# CHARACTERIZATION OF TITANIUM ALLOYS FOR CRYOGENIC APPLICATIONS

M. Reytier, F. Kircher, B. Levesy

CEA Saclay, DSM / DAPNIA / STCM Gif sur Yvette, 91191, France

# ABSTRACT

Titanium alloys are employed in the design of superconducting magnet support systems for their high mechanical strength associated with their low thermal conductivity. But their use requires a careful attention to their crack tolerance at cryogenic temperature. Measurements have been performed on two extra low interstitial materials (Ti-5Al-2.5Sn ELI and Ti-6Al-4V ELI) with different thickness and manufacturing process. The investigation includes the tensile properties at room and liquid helium temperatures using smooth and notched samples. Moreover, the fracture toughness has been determined at 4.2K using Compact Tension specimens. The microstructure of the different alloys and the various fracture surfaces have also been studied. After a detailed description of the experimental procedures, practical engineering characteristics are given and a comparison of the different titanium alloys is proposed for cryogenic applications.

# **INTRODUCTION**

The Compact Muon Solenoid (CMS) is one of the detectors to be built for the LHC project at CERN. The magnet system consists of a 4 Tesla solenoidal superconducting coil enclosed in a steel return yoke. To ensure the suspension of the cold mass inside the vacuum tank, a set of tie rods has been designed. The goal of this support system is to sustain the 225 tons of the superconducting coil and any magnetic forces due to a coil misalignment inside the yoke. Moreover, these tie rods are attached at one end on the cold mass at 4.5 K, and on the other side on the vacuum tank at room temperature.

Titanium alloy appears as a candidate for this application thanks to its high mechanical strength associated with a low thermal conductivity and low magnetic permeability. But, toughness is also an important requirement for this structural device. That is why to find the best compromise between the mechanical strength and the toughness, a characterization campaign has been realized on three alloys. A particular attention has been paid to an eventual notch sensitivity since the tie rods are threaded at both ends.

	Al	Sn	V	С	Fe	0	Ν	Н
Alloy A	5.60	2.61	-	0.011	0.05	0.072	0.036	0.0012
Alloy B1	6.38	-	4.48	0.022	0.10	0.121	0.013	0.0030
Alloy B2	6.26	-	4.27	0.014	0.13	0.116	0.0077	0.0026

**TABLE 1**. Chemical analysis (% wt)

# MATERIALS

In order to choose the proper material for the suspension tie rods of CMS, two extra low interstitial types have been melted and forged by two different suppliers. The alloy A consists of a Ti-5Al-2.5Sn ELI bar with a diameter of 110 mm, whereas the alloys B1 and B2 are of Ti-6Al-4V ELI type with a diameter of 80 mm. Alloys A and B1 come from the same producer. The control of the chemical analysis is shown in Table 1. Here, due to the cryogenic application, lowering the permissible impurity content is the first important requirement [1]. For the three alloys, the standards [2] and [3] are satisfied. The microstructure of titanium alloys also strongly depends on the thermo-mechanical conditions of the manufacturing process [4,5]. The controls realized for each alloy are presented on Figure 1. The alloy A is composed of a mix between equiaxed alpha grains and an acidular microstructure. It should also be stressed that the same microstructure is observed at different thickness in the raw bar, which reveals a good homogeneity. The alloy B1 and B2 present, near the tensile samples, the microstructure presented on Figure 1. It is characterized by equiaxed alpha grains with intragranular beta phase. But these microstructures are not homogeneous in the whole bars. The alloy B1 presents also platelike alpha grains and intergranular beta phase in the heart of the raw bar, whereas, for the alloy B2, some alignments in the rolling direction can be noticed.



**FIGURE 1**. Optical micrographs of each alloy. a) alloy A, b) alloy B1 and c) alloy B2. Observations containing the longitudinal direction of the raw bars and close to the tensile samples.



**FIGURE 2**. Shape of the samples : a) smooth tensile specimen b) notched tensile specimen c) compact tension specimen

## **EXPERIMENTAL PROCEDURE**

# **Tensile tests**

The three materials have been tested at room temperature and in liquid helium at 4.2 K. These tensile tests have been carried out at a constant cross head speed of 0.3 mm/min on both smooth and notched specimens shown on Figure 2. The load has been measured with a 150 kN cell fixed outside the cryostat and the displacement has been followed with a regular extensometer over a gauge length of 25 mm for the smooth samples and 12.5 mm for the notched specimens. Its signal is obviously calibrated at room temperature and in liquid helium at 4.2 K.

