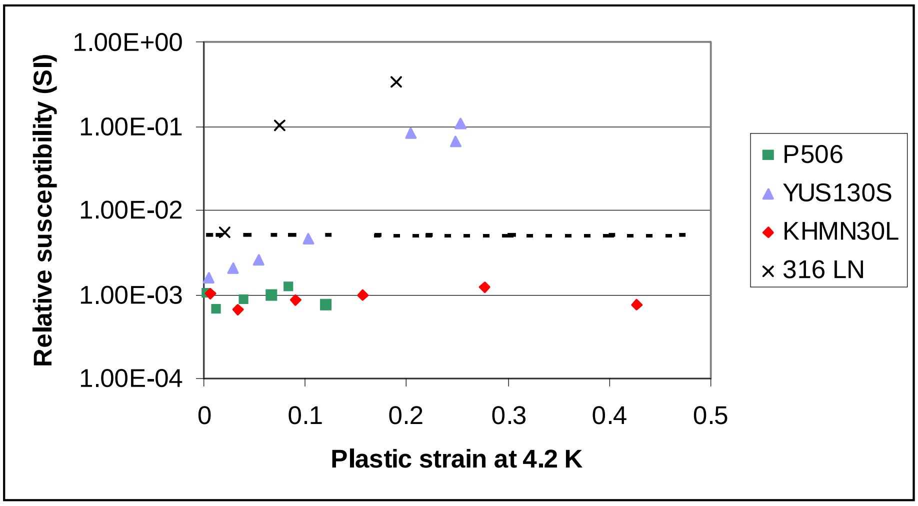

Figure 2: Evolution of room temperature relative magnetic susceptibility with plastic strain at 4.2 K. Values are average values on three tests performed for each plastic strain level. For comparison, values for standard 316 LN stainless steel are also given. The dashed line corresponds to a value of $\chi_r = 5 \cdot 10^{-3}$.

Steels containing the highest amount of alloying elements (in particular Mn), such as P506 and KHMN30L steels, show the lowest and most stable values of relative susceptibility against plastic strain at 4.2 K. The less alloyed YUS130S and 316 LN grades show a continuous increase of susceptibility with increasing strain at 4.2 K, exceeding $5 \cdot 10^{-3}$ as soon as plastic strain reaches about 10% and 2%, respectively.

5. DISCUSSION

High Mn austenitic steels are traditionally selected in non-magnetic structural applications. Their magnetic susceptibility is particularly low at temperatures under their antiferromagnetic transition temperature T_{af} (9). Since Mn is the sole alloying element capable of increasing T_{af}, very high Mn grades (such as KHMN30L steel at 28% Mn having a calculated T_{af} of 288 K) show extremely low magnetic susceptibility ($\chi_r < 2 \cdot 10^{-3}$ (5)) already at room temperature.

For the two other Mn grades considered in the present work (P506 and YUS130S steels having both a calculated T_{af} of approximately 120 K), particularly low values of χ_r are observed at the working temperature of the beam screen (10-20 K) or of the laminations (1.9 K). This makes both grades suitable for their respective applications.

On the other hand, 316 LN (calculated T_{af} = 19.4 K) shows values of magnetic susceptibility at 1.9 K above the tolerated limit ($\chi_r > 7 \cdot 10^{-3}$ vs. $3 \cdot 10^{-3}$).

The second and main advantage of high Mn steels is their stability against α' martensitic transformation.

No increase of magnetic susceptibility has been measured up to breakdown for P506 and KHMN30L tensile specimens tested at 4.2 K. This exceptional stability of P506 and KHMN30L steels to stress-induced martensitic transformation is not only due to their high Mn content (12% and 28%, respectively) but also to the high content of other alloying elements. According to Takemoto (10), non-magnetism should be maintained after deformation at room temperature if the Ni equivalent parameter (Ni_{eq}) of the alloy is higher than 19%, where

$$Ni_{eq} = Ni + 0.6Mn + 9.69(C+N) + 0.18Cr - 0.11Si^2 \qquad (1)$$

Calculated Ni_{eq} for the three high Mn steels is given in Table 3. Compared to standard 316 LN, austenite stability against room temperature deformation is insured for the three P506, YUS130S and KHMN30L high Mn steels. In other words, if high mechanical properties are conferred by cold rolling on these grades, they should keep their non-magnetic behaviour.

Steel	P506	YUS130S	KHMN30L	*316 LN*
Ni_{eq}	24.9	20.2	20.7	*18.6*

Table 3: Calculated Ni_{eq} of the three austenitic steels. For comparison, Ni_{eq} of standard 316 LN is also calculated.

A more traditional parameter used to predict stability of austenite with respect to α’ martensite transformation is Md30. Md30 is the temperature at which 50% of α’ martensite is introduced after 30% true strain.

