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Influence of the surface treatment of the CMS conductor on its adhesion properties at 300 K and 4.2 K

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ABSTRACT

In the 4 T, 12.5 m long, 6 m bore diameter superconducting solenoid for the CMS (Compact Muon Solenoid) experiment at LHC (Large Hardron Collider), the interfaces between the insulation and the conductor or the external cylinder are subjected to high shear forces during the cool down to 4.5 K and the operation phase up to 4 T. Due to the conductive cooling of the coil, the bonding at the different interfaces is a critical point, which is directly related to the quality of the surfaces. The influence of the surface treatment of the conductor on its adhesion properties has been studied at room temperature and in liquid helium at 4.2 K, using a shear force measurement procedure developed in our lab or a peeling test with tape. This paper presents the experimental results obtained on samples treated with solvent cleaning, sandblasting or anodic oxidation under 4 different sets of experimental conditions, then wrapped with glass tape and impregnated under vacuum. The robustness of the anodic oxidation process, as well as the effect of ageing a few months before impregnation is shown.

Keywords : surface treatment, aluminium, shear stress, insulation

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I- INTRODUCTION

A 4 T, 12.5 m long, 6 m bore diameter superconducting solenoid is being manufactured for the CMS experiment at LHC. The coil consists of five modules mechanically and electrically connected. Each module consists of four layers of 109 turns wound inside a mandrel, which means 45.1 km of conductor, Ref. [1].

The insulation consists of a glass epoxy composite and can be found at different places in the coil: as turn to turn insulation around the conductor, as ground insulation at the inner diameter, between layers, between the last layer and the external cylinder, between two modules. The glass fibre tape or fabric is integrated to the module during the winding process and then each module is impregnated under vacuum, according to the state of the art.

During the numerous stages of the coil life, the insulation has to meet various requirements:

- ensure the electrical insulation at each point of the coil,
- withstand the mechanical stresses. FEA stress analysis has shown that the insulation may have to support locally shear stresses up to a maximum value of 24 MPa. The rupture shear stress has been specified at 72 MPa at 4.2 K including a safety margin of 3, Ref. [2],
- ensure a thermal continuity in the coil because an indirect cooling technique has been chosen. Any loss in this continuity would decrease or suppress locally the ability of cooling the coil and could induce a quench.

This last requirement in particular requires a good quality bonding between the different elements (conductor/insulation, layer/cylinder). This bonding is related to the wetting properties of the resin and the preparation of the metallic surface. The aim is to obtain a bond at the interface that is stronger than the resin itself.

II- SURFACE TREATMENT

Several kinds of treatment have been considered to prepare the conductor surface before the insulation: degreasing with a solvent, mechanical treatment to increase the surface roughness and favour the bonding of the resin, making a bonding layer by chemical conversion with a surface chromating or by anodization.

The chemical conversion has been eliminated because it doesn't allow easy management of the frequent stops in the winding process.

The anodic treatment consists of an electrochemical oxidation in a solution containing phosphoric acid and surfactants. On the metal surface, a fine, porous oxide layer is built up that is stable and not sensitive to hydration. This is due to the incorporated phosphate ions. Switching off the external electrical supply is sufficient to stop the process.

Six treatments have been tested:

- T1 = sand blasting with glass balls, diam. 45-90 µm, and ultrasonic cleaning in alcoholic bath,
- T2 = cleaning with acetone and drying for 1h in an oven (110°C),
- T3 to T6 = anodization by Péchiney, with 4 different sets of operating conditions.

III- EXPERIMENTAL PROCEDURE

The samples have been prepared in a similar way to the coil: 2 bars of Al alloy 6082 have been treated and wrapped with glass tape (ref. 204NC1383, from Bourgeois, 0.13 mm * 25 mm), 50% overlapped. They have been vacuum impregnated with epoxy resin (100 pp araldite F + 100 pp HY905 + 10 pp DY040, by Ciba) in a mould which allows preparing 12 samples at once and curing at 12 h/80°C + 24 h/125°C under pressure. Two grooves have been machined in the sample to release the joint on a length of 5 mm (Fig. 1). This optimized lap-joint specimen geometry, Ref. [3], puts the joint mainly in shear eliminating edge effects. A special device has been used to avoid any damage during machining.