The notch shape leads to a stress concentration factor of 6.5. The ratio of the tensile strength on notched specimens (NTS) by the tensile strength on smooth specimens (TS) is used to study the notch sensitivity of each alloy and should be above one at any temperature.

#### **Fracture Toughness Measurements**

The fracture toughness ( $K_{IC}$ ) has been determined by using compact tension specimens in the LT direction (the load is applied in the longitudinal direction of the raw bars and the crack propagates in the radial direction). In order to respect the plane strain conditions, the thickness (B) of the samples is of 15 mm for the alloy A and 12.5 mm for the alloy B1 and B2 (Figure 2c). Each specimen is first fatigue pre-cracked at 77 K to start the fracture toughness test with a thin crack whose length (a) reached among half of the specimen width (W). The tests have been realized at 4.2 K with a displacement speed of 1 mm/min. To determine the mean length of the propagation, five points equally located in the thickness, have been measured after optical observations of the fracture surface.

**TABLE 2**. Tensile characteristics at room temperature

	Yield Stress (MPa)	Tensile Strength (MPa)	Elongation (%)	NTS / TS
Alloy A	$790 \pm 19$	875 ± 22	$16.2 \pm 2.1$	1.47
Alloy B1	$881 \pm 12$	$957 \pm 7$	$15.6 \pm 2.0$	1.49
Alloy B2	$827\pm7$	$897 \pm 1$	$15.2\pm1.6$	1.46

TABLE 3. Tensile characteristics in liquid helium at 4.2 K

	Yield Stress (MPa)	Tensile Strength (MPa)	Elongation (%)	NTS / TS
Alloy A	$1348 \pm 12$	$1475 \pm 1$	$9.9 \pm 0.5$	1.12
Alloy B1	$1650 \pm 13$	$1698 \pm 18$	$4.8 \pm 2.3$	0.94
Alloy B2	$1643 \pm 22$	$1673 \pm 13$	$5.1 \pm 2.5$	0.94



**FIGURE 3**. Typical tensile behavior obtained for each alloy on a) smooth samples at room temperature, b) notched samples at room temperature, c) smooth samples at 4.2 K and d) notched samples at 4.2 K.

# RESULTS

### Tensile characteristics at room temperature

The results of the tensile tests at room temperature are presented in the Table 2 on Figure 3a and 3b. No unexpected result is revealed at room temperature. The mechanical strength of the alloy A is lower than the two others, associated with the same order of ductility. The engineering properties of the three materials are in agreement with the standards [2] and [3] and also with other typical values [6,8]. No notch sensitivity is revealed at room temperature and the fracture surfaces are covered with equiaxed dimples, sign of a classical ductile rupture mode.

### **Tensile characteristics at 4.2 K**

The main differences between the three alloys are revealed at cryogenic temperature. The results are shown in Table 3 and on Figure 3c and 3d. First, the mechanical behavior in plasticity is serrated for the three materials. But alloys B1 and B2 present few load drops of high amplitude, whereas alloy A shows many little drops as presented on Figure 3c. This marked discontinuous yielding, known as adiabatic deformation [9], is still the same when decreasing the speed to 0.05 mm/min. Moreover, this behavior is so sudden for alloy B1 and B2 that the yield stress corresponds to 0.15% of plasticity whereas it has been determined with 0.2% for alloy A. The mechanical characteristics present a significant increase at this temperature, but it is associated with a loss of ductility for alloys B1 and B2. This seems to confirm the fact that the alpha type alloys (alloy A) suffer less from ductility reduction at low temperature than other titanium alloys containing beta phase (alloys B1 and B2) and that a serrated yielding at cryogenic temperature characterized by many load drops can be a sign of significant capacity for plastic deformation [9]. This loss of ductility for the alloy B1 and B2 is also associated with a notch sensitivity. The tensile tests on the notched samples reveal a notch ratio below one for these two alloys. This deformation capacity lack on a notch structure is illustrated on Figure 3d where it can be observed that the samples B1 and B2 break during the elastic loading whereas the alloy A sample sustains some plastic deformation. Moreover the fracture surface of alloy A and alloy B2 are presented on Figure 4a and Figure 5a. There is no brittle fracture surface, but alloy B2 presents a lot of secondary damage characterized by deep cracks with no apparent deformation or dimple formation. These observations also confirm that alloy A presents higher ductility capacities.

