SIMPLE ALUMINUM SEALING AGAINST SUPERFLUID HELIUM

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Abstract

Indium sealing is well established for superfluid helium in superconducting RF cavity applications but its sticky material property often causes an indium contamination in the cavity disassembly. DESY has already successfully developed aluminium alloy sealing so-called diamond ring instead of indium sealing. KEK is also looking for other simple, less expensive and reliable sealing method for superfluid helium. We are developing pure aluminium fine ring sealing having a square cross-section 1mm x 1mm. We have tested it for several material combinations of flange and bolt. Very promising results were obtained. In this paper the results will be reported.

PURE ALUMINUM FINE RING SEALING

KEK is developing pure aluminium fine ring sealing for contamination-free cavity final assembly as seen in Fig.1. Indium sealing has been used for a long time as a leak tight sealing against superfluid helium circumstance, however, its sticky material often causes particle contamination problem when it is removed from the flange. In addition, it is too soft to set on vertical flanges and needs a special tool, which complicates the cavity assembly. On the other hand, pure aluminium is soft enough to make tight sealing and stiff to keep the shape by itself. It has no problem for sealing channel, which makes easy the vertical flanges assembly. Pure aluminium sealing requires a harder material for the flange than high purity niobium. In our case the flange material is supposed to be stainless steel (SS) or titanium (Ti). In case of titanium flange, electron beam welding is easy to weld it to niobium tubes, but a question is still open for the super leaking because the titanium oxide film often has cracks. Tapping is easy on the stainless flange, which simplifies the cavity assembly but it needs a special bonding with niobium, which is now under developing in KEK using HIP technology or brazing [1]. Here, we separate the two problems: leak tightness of the aluminium fine ring sealing on stainless flange and how to get the reliable bonding stainless flange to niobium tube. We will describe the latter in other paper somewhere. In this paper we concentrate ourselves to confirm the leak tightness on the pure aluminium fine ring sealing for superfluid helium circumstance.

Figure 1: Pure aluminium fine ring sealing and stainless flanges bonded on niobium tubes.

FLANGE STRUCTURES AND THE THERMAL CYCLE TEST PROCEDURE

In this report, we have tested two material combinations (SS/SS and Ti/SS) between flange and volt with three flange structures: A, B and C in Fig.2. Both flange surfaces are flat in the type A. In the type B, top flange is flat and bottom flange has a circular channel 0.6 mm deep and 2 mm wide. The type C is the current KEK cavity flange structure and both flange surfaces are flat.

The original pure aluminum sheets (JIS1050) have the size 1m wide, 2m long and 1, 1.2 and 1.5 mm thick. The surfaces were protected by thin plastic film not to be scratched on the surface during the delivery. They were cut into small square sheets by sharing machine. Pilling off the films, stacking them, they were cut at once to fine rings by electric discharge machine (EDM). The fine rings were etched a little bit in nitric acid before the use. Some rings were annealed at 500^{O}C for one hour in vacuum before the use. The details are in Table 1. The flange combination is also summarized in Table 1. The surface roughness of the SS flanges were 2.52±0.15 microns (R_Z) and 3.38±0.30 microns (Rz) with Ti flanges. All of these flanges were annealed at 750^{O}C for 3 hours in a vacuum furnace, which was the simulation of the current KEK's niobium cavity preparation procedure. Two kinds of bolt material: stainless steel and aluminum alloy are tested with leak tightness. In addition, three kinds of sealing material were tested for leak tightness: indium, pure aluminum

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and pure copper. Fig.3 shows the leak test procedure in our thermal cycle test. Flange was tightened by the final torque 150kgcm. It was baked at 120^{O}C for one hour, which was the simulation of baking procedure for niobium cavities. After that, it was checked leak tightness at the room temperature. Sensitivity of the leak test was 1 x 10^{-8} Torr cc/sec. Then, the flange was directly immersed in liquid nitrogen, which was a very hard heat shock. Flange was cooled to the liquid nitrogen temperature (77.4K) in several minutes and kept for several ten minutes until the bubbling became quiet in the liquid nitrogen. After warming up to the room temperature, leak tightness was checked. The procedure between baking and leak test after the heat shock by liquid nitrogen was repeated 5 times. On the way, any retightening of the bolts was not done. The stainless tube on the top flange was welded (Argon welding) to that in the cryostat stand and evacuated by a helium leak detector with a turbo molecular pump (50 liters/sec). The system was well baked for one night to reduce the helium background. Flange was cooled to 4.2 K in one hour.

