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Leak rate measurements on bimetallic transition samples for ILC cryomodules

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1. INTRODUCTION

In the current design the ILC cryomodules contain a large amount of structures made of Titanium, which are both difficult to manufacture and expensive. In this note we have explored techniques to significantly reduce the amount of Titanium used by means of suitable Titanium Stainless Steel transitions made with explosion bonding by a Russian company, and discuss the results obtained from tests on samples recently received.

In particular in this note we report on the tests performed by a joint INFN-Pisa and JINR-Dubna team at Pisa, Italy, on a Titanium-Stainless Steel (Ti-SS) transition sample manufactured in Sarov, Russia. These consist of leak checks under vacuum or high pressure both at room and liquid Nitrogen temperatures. Such tests are repeated after extreme thermal cycling and have the goal to certify the explosion bonding technique for future application on ILC cryomodules.

The gas leak detector used for the leak tests is the Wis Technologies MODUL200 equipped with a scroll pump (by VARIAN) to reach a preliminary vacuum level. A LabView based data acquisition system is used to read-out the temperature probes used in our tests

2. LEAK TEST MEASUREMENTS ON THE TI-SS TRANSITION SAMPLE

The Titanium-Stainless Steel (Ti-SS) transition sample manufactured by explosion bonding is shown in fig. 1. It is made of two equal diameter pipe sections, one in Titanium and one in Stainless Steel. The pipes are connected by means of a Stainless Steel collar explosion bonded on the external surface of the pipes.

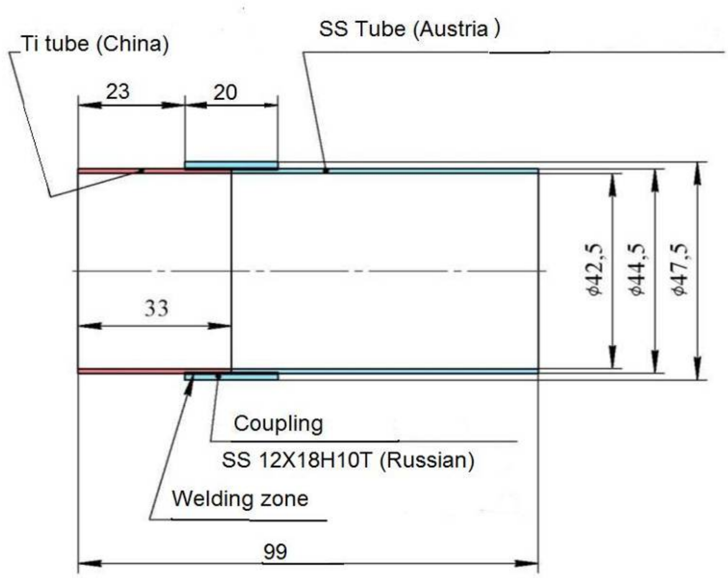
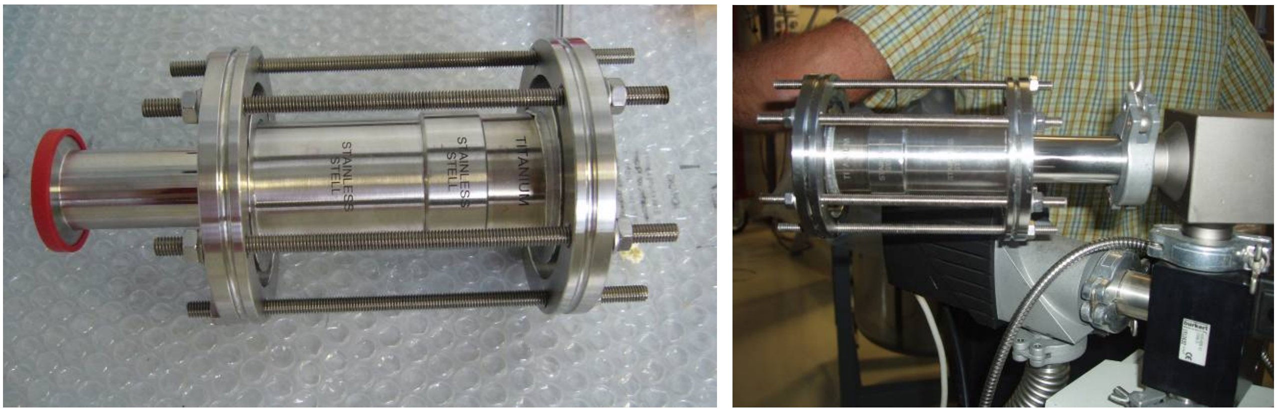


Fig.1. The applied scheme for explosion welding process..

