EUROPEAN ORGANIZATION FOR NUCLEAR RESEARCH
ORGANISATION EUROPÉENNE POUR LA RECHERCHE NUCLÉAIRE

CERN - TS Department

EDMS Nr: 473749
Group reference: TS-MME

TS-Note-2004-036
4 May 2004

AN APPLICATION OF POWDER METALLURGY FOR THE LHC

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Abstract

The cold mass of the 1232 superconducting dipole magnets of LHC, operating at 1.9 K, is enclosed by a shrinking cylinder and two end covers at its extremities. The covers are structural components that must retain high strength and toughness at cryogenic temperature. They are manufactured by Metso Oy /FI in AISI 316 LN steel by Powder Metallurgy (PM) and Hot Isostatic Pressing. PM represents an attractive near-net shaping technique for these components of complex geometry for which dimension tolerances, dimensional stability, weldability are key issues for magnet fabrication, and mechanical properties, ductility and leak tightness have to be guaranteed during operation. The material of the covers and its welds have been fully characterized and mechanically tested down to 4.2 K at CERN. The finely grained structure, the absence of residual stresses, the full isotropy of mechanical properties associated with the low level of Prior Particle Boundaries oxides result in superior mechanical properties and high ductility down to liquid helium temperature.

Presented at the TS Workshop
Archamps, France, May 4 – May 6, 2004

1 INTRODUCTION

The 1232 superconducting dipole magnets of LHC will operate at 1.9 K. The cold mass of the dipole magnets is enclosed by a shrinking cylinder and two end covers at its extremities. The end covers are domed and equipped with a number of protruding nozzles for the passage of the different cryogenic lines (Figure 1). The covers are structural components that must retain high strength and toughness at cryogenic temperature. They are MIG welded onto the magnet shrinking cylinders. The protruding nozzles of the covers will be welded to the interconnection pipes by an automatic orbital autogeneous TIG technique. Several thousands of welds are necessary.

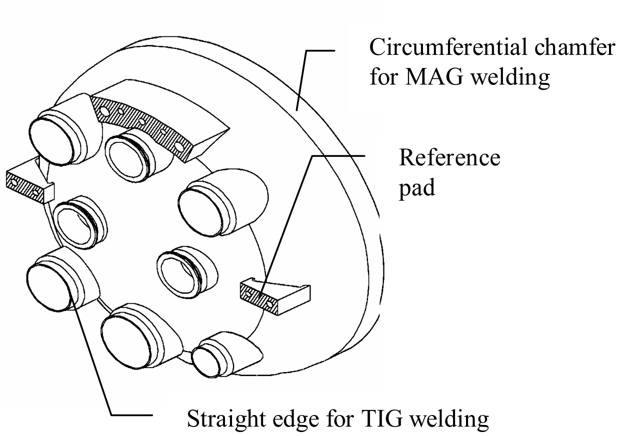

Fig. 1: 3-D view of an end cover. The diameter of the cover is 570 mm, the height 224 mm and the overall weight 100 kg. The extremities of the nozzle to be welded on the interconnection pipes have an external (internal) diameter of 84 (80) mm. The main circumference to be welded to the shrinking cylinder has a thickness of 10 mm. The most severe dimensional tolerances on the nozzles are of the order of 0.05 mm.

The austenitic Stainless Steel (SS) grade AISI 316LN has been retained because of its mechanical properties, ductility, stability of the austenitic phase against low-temperature spontaneous martensitic transformation. 316LN is readily weldable; it has been extensively shown [1] that properly qualified welds of this grade retain high ductility and toughness down to 4.2 K. Due to the complex geometry of the end covers, PM is the near-net-shaping technique retained for their fabrication. Closed or open die forging would have required significantly more machining, a welded product would have needed extensive inspections and stress relieving, while a cast solution would have resulted in poorer mechanical properties.

Dellis et al. [2] have already shown the high impact strength and ductility of low oxygen HIPed 316LN at Room Temperature (RT). Couturier et al. [3] have shown superior mechanical properties of PM 316LN compared to the same wrought grade. The improved properties of PM austenitic SS are explained by Appa-Rao and Kumar [4] on the basis of the finer grain size, low oxygen content and chemical homogeneity. Increasing oxygen content has a reported negative effect on RT impact toughness of a 316L grade, less on ductility [5,6].

