iopscience.iop.org

Home Search Collections Journals About Contact us My IOPscience

Design and fabrication of a cryostat for low temperature mechanical testing for the Mechanical and Materials Engineering group at CERN

This content has been downloaded from IOPscience. Please scroll down to see the full text.

2015 IOP Conf. Ser.: Mater. Sci. Eng. 102 012007

(http://iopscience.iop.org/1757-899X/102/1/012007)

View the table of contents for this issue, or go to the journal homepage for more

Download details:

IP Address: 188.184.3.56
This content was downloaded on 14/04/2016 at 08:07

Please note that terms and conditions apply.

Design and fabrication of a cryostat for low temperature mechanical testing for the Mechanical and Materials Engineering group at CERN

I Aviles Santillana[1, 2], **R Betemps**[1], **A Gerardin**[1], **M Guinchard**[1], **S A E Langeslag**[1] **and S Sgobba**[1]

[1] CERN, CH-1211 Genève, Switzerland
[2] University Carlos III of Madrid, Campus de Leganes, Av. Universidad 30, 28911 Madrid, Spain

E-mail: Ignacio.Aviles.Santillana@cern.ch

Abstract. Mechanical testing of materials at low temperatures is one of the cornerstones of the Mechanical and Materials Engineering (MME) group at CERN. A long tradition of more than 20 years and a unique know - how of such tests has been developed with an 18 kN double-walled cryostat. Large campaigns of material qualification have been carried out and the mechanical behaviour of materials at 4 K has been vastly studied in sub – size samples for projects like LEP, LHC and its experiments. With the aim of assessing the mechanical properties of materials of higher strength and/or issued from heavy gauge products for which testing standardized specimens of larger cross section might be more adapted, a new 100 kN cryostat capable of hosting different shapes of normalized samples has been carefully designed and fabricated in-house together with the associated tooling and measurement instrumentation. It has been conceived to be able to adapt to different test frames both dynamic and static, which will be of paramount importance for future studies of fracture mechanics at low temperatures.

The cryostat features a double-walled vessel consisting of a central cylindrical section with a convex lower end and a flat top end closure. The transmission of the load is guaranteed by a 4 column system and its precise monitoring is assured by an internal load cell positioned next to the sample in the load train. This innovative approach will be discussed together with other non-conventional instrumentation solutions.

A validation of the whole system has been carried out, where bending efforts on instrumented samples have been measured. Additionally, dedicated tooling has been fabricated for the device's optimization. The preliminary results obtained confirm an excellent performance of the system and enhance the analysis of materials under extreme conditions with state of the art instrumentation.

Content from this work may be used under the terms of the Creative Commons Attribution 3.0 licence. Any further distribution of this work must maintain attribution to the author(s) and the title of the work, journal citation and DOI.
Published under licence by IOP Publishing Ltd

