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THE INFLUENCES OF MATERIAL PROPERTIES TO MICRO DAMAGES ON VACUUM CHAMBER CF FLANGES

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Abstract

The European-XFEL, a 3.3 km long X-Ray laser facility, powered by a 17.5 GeV superconducting linear accelerator, is located at DESY in Hamburg [1]. For the diagnostics ultra-high vacuum components with high mechanical precision and strict requirements on particle cleanliness had to be developed, designed and produced. For the screen system of the facility, enabling to observe the size and shape of the electron beam, massive vessels, precisely milled out of stainless steel blocks 1.4435 (316L) have been produced. For these chambers all flange-connections are milled into these blocks. This paper will report on micro damages in these integrated knife edges and will present simulations of the damage mechanisms. It will also describe the influences of material properties of two different stainless steel brands, effects on the "knife edge" due to the penetration into the gaskets as well as the non-elastic deformation of the sealing area. The dependence of tightening forces under special conditions, like the very clean conditions in particle free applications due to the non-lubricated conditions will be reported. A "cooking recipe" to avoid such micro damages will be given.

INTRODUCTION

Particle accelerators are used as standard as well as and special diagnostics, also for the European XFEL (E-XFEL) at DESY, Hamburg. A scintillation screen monitor system [2] has been designed for diagnostic purposes in particle beams. For this system two different types of special vacuum chambers have been fabricated. More than 50 vacuum chamber with weights over 50 kg have been fabricated out of massive blocks. These two versions of chambers are varied, one with five and the other with eight Conflat (CF) [3] knife edge flanges allowed attaching other diagnostic devises. In this paper the two types call type A/C and B. For type B see Fig. 1.

The biggest amounts of the chambers have to be assembled and installed in particle reduced environment in different sections of XFEL. The vacuum leak for the UHV system rate has to be below $1*10^{-10}$ mbar l/s and tightened with copper flat gaskets.

The geometry of these chambers had to be chosen so that they match the requirements of independency and stability; therefore a massive stainless steel (SST) block was used.

SST is one of the most commonly used constructional materials for vacuum chambers and other vacuum components in general. Applications like vacuum vessels in linear accelerator or storage ring facilities require ultra-high vacuum (UHV). Finding an appropriate source for the raw material in 316 LN was the first step. The dimensions of these vessels are bigger than all designs at DESY have been in the past. Since it was very difficult to find any supplier for material in 316 LN, a compromise was found to use 316 L.

The raw material was ordered according to DESY material specification for 316 L. Essential a 3.1 certification was sent before the order was placed. In this specification of DESY the boundaries of hardness are between 160 and 200 Brinell Hardness (HB). In the material certification the value for the hardness was a little bit under the minimum bounder of DESY's specifications with 158 HB. The raw material was accepted by DESY. All vacuum chambers were manufactured by outside vendors at two companies. Rest gas analysis was not specified. The final assembling has been done at DESY.

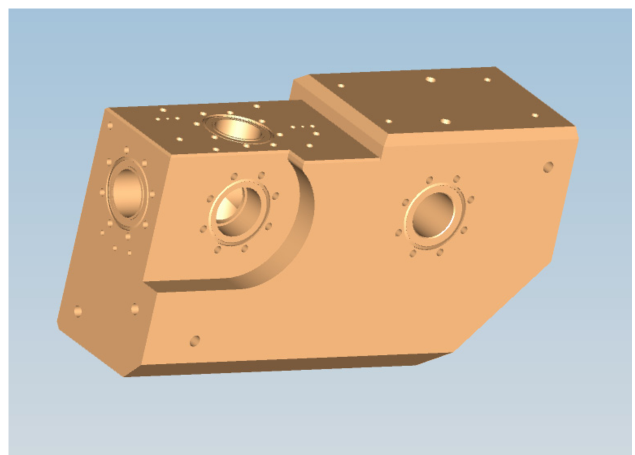

Figure 1: Show a vacuum chamber Type B with adaption ports for other diagnostic devices.

The two prototypes, one for each version, have been successfully tested under UHV conditions at DESY group Machine Diagnostic and Instrumentation (MDI).