The main goal of our measurements is to evaluate the quality of the junction obtained and its stability at cryogenic temperatures.

A dedicated set-up was made for these tests. It consisted of a blind stainless steel flange (DN63) to close the aperture on the Titanium side and a stainless steel reduction (DN63–DN25) mounted to close the stainless steel aperture from the other side. Then a DN25 connection was mounted for the gas leak detector. The both o-rings on two flanges were handmade by using the Indium wire 2 mm in diameter. The choice of this gasket was motivated by the need to carry out tests at two different temperatures: room and liquid nitrogen (300 K and 77.3 K, respectively). The two standard stainless steel vacuum components (the blind flange DN63 and the DN63-DN25 reduction) and the joint sample were assembled together with 6 stainless steel threaded rods equipped with nuts and washers assuring the right vacuum seal (see Fig. 2a).



a) b)

Fig.2. Experimental set-up of the Ti-SS transition sample:(a) - to be tested; (b) - connected to the leak detector with a C-clamp.

The same gasket (Indium wire) was applied for the connection between the flex pipe of the leak detector and the experimental set-up. The vacuum seal was completed by using a C-clamp (see Fig. 2b).

2.1 Results of the leak tests at the room and liquid nitrogen temperatures

The bimetallic sample was wrapped with a plastic bag making a small volume where the gaseous helium was flown for several seconds (typically 3-5 sec). In Fig. 3 the measurement set-up is visible during the ongoing test at the room temperature. The first measurement gave the following values:

T = 300 K
Vacuum Level = 5.7 x 10^{-3} mbar
Leak Rate background = 1.2 x 10^{-10} mbar *l/s
No changes were detected after filling the plastic bag with the gaseous helium.

Fig.3. The Ti-SS transition sample wrapped with a plastic bag for the leak test measurements.

After that preliminary test the Ti-SS joint was connected to a flex pipe, tested at room temperature again and then immersed into a cryogenic Dewar filled with liquid nitrogen for about 30 minutes (elapsed time needed to have the system at thermal equilibrium with LN_2). The obtained results are shown below:

T = 83 K
Vacuum Level = 5.7 x 10^{-3} mbar
Background Leak Rate = 6.0 x 10^{-9} mbar *l/s (at 300 K)
Background Leak Rate = 3.4 x 10^{-9} mbar *l/s (at 83 K)
No changes were detected after filling the plastic bag with the gaseous helium.

During the cryogenic test the temperatures were measured by two temperature diode sensors (model DT-630 produced by Lake Shore): the first one was attached to the sample to monitor the temperature inside the plastic bag; the second sensor was used to monitor the room temperature. In Fig. 4 we show the cool-down and warm-up temperature profiles of our sample.