1. Introduction

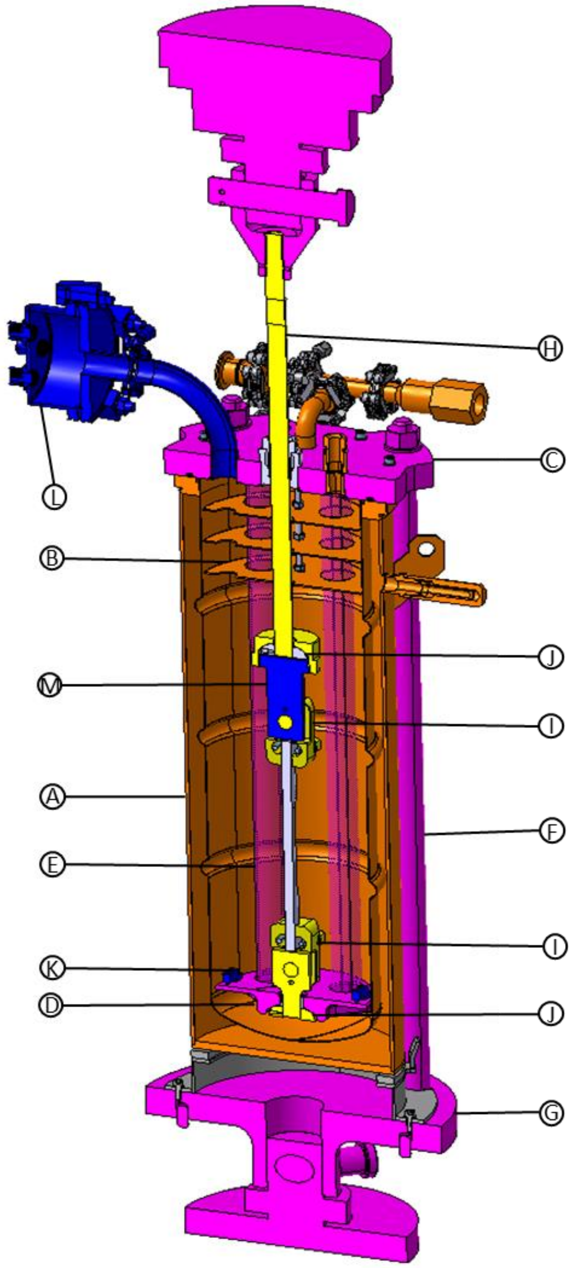
At the European Organization for Nuclear Research (CERN), mechanical testing of materials under extreme conditions has always been of great importance for the construction of state of the art equipment for the biggest particle accelerator complex worldwide. The use of cryogenics for experiments at CERN dates back to the 1960's with the track chambers, where liquid hydrogen and neon were used for working in the 30 K range at a pressure of around 5 atmospheres [1]. From the 1970's to the 1990's, the development of superconducting magnets and accelerating cavities for numerous projects (ISR, ALEPH, LEP) generalized the use of cryogenics at CERN at temperatures down to 4.5 K, and from the 1990's until today, the use of cryogenics has increased in the whole accelerator complex (superconducting accelerator and detector components in the Large Hadron Collider (LHC)). There are even centralized CERN-wide distribution lines of liquid nitrogen and liquid helium to small scale cryogenic experiments such as the Antiproton Decelerator (AD) or the Isotope Separator On Line DEvice (ISOLDE) [1].

It stands to reason that mechanical characterization of materials at low temperatures has always been one of the main pillars of the Mechanical and Materials Engineering (MME) group at CERN. In the accelerator complex, high energy magnets, superconducting radiofrequency cavities, particle detectors and all the associated instrumentation is working at a temperature below helium's boiling point (4.5 K), hence a detailed characterization and a comprehensive study of the mechanical behavior of materials in such an extreme condition becomes essential. Within this scope, a total of four reduced size cryostats like the one shown in figure 1 were designed and fabricated at CERN to be able to respond to the high demand of mechanical characterization at cryogenic temperatures. With these 18 kN cryostats, a huge number of test campaigns have been executed, not only for material characterization [2] but also for more advanced studies such as discontinuous plastic flow and local heating during tensile testing at cryogenic temperatures [3]. Despite the high reproducibility and exploitability of the results, they were obtained with use of sub-size samples according to standard ASTM E8. In the frame of new particle accelerators (Future Circular Collider), improvement of the existing ones (High Luminosity LHC) and amongst others, the push forward in high field superconducting magnets (11 T), an increasing demand of tensile tests close to the operating temperature of these High Energy Physics (HEP) devices exists. To be able to face this increasing need, the MME group decided to design and fabricate in-house a 100 kN cryostat capable of accommodating standard size tensile specimens.

Figure 1. 18kN cryostat. On the left, the female part consisting of a dewar; on the right the male part consisting of inner columns, thermal screens, sample holder and pulling rod.

2. Conception and design features

When the need of a 100 kN cryostat was acknowledged, it was also decided that the latter would have been designed, fabricated and assembled in house, profiting of the available experience of designing and fabricating cryogenic equipment and of the flexibility of a fully tailored solution. In this section, the different technical choices adopted with respect to others found in other laboratories will be presented and discussed. They are gathered in four different categories: thermal, structural, load train - sample holders and instrumentation solutions (see figure 2).



