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SHEAR TESTS ON GLUES FOR MAGNET COLLARS FOR THE LHC

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ABSTRACT

In the future Large Hadron Collider (LHC) under construction at CERN, organic materials used for the insulation of the superconducting magnets will be exposed to high radiation levels at temperatures as low as 1.8 K.

A representative selection of organic materials comprising insulating films, cable insulations, epoxy resins and composites were already irradiated and tested at the temperature of liquid nitrogen. The comparison of the results with the ones obtained at room temperature showed that there is no significant influence of the irradiation temperature on the radiation degradation of thermosets and composites. It was concluded that a good indication for the radiation behaviour of this type of materials to be used in a cryogenic environment could be assessed from room temperature tests after having measured the initial properties at low temperature.

This paper presents the results of mechanical tests on four epoxy gluing systems, candidates to be used for the front collars of the LHC magnets. Shear specimens of aluminum alloy were properly prepared and glued. Some were submitted to ageing at 1.8 K, with and without mechanical stress, other were submitted to thermal cycling (down to 77 K), and others were irradiated in a switched-off nuclear reactor up to 3 MGy. Shear tests have been carried out at room temperature and at cryogenic temperature. It shows that one gluing system, commercially available is suitable for this LHC application.

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INTRODUCTION

For the selection of organic materials to be used in the construction of the Large Hadron Collider (LHC) magnets, the cryogenic temperatures and the radiation levels have to be taken into account. In particular, the front collars of the dipole magnets will consist of piles of collar plates and spacers which will have to be glued (Fig. 1). The operation temperature of the coils will be 1.8 K, and the expected dose level will be of the order of 10 kGy per year for most of the coils, but at some places it could be at least 10 times higher [1].

Radiation resistance tests at room temperature are carried out at CERN on a routine basis, and many results are published in the form of catalogues [2]. For the qualification of organic materials for the LHC, irradiations and tests have to be done at cryogenic temperature. The modification of mechanical properties of some representative materials comprising insulating films, cable insulations, epoxy resins and composites have been measured after irradiation in liquid nitrogen [1]. The conclusion of the study was that a good indication for the radiation behaviour of this type of materials could be assessed from room temperature tests after having measured the initial properties at low temperature.

For the qualification of gluing systems for the front collars of the LHC magnets, shear samples were submitted to various stresses such as thermal cycling, ageing at 1.8 K under mechanical stress, and irradiation up to 3 MGy, they were then tested at room temperature and at cryogenic temperature. The present paper gives the results of these tests.

SELECTED MATERIALS AND SHEAR SAMPLES

The technical specification for the collars asks for a perfect alignment and a perfect parallelism of the glued plates, the alignment holes must stay free, creeping of the polymer under high pressure must be avoided during the magnet making, and last but not least, the adopted technology must be applicable to industrial process. For this, the selected adhesive must preferably be a liquid, easily applicable with a roll, or even be sprayed, in one thin layer, on both sides of the spacers. The coated spacers will be kept stored before being assembled with the plates.

The first prototypes of collars were made of stainless steel plates, it was further decided to use the AL 5083 aluminium alloy. For the purpose of the shear tests, metallic plates were cut in rectangular shape and glued as indicated on Fig. 2. The dimensions were 150 mm x 10 mm for the thermal tests and 100 mm x 10 mm for the radiation tests. As the thickness of the real spacers will only be 0.2 mm, their sanding will not be possible, and they will have to be cleaned by chemical means.

Fig. 1 : Layout of front collar for the LHC dipoles

Four epoxy-based adhesives were selected for this study: a former system, well known at CERN (AZ 15 / HZ 15), the replacement system (XD 4447/4448), and two systems which cure at room temperature (RT). Table 1 shows the selected systems, together with the pressure, the temperature and the time of polymerisation.

Preliminary tests were carried out to compare the adherence of the systems on the stainless steel and on the aluminium alloy. They allow to see the effect of a proper chemical cleaning, and the difference if one or two of the surfaces are coated. The results have shown that for the XD 4447/4448 system, the shear strength is lower with the aluminium than with the steel, while it is the inverse for the AW 106 system. The chemical cleaning by means of H_2SO_4 and $\text{Na}_2\text{Cr}_2\text{O}_7$ was selected for the aluminium alloy.

These preliminary tests have also shown that the two systems which cured at elevated temperature, thanks to their solvent content, can be sprayed, which makes them more easily applicable to an industrial process. On the contrary, the two systems which cure at room temperature are not adapted to an industrial process for this application: they are too viscous to be easily laid with a roll, the coated spacers are sticky and can not be piled for intermediate storage, and the glues creep into the alignment holes during curing under pressure. Nevertheless, the study continued on these systems.

The proper polymerisation of the XD 4447 / 4448 system was checked by differential scanner calorimetry (DSC); no exothermic peak has been detected below 200°C (which corresponds to the decomposition). It shows a T_g around 100°C.