Table 4 shows the values of the temperature Md30, calculated for the three Mn steels and 316 LN according to the relationship of Nohara et al. (11):

$$Md30\ (^{o}C) = 551 - 462(C+N) - 9.2Si - 8.2Mn - 13.7Cr - 29(Ni+Cu) - 68Nb - 1.42(8-\nu) \qquad (2)$$

where ν is ASTM grain size number.

Steel	P506	YUS130S	KHMN30L	*316 LN*
Md30 (°C)	-291	-171	105	*-167*

Table 4: Calculated Md30 of the three high Mn steels. For comparison, value of 316 LN is calculated.

The calculated Md30 of P506 steel (< 0 K) corresponds to a prevision of full stability of austenite vs. plastic deformation at any cryogenic temperature which is consistent with the present experimental results (see Figure 2).

The calculated temperature Md30 for YUS130S and 316 LN steels is higher than 4.2 K: a fully austenitic structure should not be maintained at 4.2 K during plastic deformation. This is in agreement with experimental results showing that martensitic transformation starts before 10% and 2% of plastic strain on YUS130S and 316 LN grades, respectively at this temperature.

On the other hand, previsions for KHMN30L steel do not match experimental results. The relationship (2) is probably not assessed up to very high Mn contents. The full magnetic stability has been experimentally demonstrated for this grade in the present work. As suggested by Takemoto et al. (12), the austenite stability vs. α’ transformation is due to the difficult formation of plate-like shear bands, a deformation mode which competes with dislocation mode and is typical of higher Mn containing steels.

Experimental results can be summarised as shown in Figure 3.

For structural components which must stay non-magnetic at very low temperatures, 316 LN does not fulfil requirements on magnetic susceptibility. Moreover, its stability against strain-induced α' martensitic transformation at cryogenic temperature is very poor.

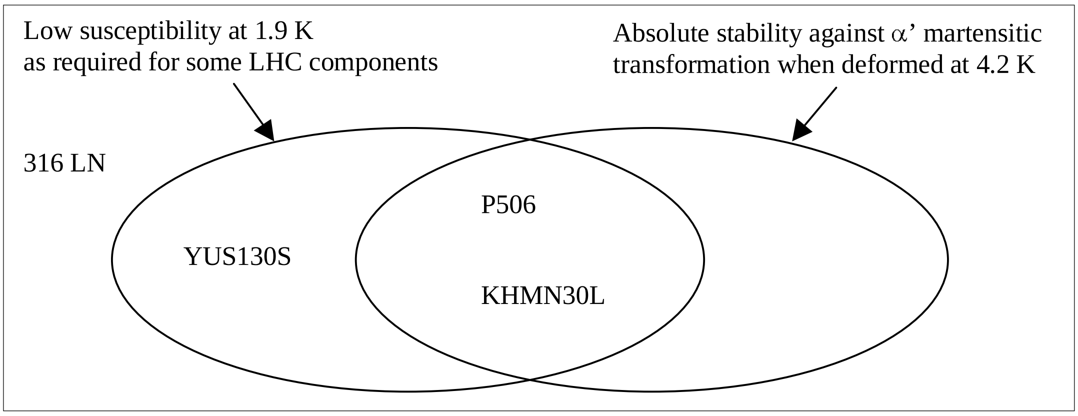

Figure 3: Distribution of the different steels studied in relation to LHC requirements.

The three other grades show a low magnetic susceptibility at the working temperature of 1.9 K. Two of them (P506 and KHMN30L) show full stability of austenite when deformed at 4.2 K up to breakdown, while YUS130S looses stability after some percent of elongation. KHMN30L steel with the highest Mn content at the expense of Ni content represents an economical solution when a high strength fully stable non-magnetic material is required. But its low Cr content makes it subject to corrosion.

The high N and reduced Ni contents of YUS130S make it a good compromise when high mechanical properties and non-magnetism are required at 4.2 K and when strain-induced martensite transformation is not of major concern. The overall increase of alloying elements (particularly Ni) in P506 stainless steel guarantees high strength, non-magnetism and full stability of austenite vs. spontaneous and stress-induced martensitic transformation, at higher cost.

CONCLUSION

At 4.2 K, high Mn austenitic steel grades retain high strength and ductility comparable or superior to the N alloyed grades of the 300-series (such as 316 LN grade).

However, at this temperature, the relatively high magnetic susceptibility of 316 LN and its poor stability to strain-induced martensitic transformation make it unacceptable for some applications.

YUS130S stainless steel is an economical non-magnetic grade but should be applied only when no deformation at 4.2 K is foreseen (loss of magnetic properties).

On the other hand, the highest alloyed grades such as P506 and KHMN30L steels are fully stable against stress-induced α' martensitic transformation. The elaboration of P506 stainless steel through a secondary melting and its high Ni content make this grade the ideal solution for non-magnetic welded structures working at cryogenic temperature. Due to its high price, this grade is selected only for special applications. KHMN30L steel offers a more economical solution at the expense of mechanical strength and resistivity to corrosion.

ACKNOWLEDGEMENTS

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