The fracture surface observations of notched samples reveal, especially near the edge, the presence of large among of elongated facets instead of classical dimples for both alloys A and B2 (Figure 4b and 5b). For Nagai [6], the appearance of these grooves would seem to correspond to a reduction in ductility or toughness and would be related to twinning deformation. Here, this phenomenon is met in both alloys but more especially on notched samples. Therefore the formation of these grooves seems to be favored by stress concentration or stress triaxiality and reveals a modification in the deformation mode near a notch. But no significant difference has been observed between the two alloy fracture surfaces.



**FIGURE 4**. Scanning electron micrographs of fracture surface for alloy A at 4.2K on a) smooth samples and b) notched samples.



**FIGURE 5**. Scanning electron micrographs of fracture surfaces for alloy B2 at 4.2K on a) smooth samples and b) notched samples.

b)

a)

b)

## **Fracture Toughness Measurements**

Finally, the fracture toughness tests presented in Table 4 confirm that the alloy A can keep deformation capacities at cryogenic temperature, even in the presence of a thin crack. It shows a significant higher toughness than the other two alloys. Our results are comparable with other values [6,8] and stresses once more that significant differences are still present between extra low interstitial titanium alloy families. The alpha type alloy (alloy A) appears softer even at cryogenic temperature, with therefore a better toughness.

	$K_{IC}$ (MPa.m <sup>1/2</sup> )		$K_Q$ (MPa.m <sup>1/2</sup> )	
	77			
Alloy A	84	$78 \pm 5$		
	74			
	58			
Alloy B1	61	$61 \pm 2$		
	63			
	67			
Alloy B2		$58 \pm 12$	60	
	50			

TABLE 4. Fracture Toughness at 4.2 K

## CONCLUSIONS

In order to choose the proper titanium alloy for the CMS tie rods, a characterization campaign has been realized on three extra low interstitial titanium alloys produced by two different suppliers.

The chemical analysis are in agreement with the standards but the study of the microstructure reveals also some lack of homogeneity for alloys B1 and B2.

No unexpected result has been found by tensile tests at room temperature. The alpha type alloy A presents lower mechanical strength associated with the same order of ductility than the two other alloys.

The major differences are revealed at cryogenic temperature. Alloys B1 and B2, containing some beta phase, present both higher mechanical properties but also lower fracture toughness and a significant notch sensitivity. Moreover, their behavior is characterized by few sudden and important load drops at the very beginning of yielding and their ductility is significantly reduced.

The microstructure obtained for the alloy A (mix between equiaxed alpha grains and acidular shape) seems to reach a good compromise between the necessary mechanical strength and the toughness for our cryogenic application.

# ACKNOWLEDGEMENTS

The authors wish to thank the CMS collaboration for its financial support as well as the technical staff of the DAPNIA / STCM involved in these tests, and more particularly S. Cazaux and G. Lemierre.

# REFERENCES

- 1. Carman C. M. and Katlin, J. M., ASTM STP, 432, pp. 124-144, (1968)
- 2. Aerospace Materials Specifications 4924 D
- 3. ASTM B 381 –97
- 4. Shannon, J.L. and Brown, W. F, *ASTM STP*, **432**, pp. 33-63, (1968)
- 5. Campbell, J. E., ASTM STP, **556**, pp. 3-25, (1974)
- 6. Nagai, K., *Cryogenics*, vol. 26, pp. 19-23, (1986)
- 7. Van Stone, R. H. and Shannon, J. L., *ASTM STP*, **651**, pp. 154-179, (1978)
- 8. Ito Y. and Nishimura T., "cryogenic properties of extra low oxygen Ti-Al-4V alloy", *sixth conference on titanium*, France, 1988, pp. 87-92.
- 9. Reed R. P., Clark A. F., *Materials at low temperature*, ASM, Metals Park Ohio, 1983, pp.237-267