Table 1 : selected adhesives

Adhesive	Pressure	Temp/Time	Ref.No	Supplier
Araldite XD 4447 / 4448	0.20 MPa	160° / 2 h	M 722	Ciba-Geigy
Araldite AZ 15 / HZ 15	0.20 MPa	170° / 5 h	M 723	Ciba-Geigy
Araldite AW 106 / HV 953	0.15 MPa	RT / 24 h	M 724	Ciba-Geigy
Stycast 2850 FT	0.15 MPa	RT / 24 h	M 725	Emerson & Cuming

The solvents in systems XD 4447 / 4448 and AZ 15 / HV 15 are respectively based on ethanol and acetone.

Fig. 2 : Shear samples

For each adhesive, five sets of shear samples were prepared for each type of thermal test and for each radiation level. The results given below are the mean values for the five samples.

More details about all the preliminary tests, the samples preparation and the chemical cleaning can be found in [3].

THERMAL TESTS

Thermal cycling

The thermal cycling consisted of dipping the samples into a bath of liquid nitrogen (77 K) for 5 minutes, and to bring them back to room temperature (293 K) for 5 minutes. The number of cycles is indicated in Table 2.

A more severe thermal cycling consists of about two weeks at 1.8 K, 3 days at 4.2 K, and about one week at room temperature (the exact lengths of the periods depend on the availability of the cryostat).

Some of the samples undergo this severe cycling under a stress of 15 MPa. No visual degradation was noticeable after two of these cycles.

Shear test results

The shear tests were carried out with a Zwick machine at a speed of 3 mm/min. The values of the shear strength at room temperature and at 77 K, prior and after thermal cycling down to 77 K appear in Table 2. The mean values are given within the accuracy of 4% to 8% (one sigma).

Table 2 : Temperature effect on the shear strength (in MPa)

	M 722 XD 4447	M 723 AZ 15	M 724 AW 106	M 725 2850 FT
Initial value at 293 K	25.8	26.4	21.1	16.9
Initial value at 77 K	40.3	17.6	18.3	23.0
after n cycles,	n = 40	n = 10	n = 10	
at 293 K	22.2	23.1	20.4	n.m.
at 77 K	39.6	19.5	21.3	n.m.

For the XD 4447/4448 system, after 5 cycles down to 1.8 K, no significant change in the shear strength could be noticed on the "free" samples, and a reduction by 25% is noticed on two samples that were under stress. The other systems are still under cycling.

RADIATION TESTS

Irradiation conditions and dosimetry

The samples were irradiated in a cobalt 60 source up to dose levels of 0.5 and 1 MGy, and in a nuclear reactor to a dose level of 3 MGy.

The cobalt source is an industrial panoramic source located in Dagneux (France). The samples are irradiated in the center of a 20 cm diameter barrel made of cobalt rods. The dose rate is about 4.5 kGy/h, the irradiation medium is air at room temperature. The dose rate is measured by means of red perspex dosimeters calibrated versus alanine.

In the switched-off ASTRA 7 MW pool reactor in Seibersdorf (Austria), the mean dose rate is of the order of 45 kGy/h, and the contribution of neutrons to the total dose is negligible. The irradiation medium is also air at room temperature. The doses are measured by means of an ionizing chamber for CH₂-equivalent materials.

More details about irradiation facilities and dosimetry can be found in [4].

Shear test results

The shear tests were carried out with an Instron 1026 machine at a speed of 2 mm/min. The variation of the shear strength with respect to the absorbed dose appears in Table 3 and on Figure 3 [5]. The standard deviation for the five samples is between 5% and 15%.

Table 3 : Radiation effect on the shear strength at 293 K (in MPa)

	M 722 XD 4447	M 723 AZ 15	M 724 AW 106	M 725 2850 FT
Initial value	24.9	20.5	28.3	20.8
after a dose of 0.5 MGy	22.6	16.9	27.4	20.2
after a dose of 1.0 MGy	22.4	12.8	29.4	22.5
after a dose of 3.2 MGy	8.7	5.0	28.5	21.6

Within experimental errors, Araldite AW 106 and Stycast 2850 (cured at RT) do not show any radiation degradation up to a dose of 3 MGy. It is against expectation that the systems which cure at higher temperature are more radiation sensitive. The explanation for this may come from their solvent content.

CONCLUSION

After the preliminary tests, the choice of the system was reduced to one, e.g. the new XD 4447 / 4448. The system AZ 15 / HV 15 is not available anymore, and AW 106 and Stycast 2850 FT are not applicable to industrial process for this purpose.

This study has confirmed that the new XD 4447 / 4448 gluing system is a valid candidate for the LHC application. The thermal cycling does not affect its shear strength significantly, and its radiation resistance is satisfactory up to 1 MGy which is sufficient in the LHC radiation environment. The study has also shown that some epoxy gluing systems which cure at room temperature resist without any damage to radiation levels as high as 3 MGy.

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