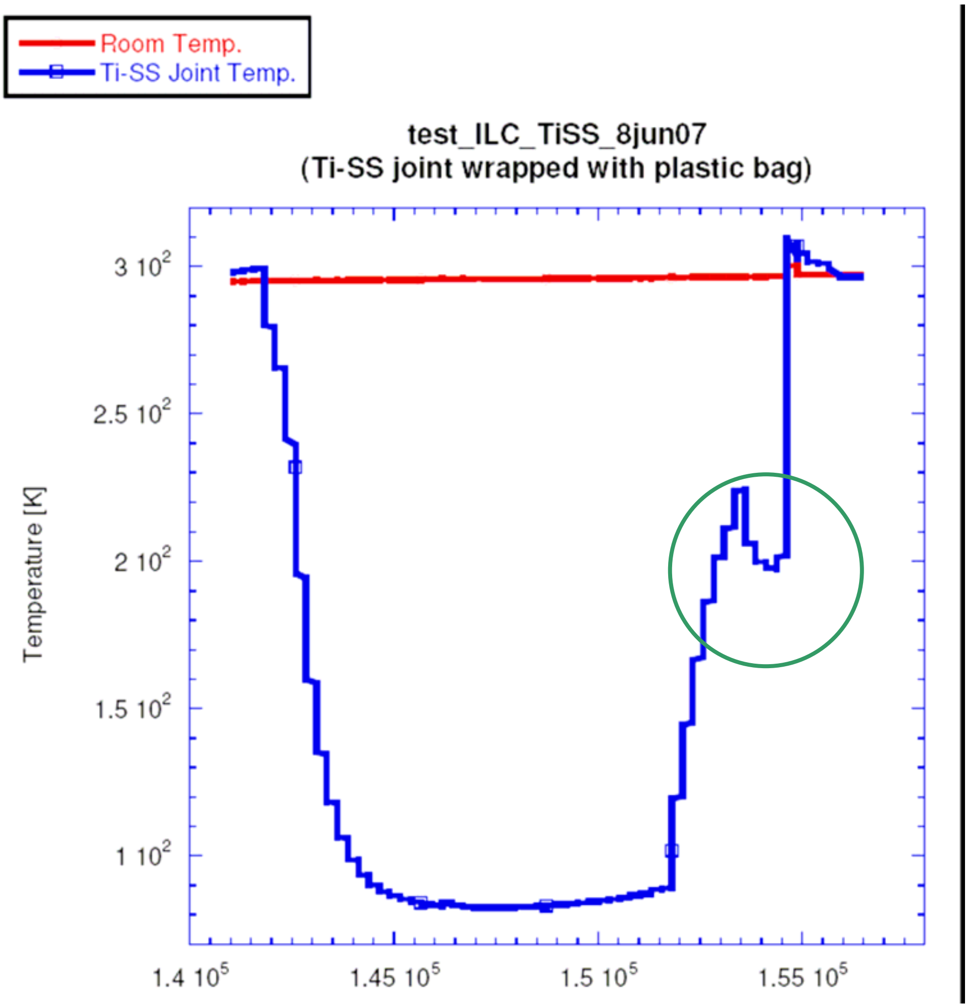


Fig. 4. The plot of the temperatures during the leak test measurement of the Ti-SS transition sample. The room temperature is given by the red curve while the blue one represents the sample temperature. On the right side of the plot a circle highlights the temperature of the second cold test.

A second leak test was also performed at about 200 K (see circle in fig. 4). The set-up was taken out from the Dewar and when the temperature reached almost 200 K the second leak test was carried out. No variation of the background level was observed:

T = 200 K
Vacuum Level = 6.7 x 10^{-3} mbar
Background Leak Rate = 8.6 x 10^{-9} mbar *l/s (at 200 K)
No changes were detected after filling the plastic bag with the gaseous helium.

2.2 Leak tests after thermal cycling

A deeper investigation on the Ti-SS transition sample behaviour is of great importance for ILC applications considering that this part will be thermally stressed passing, in standard working conditions, from room temperature down to cryogenic temperatures. For these reasons we planned to thermally stress the sample making several thermal cycles starting from room temperature and cooling down it to liquid nitrogen temperature.

By using the same experimental set-up described above without the plastic bag, a set of leak rate and vacuum level measurements has been carried out during the thermal cycles of the bi-metallic sample at 300 K and 77 K. At the end of the cycles a measurement of the sample helium leak rate at room temperature has been repeated. The photos shown in figure 5 show different moments of the test.

Fig. 5. Different moments of these tests at the VIRGO cryogenic laboratory.

The cooling down procedure was very quick: it took a few minutes. The sample remained in contact with liquid nitrogen in the Dewar for about 15 minutes (the elapsed time necessary to have the system at thermal equilibrium with LN_2). The temperature of each thermal cycle was monitored by using two sensors: the first one attached to the sample (see the blue curve in Fig. 6) and the second one - monitoring the room temperature (see the red curve in Fig.6). Then the sample was taken out from the Dewar and warmed up by means of a heat gun for fast heating and high thermal stress. The total number of thermal cycles performed in the range from the room to liquid nitrogen temperatures was seven, as shown in the two plots of Fig. 6.