A) Dewar
B) Thermal screens
C) Top flange
D) Bottom plate
E) Inner columns
F)Outer rods
G) Bottom flange
H) Pulling rod
I) Sample holder
J) Ball joint
K) Connector
L) Instrumentation feedthrough
M) Internal load cell

Figure 2. Assembly drawing of the cryostat tensile system

2.1. *Thermal*

The thermal performance of a cryostat with such a big volume becomes very important in order to minimize cryogen consumption. The main part which ensures the thermal performance of the system is a stainless steel double-wall vessel (dewar) (A) with a 0.8 mm inner wall thickness. The convolutions shown in Figure 2 avoid buckling during the purging which needs to be completed prior to cryogen filling. Between the two walls, a thermally isolating volume pumped down to a vacuum of 10 $^{-4}$ mbar and 20 layers of a multi-layer insulation (MLI). A single dewar was used instead of the double one sometimes employed when working with liquid helium mainly to avoid complexity and to ease handling. Time between tests consumption spent in disassembling and reassembling the system for the single dewar solution greatly compensates the diminution in thermal efficiency when compared to the more complex double dewar.

Three ETP copper thermal screens (B) each 1.5 mm thick are used in the top part in order to minimize the heat exchange with the massive top plate. Copper is chosen due to its excellent thermal conductivity. The three thermal screens are pierced to provide access to the filling pipe.

For the insertion of the cryogens in the system, an inlet and outlet are located in the top flange, the former being aligned with the pierced surfaces of the thermal screens. Their leak tightness is guaranteed by a sliding rubber o – ring seal within a bronze threaded tube to prevent the gas to escape. Bronze is used for this kind of connections due to its low friction coefficient and its increased thermal contraction with decreasing temperature with respect to steel (which avoids galling) [4].

When designing new tooling, the main approach is minimization of their mass together with an increase in the contact surface with the coolant. This has to be ensured without jeopardizing the mechanical stability of the part.

All in all, the cool down time between two tests stays in the order of 20 minutes to 25 minutes if the filling is done at 1.2 bar – 1.3 bar absolute. Helium consumption for a single test is calculated to be between 25 liters and 30 liters, depending on sample ductility. These two figures prove the excellent thermal performance of the cryostat, considering its internal volume is approximately 15 liters.

2.2. *Structural*

The structural part of the system consists of four internal columns, four outer rods, a top flange, an inner bottom plate and an outer bottom plate. Their main purpose is to provide mechanical stability to the system without introducing undesired efforts. They also contain instrumentation elements such as connectors and cabling.

2.2.1. Top flange. The material for the top flange (C) is austenitic AISI 316 LN (1.4429) stainless steel conform to the CERN technical specification, which requires electroslag remelting and multi directional forging, resulting in a homogeneous product, completely austenitic and featuring a low inclusion content. The top flange has a thickness of 27 mm and contains the inlet and outlet for cryogenic fluids, a connection for the instrumentation feedthrough and a connection for the overpressure security systems (a 1.5 bar valve and 2 bar burst disk), respectively.

Three elements aim at ensuring leak tightness: a groove which is machined in the lower surface in which a rubber o – ring is placed, eight silver coated screws which pull the dewar towards the flange and four outer rods, which are tightened with a precise 50 N•m torque to secure the parallelism of the top flange and the outer bottom flange.

In order to give to the pulling rod access to the test volume, an o – ring system similar to the one used for the inlet and outlet previously described is used. This solution was chosen due to its simplicity with respect to more complicated ones like bellows. However its implementation is more delicate as an incorrect positioning of the hole in the flange would induce a misalignment and a deviation from uniaxial stress in the material studied. Because of this, the deviation from the ideal position has been measured to be lower or equal to 22 μm.

2.2.2. Inner bottom plate. The inner bottom plate (D) hosts the lower sample holder including a ball joint, the four instrumentation connectors and the four internal columns. Its design has been optimized to reduce mass without endangering its mechanical stability (figure 3). A titanium base alloy (Ti6Al4V) was chosen with the goal of maximising the yield strength while at the same time minimising the heat input into the cryostat.