The shear tests have been carried out with a screw-driven machine at room temperature and in liquid helium at 4.2 K. The crosshead displacement speed has been set at 0.2 mm/min. The load has been measured with a 150 kN cell. All samples have been loaded to rupture. For each surface treatment type, at least three samples have been tested. The shear stress values have been calculated by dividing the rupture load by the joint specimen area ($\approx 5*20\ mm^2$).

Tape tests have also been performed by applying a piece of tape on the surface to be studied: the force needed to pull off the tape is evaluated and the quantity of glue remaining on the surface is observed. With good bonding, the glue is partly transferred to the surface and a strong force is needed to pull up the tape. With poor bonding, the tape is easily pulled off and there is no glue left on the surface.

IV- RESULTS

Table 1 and Fig. 2 summarize the rupture shear stress obtained with the different surface treatments. First the samples were impregnated just after the surface treatment. Figures 2 a) and 2 b) show the results at room temperature and 4.2 K.

The solvent cleaning gives the lower values with a very large scatter (42%). The shiny surface which is observed after breaking shows that the fracture occurs at the interface between the metal and the resin.

In the case of anodic oxidation, the results show a very good reproducibility and it is not possible to see any real difference between the 4 sets of conditions. The fracture occurs inside the insulation. At 4.2 K, the mean value is at least 101 MPa, with results above 110 MPa.

The results with sand blasting are very sensitive to the initial conditions of the surface. Samples T1/A realized with bars of the set A (machined with pure water) have given results almost similar to those of anodic oxidation but with a larger scattering. Samples T1/B obtained from bars of the set B (produced by another company, with few % of additives in water) have broken during manufacturing in the majority of cases or given very low results. Samples T1/C have been produced from bars of the set C machined with pure water, similar to set A. As the control of the surface with the test of the drop of water has given bad results, half of the bars have been cleaned with detergent DT423 from NGA laboratory. Results of the two groups, T1/C1 not cleaned and T1/C2 cleaned, are similar with a large scatter and half of the samples don't fulfill the technical specification at 4.2 K.

Figures 2 c) and 2 d) show the results after some months of ageing between the treatment and wrapping of the samples and their impregnation. The bars treated by anodic oxidation could be aged 6 months without any decrease in results. After 2.5 months of ageing, 5 of 6 samples T1/C1 have broken in the time between machining and the test, the last one giving a very low result. The results with the series T1/C2 are correct at 300 K, but only partly acceptable, with large scatter, at 4.2 K.

On a piece of conductor treated by anodic oxidation T4, we have not observed any difference between the pure aluminium (central part) and the 6082 alloy (side parts) surfaces, in term of oxide thickness (measured using optical methode) or bonding (tape pull up, Tab. 2). The Figure 4 shows the conductor after removal of the tape. The white zones correspond to the surfaces covered by the tape: the glue is partly transferred on there, even after several months ageing.

V- CONCLUSION

The surface treatment by anodic oxidation appears to be the most interesting solution to prepare the conductor. Rupture shear stress values above 100 MPa have been obtained at 4.2 K, with little scatter between samples. No decrease of the results has been observed after 6 months ageing. No behaviour difference has been noticed between the two components of the conductor exposed to the process and no real differences between the 4 sets of operating conditions. That gives a good flexibility for the adjustment of these conditions and shows robustness of the process. Supplementary tests are needed to verify the compatibility of this process with the frequent stops of the winding line.

As good results can also be obtained with sand blasting if the surface preparation is fully qualified and some scatter accepted, sand blasting has been retained for the actual winding.

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Table 1: Rupture shear stress for the different surface treatment with and without ageing. (*) several series broken before test, (**) samples broken before test.