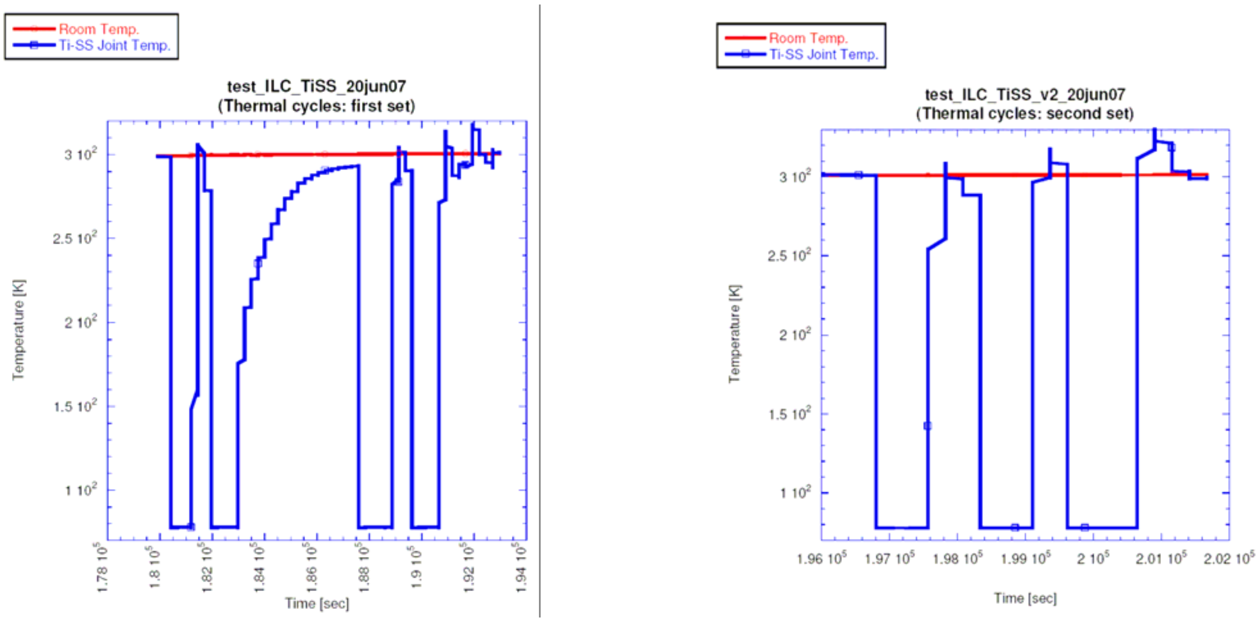


Fig. 6.Thermal cycling of the sample from the room to liquid nitrogen temperatures.

At each cycle we measured the values of the vacuum level and background leak rate both at room and liquid Nitrogen temperatures. The results are summarized in table 1.

Table 1. Total measurements of the set-up vacuum level in seven cycles.

Number of the cycle	Vacuum level, mbar		Background leak rate, mbar *l/s	
	at 300 K	at 77.7 K	at 300 K	at 77.7 K
1	6.7×10^{-3}	6.0×10^{-3}	2.4×10^{-9}	2.3×10^{-9}
2	6.7×10^{-3}	6.0×10^{-3}	1.4×10^{-9}	1.3×10^{-9}
3	6.7×10^{-3}	6.3×10^{-3}	9.0×10^{-10}	4.2×10^{-10}
4	6.7×10^{-3}	9.0×10^{-3}	1.7×10^{-9}	2.5×10^{-11}
5	7.3×10^{-3}	6.7×10^{-3}	7.1×10^{-9}	6.9×10^{-9}
6	7.0×10^{-3}	6.7×10^{-3}	5.0×10^{-9}	4.5×10^{-9}
7	6.7×10^{-3}	6.3×10^{-3}	3.6×10^{-9}	3.3×10^{-9}

It should be pointed out that between the fourth and fifth cycles the gasket (Indium wire) between the leak detector flex pipe and our sample was replaced due to vacuum tightness problem. At the end of these thermal cycles the sample was wrapped again with a plastic bag and filled by gaseous helium for the vacuum level and leak rate measurements obtaining the following results:

T = 300 K
Vacuum Level = 7.0 x 10^{-3} mbar
Leak Rate background = 3.1 x 10^{-9} mbar *l/s
No changes were detected after filling the plastic bag with the gaseous helium.