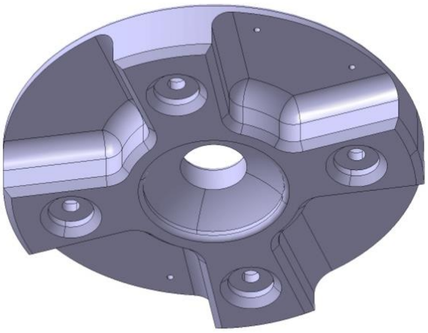


Figure 3. 3D model of the inner bottom plate

2.2.3. Internal columns. The top flange and the bottom plate are connected through four austenitic stainless steel (AISI 316L) hollow columns (E). As these elements work under compressive loads, steel was preferred to titanium due to its higher rigidity in order to avoid buckling. The wall thickness (2.6 mm) and outer diameter (30 mm) were again optimised to minimise heat input guaranteeing mechanical stability for a given permissible load. On the top part of each column, a ring was welded to ensure a correct perpendicularity of the columns with respect to the top flange. In order to have a better access to the sample and more room for instrumentation, a four columns system was chosen instead of a three columns one.

2.2.4. Outer rods. The outer rods (F) connect the top flange with the outer bottom plate. They are solid bars of 36NiCrMo16 carbon steel (1.6773), typically used for tooling subjected to high stresses. With a diameter of 28 mm, they are connected to the top and bottom flanges with a threaded M20 connection. Their length after taking into account the thread length has to be accurate to a tolerance of 100 μm (± 50 μm) to safeguard the parallelism of the two flanges and thereby avoid deviations from uniaxial stress regime.

2.2.5. Bottom flange. For the connection of the lower part of the cryostat to the tensile machine, EN S355J0 steel (1.0553) flange (G) is used. As it is shown in Figure 2, the attachment is performed with a standard pin connection, available for most universal testing machines.

2.3. *Load train and sample holders*

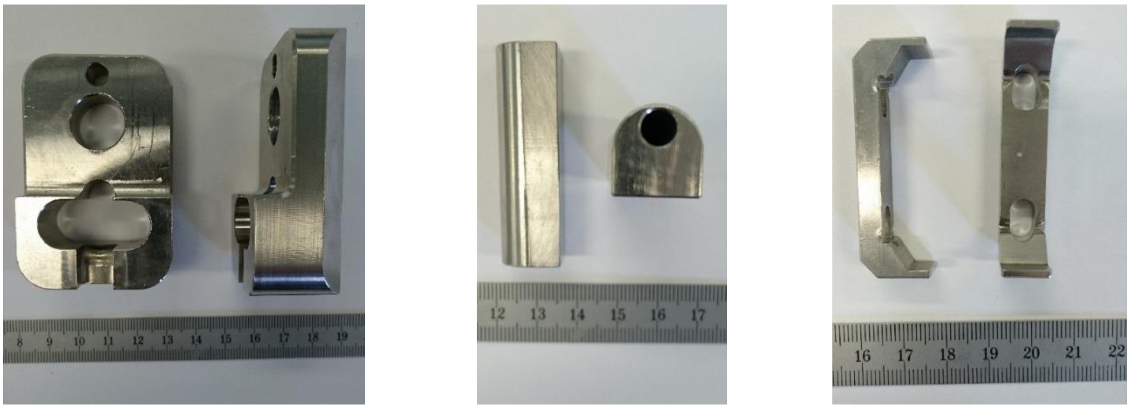
Experience has shown that in order to obtain satisfactory results during tensile testing and especially with highly brittle materials, it is essential to achieve and maintain a precise specimen alignment during the test. On this basis, the load train of the cryostat has been conceived to provide an accurate specimen alignment mainly allowed by a shoulder – headed sample holders and ball joints at both extremities, respectively.

2.3.1. Pulling rod. The pulling rod, in its lower extremity, is screwed to the upper ball joint which is in turn connected to the sample holder. Its upper extremity extends beyond the cryostat through the sliding rubber o – ring seal as described in §1.1. It is then fixed to an adaptor which is connected to the machine through a pin connection to allow free axial movement. The roughness of the rod is specified to be Ra < 1.6 µm in order to avoid high frictional forces at the o – ring seal. The material used is grade 5 titanium, which among commercial metals shows the required ductility in uniaxial tension at low temperatures and features a favourable combination of high yield strength and fair thermal conductivity, hence minimizing heat conduction into the cryostat along the rod [5].