The sealing flange was cooled to about 1.5K. Subsequently during the cool down, leak tightness was in-situ monitored by the helium leak detector. After confirming leak tightness at 1.5K, the flange was hermetically closed by a metal valve at the cryostat top flange. The sealed flange was exposed to superfluid helium for 2 hours.

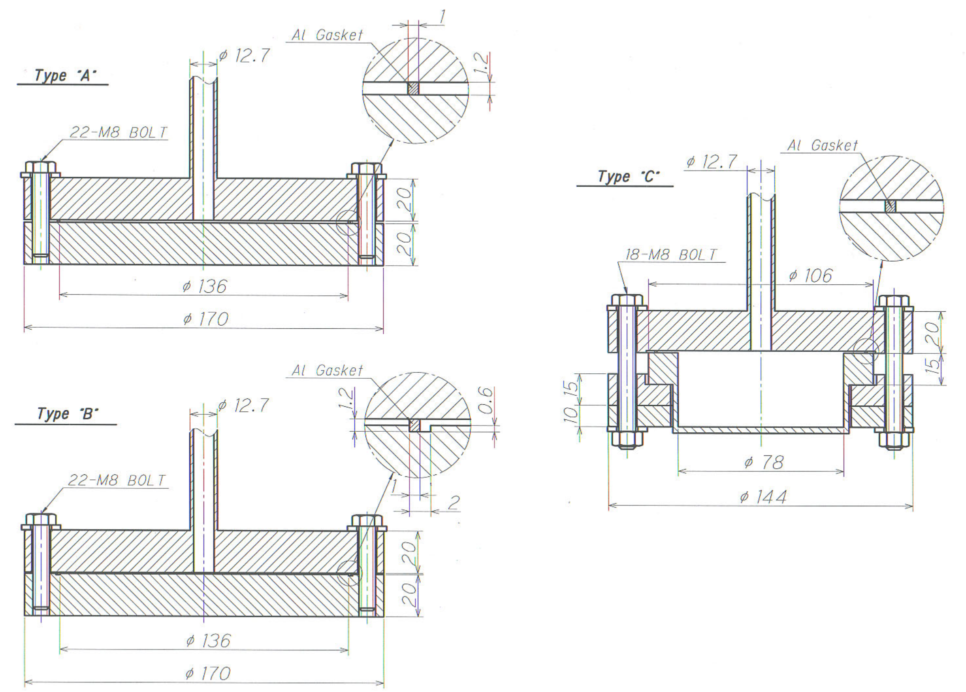

Figure 2: The flange structures.