2.3 *High Pressure tests*

Since the bimetallic transition sample could be used in the liquid helium distribution circuit of the ILC cyomodule, leak detection measurements of the sample were performed also at high pressure. For this reason our sample was connected to the helium bottle and filled with gaseous helium up to a pressure of about 6 bar (Fig. 7).
Then the sample was carefully checked for the leaks by scanning the external explosion bonding joint with a probe connected to our leak detector. With this measurement technique it is possible to localize a leaking area, but the resolution on the real leak rate is considerably worse than the previous measurements. No leaks were found up to 10^{-7} mbar *l /s; this value is the best instrument sensitivity for this type of measurement.

Fig.7. The sample connected to the helium bottle and filled with gaseous helium up to a pressure of about 6 bar

The measurements of the leak rate at T=300 K gave the following results from the o-ring of the Indium wire near the flanges:

3. THE TI-SS TRANSITION SAMPLE WITH WELDED PLATES

In order to test the resistance of the bimetallic transition sample to TIG welding in its neighborhood we processed the sample previously tested as follows: a Ti plate was welded to close the Ti pipe and the stainless steel side of the joint was welded with a 3 mm thick stainless steel disk with a hole in the centre for welding a DN25 connection. The complete assembly is shown in fig. 10.

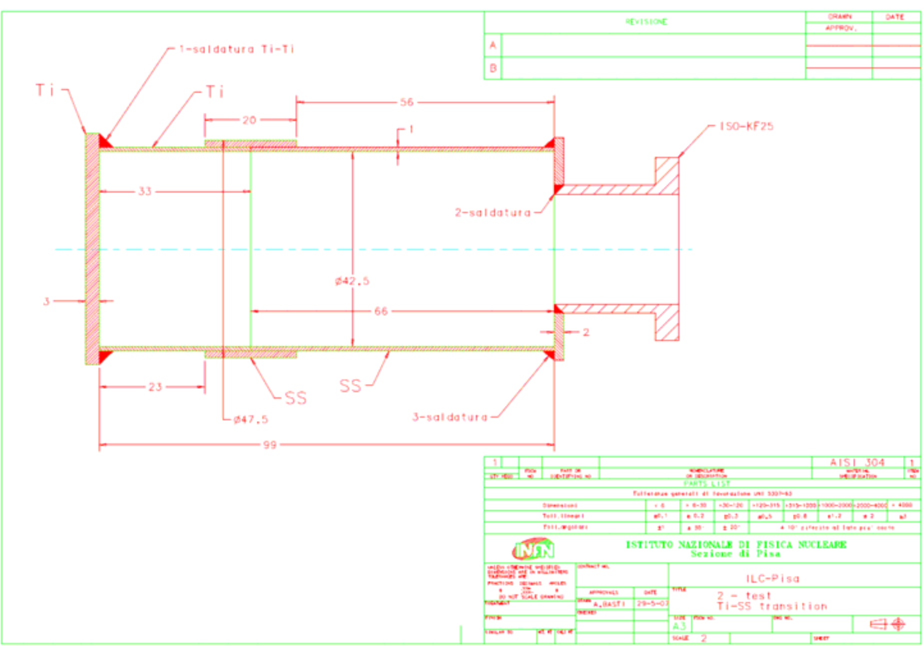


Fig. 10 – Technical drawing of the Ti-SS joint with welded components.

The welding procedure was as follows: inside the welding box we used a container with ice and water to cool the Ti-SS sample during welding (Fig.11). The water-ice level was close to the welding area. We carried out monitoring of the transition sample temperature by means of a probe connected to the external surface of the stainless steel collar. This temperature sensor is shown in Fig. 12. The welding procedure was very fast (about 5 min) and the registered temperature was always about +3˚ or +4˚ C . The finished welded assembly is shown in fig. 13.

We repeated on this welded part all the tests performed before with the simple Ti-SS transition sample using the same measurement techniques:

- leak check at the room temperature with a plastic bag filled with gaseous helium;
- thermal cycles between 300 K and 77.3 K and after the final (fifth) cycle new leak checks were performed at the room temperature;
- leak tests were carried out under high pressure (about 6 bars of gaseous helium) inside the Ti-SS transition sample.