Rupture shear stress (MPa)	T1/A	T1/B	T1/C1	T1/C2	T2	T3	T4	T5	T6
No ageing									
σ_{mean} (300K)	51	18 (*)	47	35	33	55	55	55	50
Standard deviation	4%	56%	16%	21%	42%	4%	1%	3%	14%
σ_{mean} (4K)	99		68	82		101	110	101	105
Standard deviation	8%		30%	23%		5%	1%	6%	4%
Ageing (in months)			*2.5 m*	*2.5 m*		*6 m*	*6 m*	*6 m*	*6 m*
σ_{mean} (300K)			(**)	45		51	48	52	55
Standard deviation				14%		7%	17%	3%	2%
σ_{mean} (4K)			(**)	63		108	107	107	91
Standard deviation				20%		7%	3%	10%	15%

Table 2: Test with tape pull up on different surface treatments.

Treatment	Force	Marks on the surface
Sand blasting (T1) on 6082	weak	none
Cleaning (T2) on 6082	weak	none
Oxidation (T4) on 6082	strong	Glue transferred on all the surface
Oxidation (T4) on pure Al	strong	Glue transferred on all the surface

Fig.1: Shear stress sample geometry.

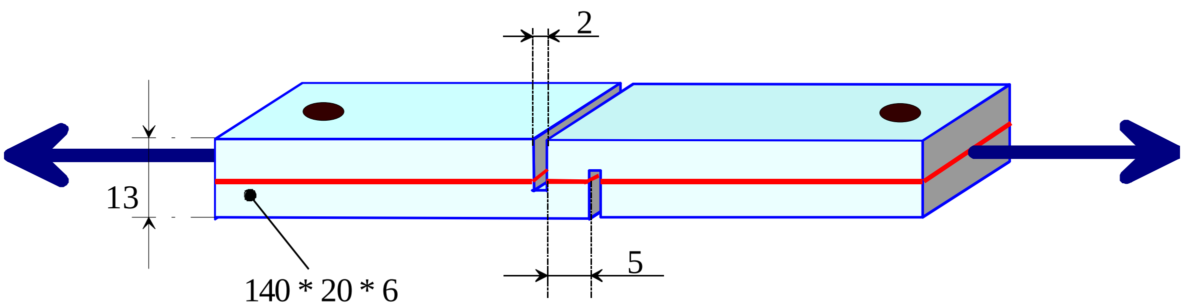
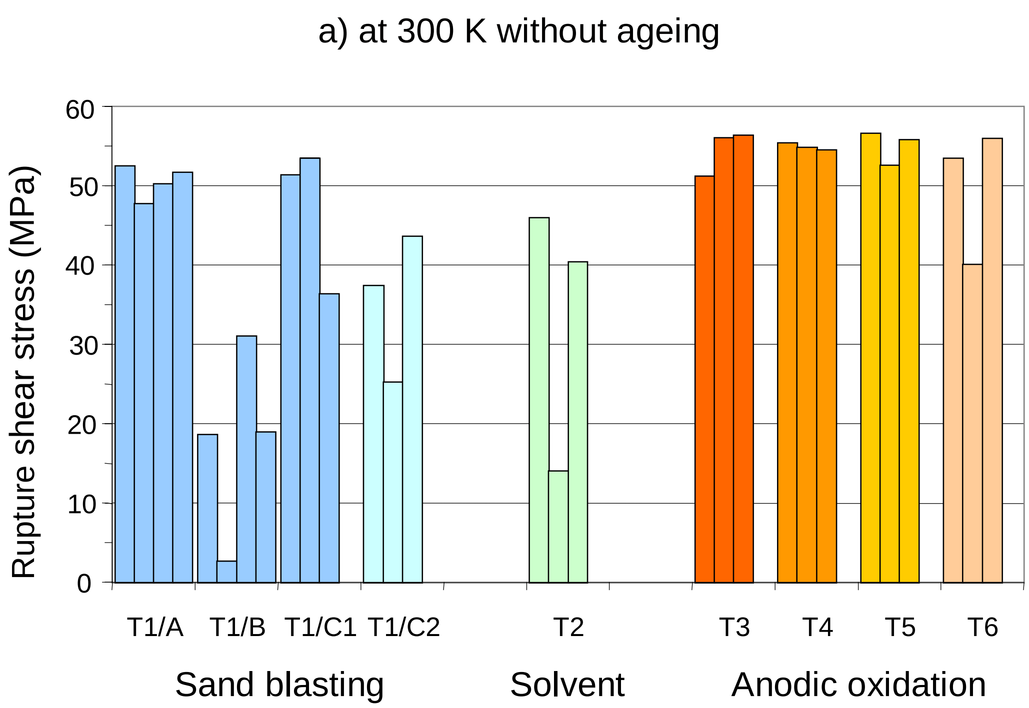
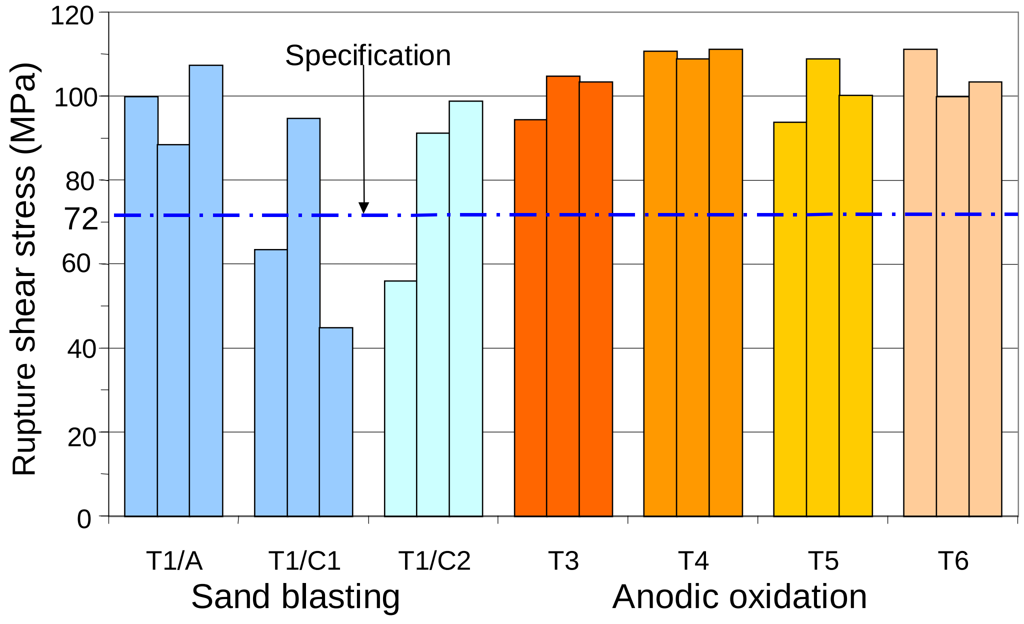