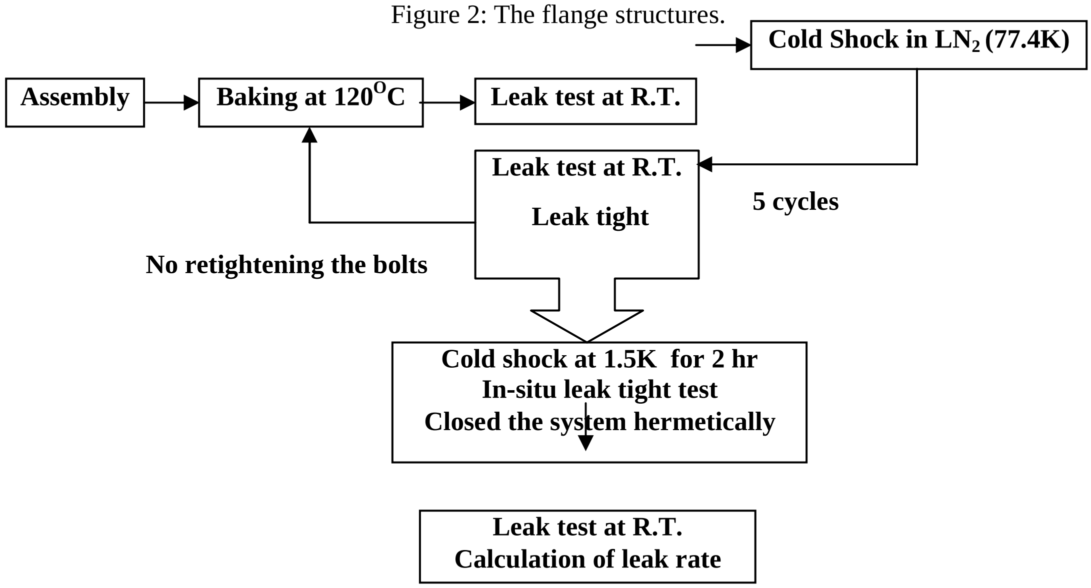

Figure 3: Procedure of the thermal cycle test in this experiment.

After warming up to the room temperature, the system was opened. The helium gas in the hermetically sealed system was evacuated by the helium leak detector. The output signal from the leak detector was integrated and calculated the total amount of helium gas penetrated during the superfluid helium exposure. This method is the same as Rao's method [2] in JLAB but he uses a high sensitive RGA for measurement of partial helium gas pressure. This method can improve the sensitive by several orders of magnitude, which is proportional to the exposing time. In our case, the sensitivity was upgraded by 2 - 3 orders of magnitude compared with normal leak checking method at room temperature.

TEST RESULTS

INDIUM SEALING

Indium sealing is well established as leak tight sealing against superfluid helium. We made test it as a reference. The flange configuration: SS/SS, flat/flat surfaces and using SS bolts (type-A in Fig.2), was tested twice for the leak tightness. Both had no detectable helium leak during in-situ leak checking. The averaged leak rate by the high sensitive leak test after warming up was $5.2x10^{-11}$ and $1.5x10^{-10}$ atm cc/sec. The test results are summarized in Table 2. The other combination was also tested by the same experiment method, where the used bolt material is different, i.e. aluminum alloy. It was leak tight with the liquid nitrogen, but is not yet tested with the superfluid liquid.

PURE ALUMINUM FINE RING SEALING

This leak tightness with superfluid helium was measured for several flange combinations: 1) SS/SS, flat/flat surfaces, SS bolts, 2) SS/SS, flat/flat surfaces, aluminum alloy bolts, 3) SS/SS, flat/channel surfaces, aluminum alloy bolts, 4) Ti/S, flat/flat surfaces, SS bolts, 5) Ti/SS, flat/flat surfaces, aluminum alloy bolts and 6) Ti/SS, flat/channel surfaces, aluminum alloy bolts.

SS/SS(flat/flat)-aluminum sealing-SS bolts

In this combination, a pure aluminum fine ring of inner diameter 136 mm was sandwiched between flat SS flanges (type-A in Fig.2) and tightened used SS bolts at the final torque of 150kgcm. Leaking occurred after the forth LN_2 thermal shock.

SS/SS(flat/flat)-aluminum sealing-aluminum alloy bolts

This is only different from above 3.2.1 in the used bolt material, i.e. aluminum alloy. This case was leak tight after the 5 times LN_2 heat shocks. Any detectable leaking was not found by the in-situ leak test during the superfluid helium exposure. The averaged leak rate by the high sensitive leak test at room temperature was $5.8x10^{-12}$ atm cc/sec. This tightness is better than that of the indium sealing in 3.1 by one order of magnitude.

SS/SS (flat/channel)-aluminum sealing-aluminum alloy bolts

We need the seal surface configuration: flat/channel for the vertical flange assembly in final cavity assembly for a cryomodule. As we succeed to have leak tight sealing with the pure aluminium fine ring sealing by using aluminium alloy bolts, we made further test for this configuration. In this test, we used a 1.5 mm thick pure aluminium fine ring for the 0.6mm deep channel. This configuration was also successfully leak tight for superfluid liquid helium and the average leak rate was $3.5x10^{-11}$ atm cc/sec. This tightness is similar to the indium sealing.