2.3.2. Sample holders. Shoulder–headed sample holders (I) were preferred over pin connections to minimise stress concentration in the heads of the tensile specimen. A very tight shape tolerance of 0.12 µm is imposed in the shoulder region to guarantee a correct positioning of the sample and a homogeneous application of the load. The fabrication of the remainder of the tensile specimen obviously imposes the same shape accuracy. The first set of sample holders were fabricated in AISI 316L stainless steel, but in order to be able to test highly resistant materials without introducing deformation at the level of the tooling, a new set will soon be fabricated. The material properties demanded are high resistance and reasonable ductility at cryogenic temperatures, leaving a shortlist of two candidates: Ti6Al4V and Inconel 718.

The sample holders are fabricated in two identical halves which are placed symmetrically at both sides of each sample head. They are connected through a large pin to the load cell at the top and a connector at the bottom. This connection allows rotation around the tensile axis. A set of collars is placed around the two halves of the sample holders to keep them (and the specimen) in position and avoid their separation during testing or cool down.

When testing flat samples, the sample holders are designed to be able to hold samples of different thicknesses by adding a set of adaptors which prevent the movement of the specimen in the direction perpendicular to the application of the load. These adaptors, the sample holders and the collars are shown in figure 4.



a) b) c)

Figure 4. a) Flat sample holders. b) Adaptors for different thicknesses. c) Collars for sample holders

2.3.3. Ball joints. The idea of placing the tensile specimen between two ball joints comes from beam theory, which states that no bending moments can be induced in any beam section found between the ball joints (or hinges) [6]. The system is designed in such a way that one ball joint (J) and its counterpart have exactly the same geometry, while individually maximising the contact area. In this way, a better distribution of the load is achieved and hence, a lower frictional force is implied between the parts. A potential beneficial effect on the performance of the ball joints induced by solid lubricants is currently being studied.

2.4. *Instrumentation*

A key aspect during the conception of a cryogenic tensile system is the instrumentation. It is extremely important not only to guarantee an accurate monitoring of the signals but also achieving flexibility of the system, meaning creating the possibility of adding new sensors according to the test campaign to be carried out.

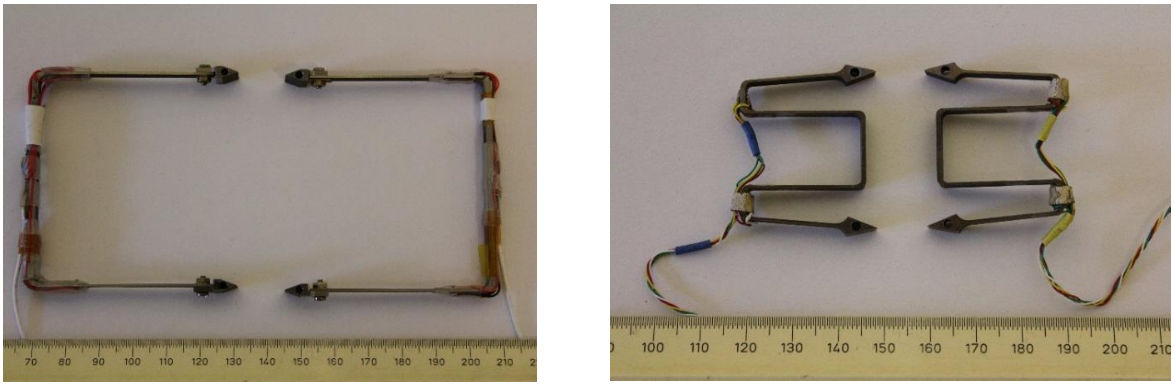
2.4.1. Connectors. Four sets of 3M® connectors (K) are attached to the inner bottom plate. One of them is dedicated exclusively to the internal load cell. Another one is used either for two extensometers or possibly for two LVDT sensors for measuring displacement. The last two are available for any additional sensors which could be required (temperature sensors, strain gages, capacitive sensors), each of them counting 12 pins. Additionally, there are four optical connections where optical fibres can be coupled, which can be used as sensors. The presence of these four optical connections opens the possibility of implementing a great variety of sensors based on glass fibre, which are unaffected by magnetic field. All the cabling associated to the connectors is sent to the outside through the instrumentation feedthrough (L) which offers a total of four Fischer® connections to read out all the different signals.