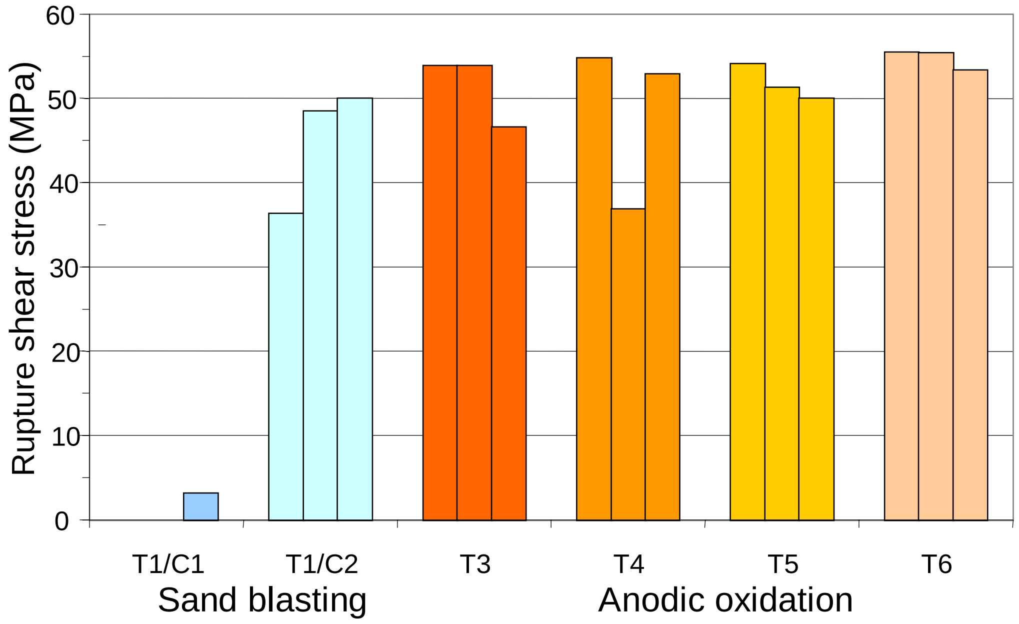
Fig.2: Rupture shear stress for the different surface treatments:



b) at 4.2 K without ageing



c) at 300 K with ageing



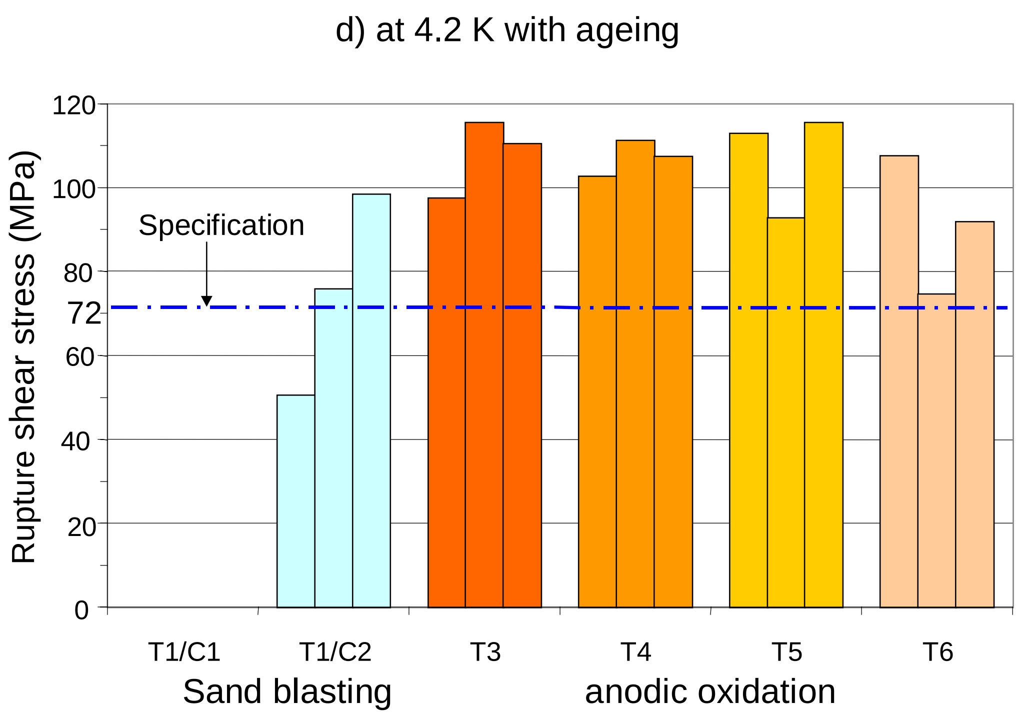


Fig.3: Conductor treated by anodic oxidation after tape pull test.