Ti/SS (flat/flat)-aluminum sealing-SS bolts

Titanium flange is easy to join niobium beam tube by electron beam welding, however, the titanium-oxide film on the surface is worried about the cold leak in superfluid helium. We made test the pure aluminium fine ring sealing (I.D. 90mm) sandwiching between a flat SS flange and a flat titanium flange (type C in Fig.2). These flanges were tightened using SS bolts. In this case, leaking occurred after the 4th thermal cycle with LN_2.

Ti/SS (flat/flat)-aluminum-sealing-aluminum alloy bolts

The same flange configuration was tested changing the SS bolts to aluminium alloy bolts. This case was leak tight after the superfluid helium exposure. The average leak rate was $7.2x10^{-12}$ atm cc/sec by the high sensitivity method. The tightness is better than indium sealing in 3.1 by one order of magnitude. This configuration was successfully reconfirmed the leak tightness with LN_2 shocks. The superfluid helium test is not yet done.

Ti/SS (flat/channel)-aluminum sealing-aluminum alloy bolts

By the same reason as above 3.2.3, we tested the configuration with aluminum sealing: Ti/SS-flat/channel-aluminum alloy bolts. In this case, any detectable leak was not observed during superfluid exposure. The averaged leak rate was $8.8x10^{-12}$ atm cc/sec by the high sensitive method. The tightness is better than indium sealing.

COPPER FINE RING SEALING

Copper gasket is often used for small ICF flanges in superfluid helium however, it is not guaranteed in the superfluid helium temperature with larger flange size than ICF70. In addition, the edge structure of ICF flanges is expensive for machining compared with flat flanges. In this experiment, we made test how fine copper ring sealing works for superfluid helium.

SS/SS (flat/channel)-Cu sealing-SS bolts

In this configuration (type B in Fig.2): SS/SS, flat/channel and SS bolt, leak happened after the 4th LN_2 thermal cycle. The reason might be in the wrong use of bolt material.

Table 1: Materials of the flanges and bolts

Type	Flange*			Bolt	Sealing		
	Material of flanges	Surface and flange structure	Annealing 750°C 3hr (No chemistry after the annealing)	Material	Material	Thickness	Annealing 500°C 1hr
1	SS/SS	flat/flat, Type-A	Yes	SS	In	1□ wire	No
2	SS/SS	flat/flat, Type-A	Yes	SS	Pure Al	1t x 1w ring I.D 136mm	No
3	SS/SS	flat/flat, Type-A	Yes	Al-alloy	In	1□ wire	No
4	SS/SS	flat/flat, Type-A	Yes	Al-alloy	Pure Al	1t x 1w ring I.D 136mm	No
5	SS/SS	flat/0.6 mm deep & 2mm wide channel, Type-B	Yes	Al-alloy	Pure Al	1. 5t x 1w ring I.D 136mm	Yes
6	SS/SS	flat/0.6 mm deep & 2mm wide channel, Type-B	Yes	SS	Cu	1. 2t x 1w ring I.D 136mm	Yes
7	SS/SS	flat/flat, Type-A	Yes	Al-alloy	Cu	1.2t x 1w ring	Yes
8	Ti/SS	flat/0.6mm deep & 2mm wide channel, Type-B	Yes	Al-alloy	Pure Al	1. 2t x 1w ring I.D 136mm	Yes
9	Ti/SS	flat/flat, Type-C	Yes	SS	Pure Al	1t x 1w ring I.D 85mm	No
10	Ti/SS	flat/flat, Type-C	Yes	Al-alloy	Pure Al	1t x 1w ring I.D 85mm	No
11	Ti/SS	flat/flat, Type-C	Yes	Al-alloy	Pure Al	1t x 1w ring I.D 85mm	No

*Flange was annealed before heat-cycle tests simulating the hydrogen outgas annealing of the sc cavities.
Surface roughness of the flanges: SS flange R_Z=2.52±0.15 microns, Titanium Flange R_Z=3.38±0.3 microns