2.4.2. Internal load cell. One of the most innovative solutions which the system features is the implementation of an internal load cell (M) placed close to the top part of the specimen to be tested. The material chosen was Ti5Al2.5Sn ELI due to the combination of its high tensile properties, toughness and reasonably low fatigue crack growth rates at cryogenic temperature. The load cell measuring function is based on a Wheatstone bridge configuration formed with four strain gages acting as the four active arms of the bridge. The strain gages used for this load cell are HBM's 1-XC11-3/350, 0 and 90° T rosettes with 350 Ω resistance and a k factor of 2.22.

The design of the piece to which the Wheatstone bridge is glued to be implemented as a load cell has been designed in such a way that the top part of the sample holder can be directly mounted through a pin and the pulling rod can be easily screwed to it via a specially designed adaptor. A finite element analysis has been carried out to verify that the deformation in the flat surfaces, at the position of the strain gages, is homogeneous and it remains below 25 % of the elastic limit.

Following the instrumentation of the load cell, a calibration was performed according to the instructions of the international standard ISO 376:2011. Overall 10 tensile loading steps have been performed to up to 80 kN at room temperature. These loading steps have been repeated for six cycles overall in order to validate the measurement uncertainty of the results including factors such as repeatability, reproducibility and creep errors. According to this calibration, the sensor shows a combined standard uncertainty of **uc (F) = 1.9000e-03 + 1.8390e-02**, what makes the load cell a class 1 force proving instrument, as required by ISO 6892.

2.4.3. Extensometers. The extensometers used for low-temperature tensile testing generally utilize strain gages mounted to a bending beam element. In our case, we have two different types available: the C–shape and the W–shape (figure 5), which are used depending on the gage length of the specimen. They are both a development of the Cryogenic Engineering & Materials Expertise (CEME). The C–shape one is very suitable for standard size flat samples, with a gage length of 50 mm and a maximum elongation at break of 50 %. The W–shape is more adapted to small size standard samples, with a gage length of 25 mm. They have both been calibrated by the supplier and also in house at 4 K, 77 K and room temperature.



a) b)

Figure 5. Extensometers for uniaxial tensile tests. a) C–Shape extensometers. b) W–shape extensometers

3. Validation and results

3.1. *Validation*

For the validation of the cryostat, ASTM E1450 was used as a reference. This international standard states that the maximum bending strain should not exceed 10 % of the axial strain. The procedures for measuring specimen alignment are inspired on ASTM E1012. Flat and round (buttonhead) specimens were utilized for verification of the alignment. The loading scheme was also inspired by ASTM E1012, which states the percentage of bending has to be measured in at least three points in the elastic region, preferably equispaced between them. In our case, five points were measured, being the highest at approximately 75% of the expected yield strength of the material to avoid plastic deformation. Stainless steel AISI 304L is used for the validation.

3.1.1. Flat specimens. The specimens are fabricated following the requirements of both ASTM E8 and ISO 6892-1. Special attention was put in the shape accuracy as described in §2.3.2 and the flatness, with a tolerance of ± 15 µm. A single strain gage is glued to the parallel surfaces in the middle of the calibrated length. They are then connected in a half–bridge configuration which gives as an output the difference of strain measured over the strain gages. The tensile strain is calculated with the force measured by the internal load cell, the cross section and the Young's modulus of the material. Percentage of bending subsequently follows from the fraction of bending strain over the tensile strain. The values for ten room temperature tests in figure 6. Between every test, the specimen was demounted and remounted in the sample holders.

3.1.2. Buttonhead specimens. The specimens are fabricated following the requirements of both ASTM E8 and ISO 6892-1 for tensile tests at room temperature. In this occasion, 8 strain gages are positioned as described in ASTM E1012 in two rows: one on the top and one on the bottom. As an independent reading of the 8 signals was impossible at cryogenic temperature, the strain gages of the same row situated diametrically opposed one to the other were connected and measured as described in §3.1.1. The validation procedure was the same as for the flat specimen. figure 6 summarizes the results, which show consistently a percentage of bending below 10 % for all signals or slightly above for some isolated positions.