To be retained for the end covers, the PM solution must guarantee a fully austenitic microstructure (showing high ductility down to 1.9 K), ready weldability and absence of susceptibility to hot cracking in fully austenitic welds. The stability against spontaneous martensitic transformation is required in order to maintain the dimensional stability of the covers, that will be repeatedly cycled during the life of the machine between 293 K and 1.9 K. The base metal shall show high mechanical properties (the maximum calculated stress on the covers is 121 MPa), and high ductility at low temperature (the pressure vessels codes typically require an impact energy larger than 40 J at the working temperature). For reasons of compatibility, the thermal contraction coefficient shall be comparable to the one of a wrought stainless steel of the same grade.

2 EXPERIMENTAL

Fully dense PM Hot Isostatic Pressed (HIP) covers of 316LN grade have been produced by Metso Oy according to the drawing of Figure 1. The chemical composition of the supplied covers is reported in Table 1. The covers have been produced from powders atomized, blended, homogenized and filled into capsules with geometry approaching the cover shape. After evacuation and sealing, a 1180 °C-3 h HIPing cycle has been performed under a pressure of 100 MPa. The capsule tightness has been argon tested after HIPing. The capsulated covers have been solution annealed at 1070 °C for 4.5 h and water quenched, pickled to remove the capsules, ground and machined to the final dimensions. A final pickling followed the dimensional inspection. 100% dye penetrant, visual inspection and US inspection (to measure the wall thickness and detect possible defects) have finally been performed on the finished covers, showing no relevant defects and full soundness and compactness of the components.

Table 1. Compositions of the PM 316LN end covers and of wrought 316LN plates from two different producers

	Producer	C	Cr	Ni	Mn	Mo	N	Si	P	S	O
PM Cover	**Metso Oy**	**0.017**	**16.98**	**13.07**	**0.71**	**2.53**	**0.16**	**0.59**	**0.012**	**0.005**	**0.011**
Wrought 316LN	Avesta	0.017	17.2	12.5	1.2	2.58	0.15	0.4	0.035	<0.001	-
Wrought 316LN	Creusot-Loire	0.020	17.33	13.03	1.26	2.61	0.17	0.61	0.025	0.0005	-

The fabricated covers have undergone thermal cycling (5 quenches in liquid nitrogen and re-heating to RT) followed by a RT pressure and leak test. Samples were removed from the covers to perform the following tests which have been carried out at CERN, unless otherwise stated:

1) Microstructural investigations, performed on an optical microscope DMRME of Leica coupled with an image analyzer Quantimet 600S. Scanning Electron Microscopy (SEM) observations on a LEO 430i microscope have also been performed on impact test broken specimens.

2) Magnetic permeability measurements, performed through a Fischer magnetoscope type 1.068 (0.1 T probe).

3) Tensile tests at 4.2 K, performed in a special apparatus developed at CERN and described in a previous paper [7]. Specimens of 6 mm^2 square section with a calibrated length of 25 mm have been tensile tested in a liquid He cryostat. Room temperature tensile tests have been carried out on normalized specimens.

4) Charpy impact testing (according to the standard EN-10045), performed at 4.2 K and RT by Linde AG on normalized V-notch samples. Samples have been encapsulated in order to guarantee stable temperature during transfer from the liquid He cryostat to the impact testing machine. Encapsulation is performed according to a Linde design, based on 50 years experience.

5) Full penetration TIG and MAG welding tests, performed following welding procedures specified and approved according to EN 288-3. Orbital pulsed TIG welds have been carried out at a travel speed of 0.015 m/s, a peak (base) current of 68 (32) A under 100 % Ar shielding. Circumferential MAG weldments between plates produced by Avesta (Table 1) - formed to the diameter of the shrinking cylinders and longitudinally welded - and the covers have been performed using a WN 1.4455 filler under an Ar-CO2-N2 shielding. The reference thickness for the MAG welds is 10 mm.

6) Thermal contraction measurements between RT and 4.2 K, performed by CEA Grenoble in a specially designed two-stage cryostat.

3 EXPERIMENTAL RESULTS

After the final solution annealing, the structure of the steel is fully austenitic and free of nitrides or carbonitrides (as observable by optical microscopy up to a magnification of 500 x). The steel shows a low inclusion content (according to ASTM E45 method A, no A,B,C type inclusions; average D-type inclusion up to 2.2). The inclusion content has also been evaluated by image analysis according to the standard ASTM E1245, with the results shown in Table 2. This procedure is more adapted to PM products, containing only spherical inclusions. The typical ASTM E112 grain size number is 6 to 7.

Table 2. Inclusion rating according to ASTM E1245 on the PM 316LN end covers. The inclusion diameter is an additional parameter not mentioned in the standard, defined as the feature equivalent diameter. Reference is made to the standard for the explanation of the items.