Fig. 11.

Fig. 12.

Fig. 13.

3.1 Results of the leak test at room temperature

The results of the tests at the room temperature are given below.

T = 300 ˚K
Vacuum Level = 6.7 x 10^{-3} mbar
Leak Rate background =3.4 x 10^{-9} mbar *l/s
No change has been detected after filling the plastic bag with the gaseous helium for 3-5 seconds.

3.2 *Leak test of the sample after thermal cycling*

The procedure of this test has already been described in section 2.2. It was repeated with the Ti-SS transition sample having the welded components. Figures 14 and 15 show the measuring procedure.

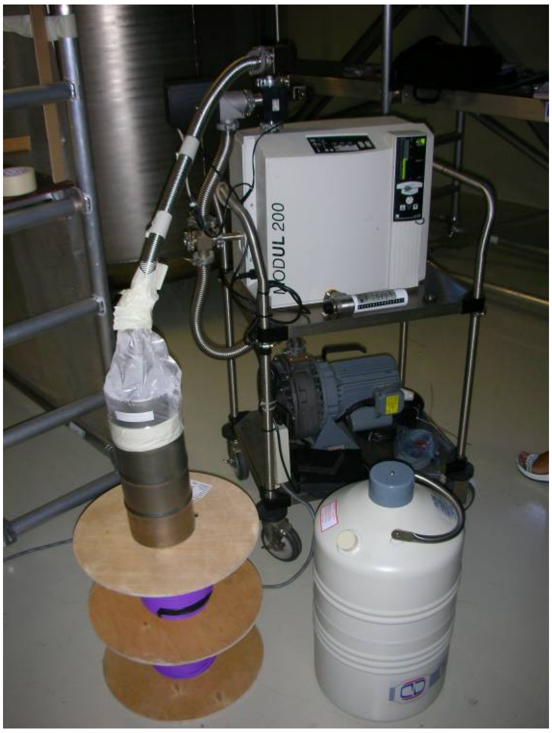


Fig. 14


Fig. 15.

The results obtained are given in Table 2 for 5 cycles at two different temperature levels. It should be noted that during the forth cycle we did not record the vacuum level and background rate for our forgetfulness, but not relevant changes have bee observed.

Table 2. Total measurements of the set-up vacuum level in five cycles.

Number of the cycle	Vacuum level [mbar]		Background leak rate [mbar *l/s]	
	at 300 K	at 77 K	at 300 K	at 77 K
1	7.0×10^{-3}	6.0×10^{-3}	1.0×10^{-9}	1.1×10^{-9}
2	6.3×10^{-3}	6.0×10^{-3}	9.0×10^{-10}	1.1×10^{-9}
3	6.3×10^{-3}	6.0×10^{-3}	3.5×10^{-10}	2.3×10^{-10}
4	-	-	-	-
5	6.0×10^{-3}	6.3×10^{-3}	3.9×10^{-9}	2.3×10^{-9}

The plot in Fig. 16 illustrates the thermal cycling process.