Table 2: Leak test results

No.	Flange	Bolt	Seal	Flange and structure	400K → 77K (liquid nitrogen) quick cooling cycle test					300K→1.5K cooling test Result and the leak rate (exposed LHe-II for 2hr)	COMMENTS
					1	2	3	4	5		
1	SS/SS	SS	In	flat/flat, type-I	○	○	○	○	○	○ 5.16E-11	Tested twice Okay
					○	○	○	○	○	○ 1.46E-10	
2	SS/SS	SS	Al	flat/flat, type-I	○	○	○	X			77K leak
3	SS/SS	Al-alloy	In	flat/flat, type-I	○	○	○	○	○	Not tested	
4	SS/SS	Al-alloy	Al	flat/flat	○	○	○	○	○	○ 5.8E-12	Okay
5	SS/SS	Al-alloy	Al	flat/0.6 mm deep & 2mm wide channel, type-I	○	○	○	○	○	○ 2.8E-11	Tested twice Okay
					○	○	○	○	○	○ 3.5E-11	
6	SS/SS	SS	Cu	flat/0.6mm deep & 2mm wide channel, type-I	○	○	X				77K leak
7	SS/SS	Al-alloy	Cu	flat/flat	○	○	○	○	○	Not tested	
8	Ti/SS	Al-alloy	Al	flat/0.6mm deep & 2mm wide channel, type-I	○	○	○	○	○	○ 8.8E-12	Okay
9	Ti/SS	SS	Al	flat/flat, type-II	○	○	○	X			77K leak
10	Ti/SS	Al-alloy	Al	flat/flat, type-II	○	○	○	○	○	Not tested	
11	Ti/SS	Al-alloy	Al	flat/flat, type-II	○	○	○	○	○	○ 7.2E-12	Okay

SS/SS (flat/flat)-Cu sealing-aluminium alloy bolts

In this configuration (type A in Fig. 2): SS/SS, flat/flat surface, aluminium alloy bolt, it was confirmed leak tight after 5th LN_2 thermal cycle but the further experiment is not yet conducted. The use of aluminium alloy bolts would be the reason for the leak tightness with the LN_2 cycle tests.

DISCUSSION

Through these experiments, one will find out that use of aluminum alloy bolts is very effective for the leak tight sealing. The reason might be in the integrated thermal expansion coefficient. Table 3 shows the values for aluminum alloy, stainless steel, copper and titanium, which are integrated from 300K to 2K, 77K and 400K. Aluminum alloy has a biggest number for cryogenic temperatures. The sealing will be more tightened by the large thermal shrinking of the aluminum alloy bolts and that results in the leak tight sealing. However, in contrast this effect is worried for baking, where aluminum alloy bolts are loose and might result in leaking at 120^{O}C. This question is still open, and must be tested soon.

The leak tightness for the superfluid helium has no concern of the flange surface combinations: flat/flat surfaces or flat/channeled surface. Pure aluminum fine ring gets a better sealing if one uses aluminum alloy bolts. Titanium flange also can be used for the superfluid helium.

Table 3: Integrated thermal expansion coefficients ($x10^{-3}$) with various material.

Materials	300K-2K	300K-77K	300K-400K
Al	-4.31	-4.10	+2.40
SS	-3.54	-2.72	+1.53
Cu	-3.31	-3.09	+1.71
Ti	-1.56	-1.49	+0.90

SUMMARIES

We made sealing test with superfluid liquid helium and got the following results:

1) If one uses aluminum alloy bolts, pure aluminum fine ring is useable for superfluid helium.
2) Titanium flange also be usable for superliquid helium if aluminum bolts are used.

REFERENCES

[1] K.Saito et al., Proc. of the 10th Workshop on Superconductivity, 2001, Tsukuba, Japan, pp.531-534.

[2] M.G.Rao, “High sensitivity He desorption leak detection method”, J. Vac. Sci. Technol. A 11(4), Jul/Aug 1993, pp.1598-1601.