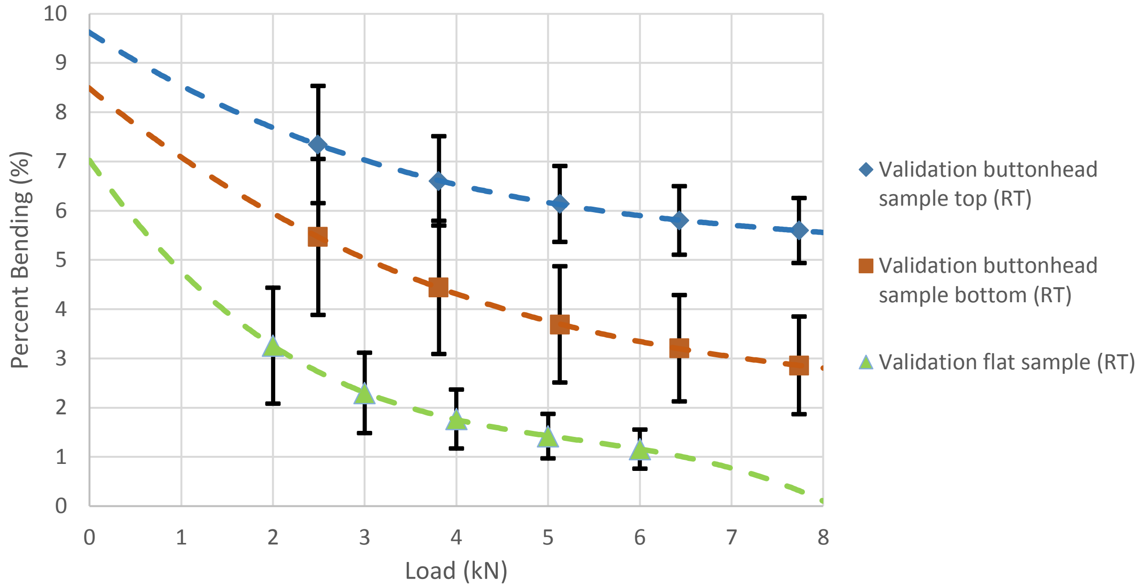


Figure 6. Summary of the results for uniaxial tensile validation tests of flat and buttonhead samples at room temperature. The trend lines correspond to a grade 3 polynomial

3.2. Results

Preliminary results for uniaxial tensile tests of two different materials are summarised in table 1. For INERMET®180 flat tensile specimens were tested at a constant stroke displacement of 0.5 mm/min. For the OFE – copper, buttonhead round specimen geometry was used. It is in a ¼ hard temper state. The stroke displacement was set to 1 mm / min. Both testing speeds are fulfilling ASTM E1450, where it is stated that strain rate should not exceed 10^{-3} s^{-1}.