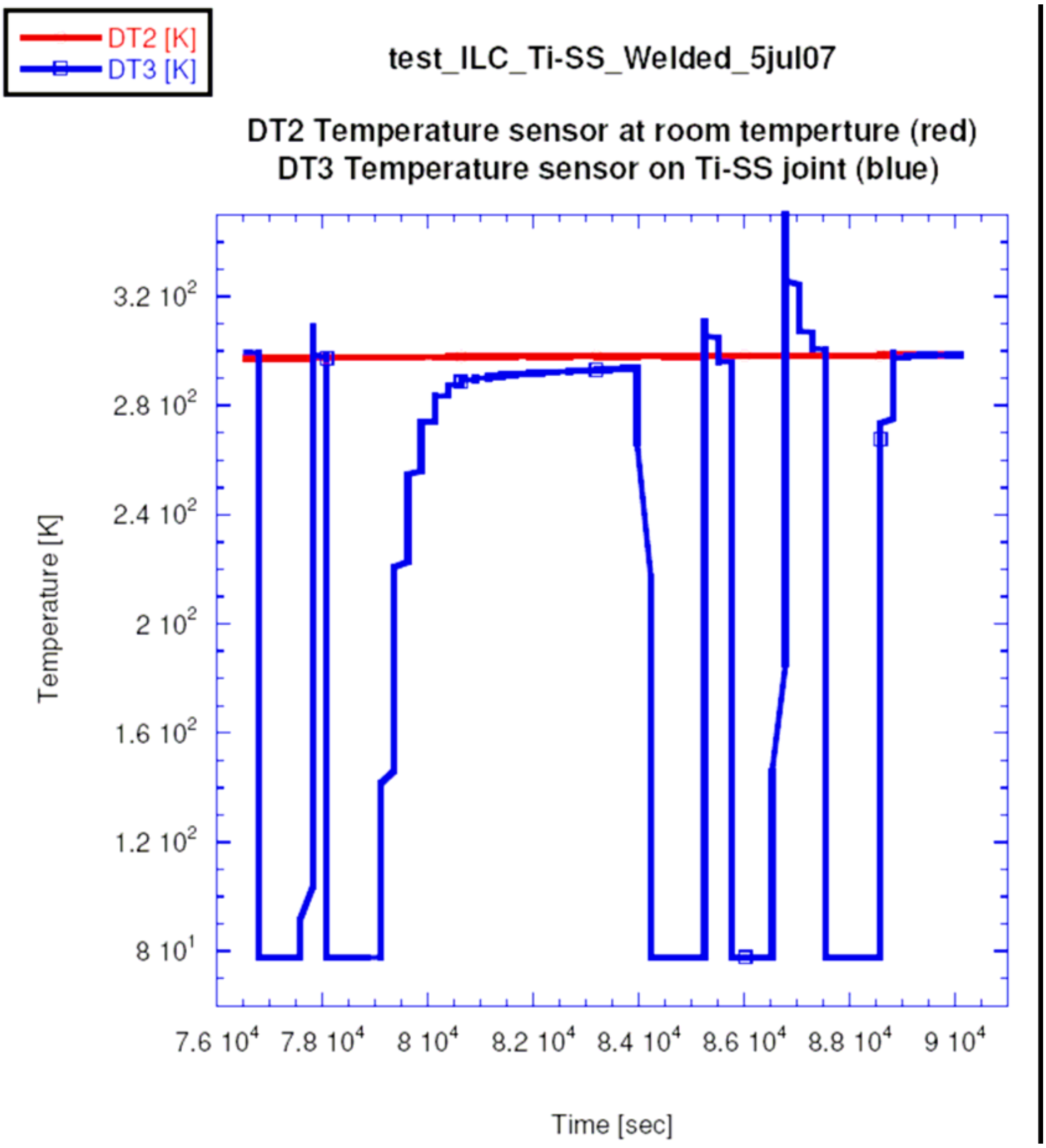


Fig. 16. The plot of the temperature during the leak test measurement of the Ti-SS sample. The room temperature is given by the red curve while the blue one represents the sample temperature.

After the last thermal cycle the sample was warmed up to room temperature with a heat gun and the vacuum level and leak rate background were measured. The results are shown below.

T = 300 ˚K
Vacuum Level = 6.3 x 10^{-3} mbar
Leak Rate background =3.5 x 10^{-10} mbar *l/s

After filling the bag with He gas we observed a significant leak. After careful study such leak was traced to a problem with the SS-SS weld. Unfortunately this prevented any further test on this sample.

4. CONCLUSIONS

The Ti-SS transition sample under consideration has shown excellent behaviour both at the room and liquid nitrogen temperatures as well as under high pressure tests and after thermal cycling. The helium leak rate of the sample was ($1x10^{-10}$ ±10%) mbar* l/s. This result is very good for initial tests of this type of welding, i.e. – by using the explosion bonding technique. To obtain more reliable data and statistics, it is necessary to perform similar tests with a larger number of samples. These experiments are supposed to be continued to reach this goal. We are also planning to carry out additional tests with the samples at liquid helium temperature. A metallographic analysis of the explosion bonding welded joint will also be performed to get more information about the structure of the welded surface and the surrounding area.