	Units	**Mean value**	**Max value in a single field**
Area fraction of inclusions	%	**0.07**	**0.55**
Number per unit area of inclusions	1/mm^2	**206**	**564**
Average area of the inclusions	μm^2	**4.47**	**510**
Mean free path of the inclusions	μm	**72**	**Min. 18.5**
Number of interceptions of inclusions per unit length of scan line	1/mm	**18**	**54**
Diameter of the inclusions	μm	**2.01**	**38**

The relative magnetic permeability at RT is between 1.0027 and 1.0036. Such a low permeability value indirectly confirms the absence of δ-ferrite or α'-martensite.

Thermal contraction measurements between 332 K and 4.2 K have been performed on three different samples removed from the covers and arbitrarily oriented. The results are shown in Figure 2 and compared with a wrought 316LN plate. The thermal contraction of the PM 316LN is independent on the removal position and direction and equal to the one of the corresponding wrought grade.

Results of tensile tests at 4.2 K and RT are reported in Figure 3a. As expected [8], the so-called "serrated yield" (characteristic spikes on the tensile test curve) was observed at 4.2 K. A change of curvature of stress-strain curves was observed at 4.2 K at a strain 10 % < ε < 15 %. Compared to RT measurements, we notice a general increase with decreasing temperature of the yield stress Rp0.2 (from 334 MPa to 1118 MPa) and of the ultimate tensile stress Rm (from 664 MPa to 1768 MPa), as typical for stainless steels at low temperatures.

Note the high values of strength at 4.2 K, accompanied by a total elongation A of 45 % close to the RT value (49 %).

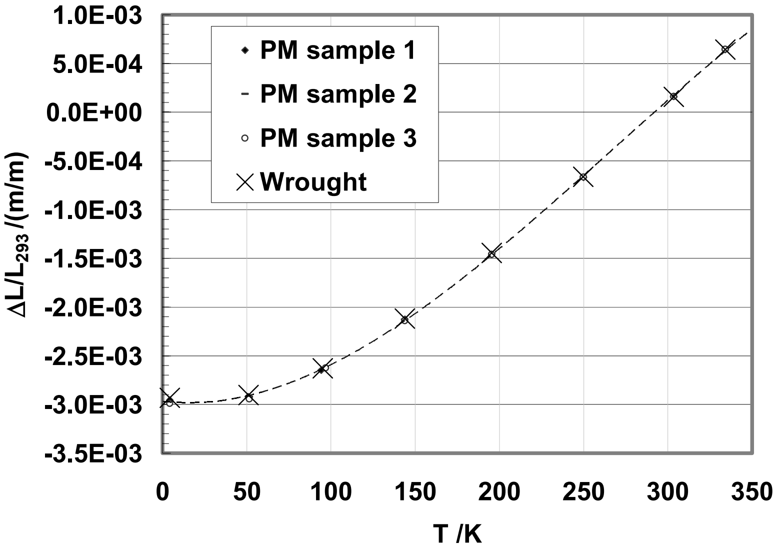

Fig. 2: Thermal contraction of samples removed from the PM cover and a wrought 316LN hot rolled plate produced by Creusot-Loire Industrie (Table 1). All curves are indistinguishable.

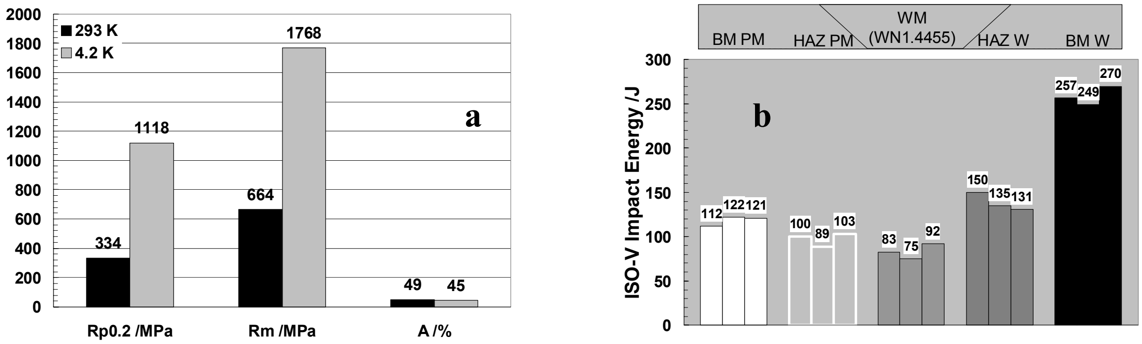

Fig. 3: a) tensile properties of PM 316LN at RT and 4.2K; b) values of impact energy evaluated at 4.2 K across a MAG weld. From left to right, base metal, PM-side (BM PM); HAZ, PM side (HAZ PM); weld metal (WM); HAZ, wrought side (HAZ W); base metal, wrought side (BM W).