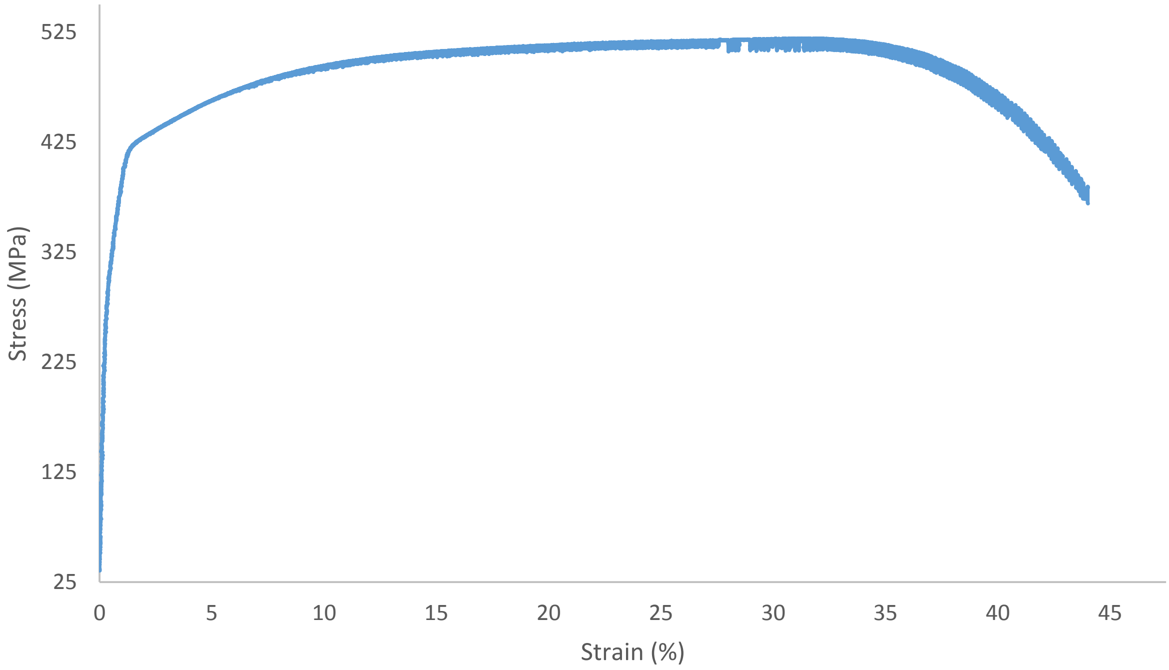


Figure 7. Stress vs strain curve for a representative sample of OFE – copper. ¼ hard temper state. Buttonhead specimen. Test speed: 1 mm/min. Serrated yielding is observed in the plastic region

The results show a very brittle behaviour for the INERMET®180, not being possible to calculate its yield strength as rupture occurs in the elastic region. For the OFE – copper, the results correspond to a cold worked temper state, with the associated increase in ductility typical at cryogenic temperatures for this material. It can be seen in figure 7 that with the current setup, discontinuous plastic flow can be observed for the three OFE – copper sample which were tested, becoming more pronounced after necking occurs.

Table1. Summary results of the tensile tests at 4 K for different materials

Material	*Rp0.2*	UTS	*A*
OFE - copper	427 MPa	525 MPa	43.4 %
INERMET®180	Broke in elastic region	1048 MPa	0.4 %

4. Conclusions

A 100 kN cryostat has been designed, fabricated, assembled and commissioned to perform uniaxial tensile testing at temperatures ranging from 300 K to 4 K. Its dimensions have been optimized to be able to work in the current UTS machine available in the EN/MME group at CERN and would also fit in a standard size servo-hydraulic machine. It is able to test different standard size specimens, which give the opportunity to assess material properties at cryogenic temperatures of flat and round products thanks to the tooling and ancillaries which have also been designed and manufactured in house.

All the different solutions which have been implemented have been comprehensively analysed and are herein presented and discussed in detail, including geometries, tolerances and materials. The instrumentation of the device, which is a key aspect of the design, has been successfully installed and calibrated. The sensors and connectors which are chosen are thoroughly described and discussed.

A validation of the cryostat has been carried out according to the international standards in use. The deviation from uniaxial stress is confined to less than 10 % even when extrapolating the trend lines to low loads. Additionally, some preliminary test results at 4 K are shown, where a tungsten alloy show a very brittle behaviour at 4 K while OFE – copper exhibits typical values obtained for a cold worked temper state, including a very high ductility. Discontinuous plastic flow is observed for all the three OFE – copper samples which were tested, confirming this phenomenon can be measured with the current setup.

5. References

[1] Barth K 2006 Operation of cryogenics for CERN experiments and LHC test facilities *Workshop on Cryogenics Operations* (California).

[2] Sgobba S and D'Urzo C, Fabbricatore P, Sequeira Tavares S 2004 Mechanical performance at cryogenic temperature of the modules of the external cylinder of CMS and quality controls applied during their fabrication, *IEEE Trans. Appl. Supercon.*, **14** (2), 556-559.

[3] Barisone E et al. 2007 Development and Use of Advanced Equipment for Measuring Serrated Flow and Local Heating during Tensile Testing of Materials at Cryogenic Temperatures EUROMAT (Germany).

[4] Ekin J W 2006 Experimental techniques for low – temperature measurements (New York: Oxford University press)

[5] McClintock R M and Warren K A 1960 Tensile cryostat for the temperature range 4 ° to 300 °K *Advances in Cryogenic Engineering, Proceedings of the 1960 Cryogenic Engineering Conference University of Colorado and National Bureau of Standards Boulder*, 372 – 378 (Colorado: Springer).

[6] Beer F P and Johnston E R Jr 2012 Mechanics of Materials (McGraw-Hill)