Tensile tests across the MAG weldment performed at RT (4.2 K) show a joint yield strength of 404 (1278) MPa and a joint tensile strength of 681 (1649) MPa. Except for the tensile strength at 4.2 K, the tensile properties of the joint are higher compared to PM base metal properties.

As shown in Figure 3b, the average impact strength value evaluated at 4.2 K is 118 J (compared with an impact strength of 259 J of the wrought base metal side). In the Heat Affected Zones (HAZ) of the MAG welds near the PM side the average strength is 97 J (compared with 139 J in the HAZ of the wrought side). All these values are well above the minimum required 40 J of the pressure vessel codes.

4 DISCUSSION

The experimental results on HIPed 316LN covers produced by Metso Oy on CERN specification confirm that PM 316LN shows superior and isotropic yield and tensile strength compared to wrought 316LN. This feature is generally attributed to the finer microstructure of the PM product. At 4.2 K, this grade presents a Rp0.2 (1118 MPa) slightly lower than a wrought 316LN-plate of similar thickness. For comparison, hot rolled plates of 316LN produced by Creusot-Loire Industrie (Table 1) for the shrinking cylinder have Rp0.2 = 1198 MPa at 4.2 K. On the other hand, the 4.2 K tensile strength Rm is higher for PM than for rolled 316LN (1768 MPa vs. 1674 MPa). Since the observed increase in work hardening on the 4.2 K stress-strain curves is due to the formation of α' strain-induced martensite, and transformation points are very sensitive to the alloy composition, the difference in ultimate strength might partly depend on the different amount of transformation undergone by the PM vs. the wrought grade. Finally, a higher ultimate strain was measured at 4.2 K for the PM steel (45 % vs. 38 %).

Impact energies at 4.2 K are substantially lower than for wrought products (Figure 3b), due to higher oxygen content. SEM observations of impact fractures show the typical presence of μm-size oxide inclusions within dimples. Nevertheless, the values of 118 J (i.e. 153 J/cm^2) measured by us at 4.2 K are much higher than the values reported by Couturier et al. [3] for a PM 316LN who measured at 77 K an impact energy lower than 100 J/cm^2. This high impact toughness, compared to other PM products of the same grade, can be interpreted in terms of the low oxide inclusion content. According to Zou and Grinder [6], the RT impact toughness of a 316L grade containing 1360 ppm of oxygen is only 50% of that with 300 ppm oxygen. The 316LN of the present study is produced with an oxygen content of only 110 ppm. The absence of nitrides and a very homogeneous microstructure also contribute to confer ductility and toughness to the grade, which are maintained down to 4.2 K.

The specified composition limits and the reduced impurity (P, S) content result in ready weldabilility (as observed in autogeneous TIG or MAG welding with filler). The austenitic microstructure of the weld beads is not accompanied by the presence of traces of liquation or hot cracking. The weld impact energies are largely above the minimum required, both in weld metal and HAZs.

The coefficient of thermal contraction between RT and 4.2 K depends on the antiferromagnetic transition temperature varying with the chemical composition of the steel [9,10]. In view of the very similar compositions, the almost identical contraction behavior of PM and wrought 316LN could be predicted.

5 CONCLUSIONS

The relevant mechanical and physical properties, as well as the weldability of a fully-dense, low-oxygen PM 316LN SS grade have been assessed down to 4.2 K on samples issued from real scale, complex shape HIPed products, and on their welds. This study shows that PM is a technique fully adapted to the fabrication of complex shape components such as LHC end covers, working in a severe cryogenic environment. This is demonstrated by the excellent behavior of dipole magnets equipped with PM covers, which have performed satisfactorily for several years in the String 2 experiment. This near-net shaping technique, finally retained for the series production, was selected as well on the basis of its price competitiveness.

6 ACKNOWLEDGMENTS

CEA Grenoble assessed low temperature contraction properties, M. Caccioppoli and P. Mésenge performed welding tests, H. Mitterbacher (Linde) performed impact tests, M. Fassone wrote the image analysis program and performed the evaluations. T. Polimeno, N. Schmidt and Y. Giraudier contributed extensively to the experimental work. F. Savary, J. Liimatainen and M. Kumpula (Metso Oy) coauthored a previous paper on the subject [11].

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