After a European wide open call for tender the two versions were placed at two different manufactures in Germany. The cleaned and packaged chambers arrived at DESY and they had to be stored for about 1 year before final assembling. During the final assembling, in a class 5 cleanroom, cleanness problems were detected on one of the attached vacuum window. Due to these problems all chambers had to be dismounted and inspected. During this inspection by chance micro damages on the sealing areas of the CF knife edge were detected. DESY calls this appearance "orange skin" (see Fig. 2). After a few chambers have been cleaned and reassembled the final leak check had shown vacuum tightness problems.

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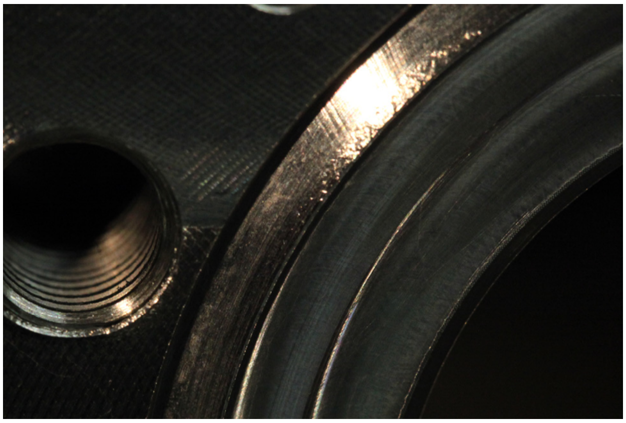

Figure 2: Show of the micro damages (“orange skin) on sealing surface.

The SST material characteristics turned out to be more complicated as expected. The challenge was to produce these massive vacuum chambers without quality problems. Therefore more detailed specifications had to be defined.

GENERAL INFORMATIONS OF SST MATERIAL, MANUFATURING AND VACUUM SEALING TECHNOLOGY

The first industrial development with stainless steel is dating to around 1910. Since the beginning SST has become more and more recognized. The none magnetic properties, durability of surface conditions, good welding properties and exceptional corrosion resistance have made this material group one of the major materials for constructing vacuum systems in accelerator technology.

All institutes worldwide have made their own experiences in building a lot of different accelerators. Most of them have different material specifications. The different accelerators can roughly be classified in warm or cold and linear or circular accelerator. All require different vacuum flange technology, boundary, purity classification and material permeability.

Therefore, there is a need for better knowledge of the intrinsic properties of stainless steels, including corrosion and oxidation resistance, high temperature mechanical strength, material peculiarity and together with all the other characteristics necessary for their use as material.

But even more important are the properties to be processed by various cold and hot forming techniques, like the remelting processes electro-slag or vacuum remelting and the different methods of joining and vacuum brazing. The gasket sealing technologies are defined by the American Vacuum Society [4]. The goal was to seal vacuum connections effectively by compressing a gasket between parts to be sealed.

The choice of the adequate flange/gasket system is driven by the requirements of the vacuum system. Different material selections for gaskets are on the market.

They can be separated into two groups, metal and non-metal gaskets. For both groups there are gaskets for single and multiple use. Another difference is given by the geometry of the gasket. For CF, ISO [5] or KF [6] flange connections flat gaskets are used. Furthermore gaskets with special kinds of geometries are in use for other flange concepts. The last aspect is the vacuum tightening principle. Gaskets can be tightened under pressure or using under shear. Different vacuum qualities are defined by pressure levels, like vacuum ($<1*10^{-7}$ mbar), high vacuum (HV $<1*10^{-8}$ mbar), ultra-high vacuum (UHV $<1*10^{-10}$ mbar) and extreme UHV (XHV better $<1*10^{-10}$ mbar).

An overview of common gaskets in vacuum applications is shown the following two tables. The various types of gaskets can be classified in following groups: The first group, presented in Table 1a, is the soft on hart group. The gasket material is declared as the soft part. The flanges are even been the hart part in these combinations. In Table 1b the gasket materials are made of hard material, too.

Table 1a: Soft on Hard

Type	Material	Multiple use	Pressure level
O-Ring	Natural rubber	yes	vacuum
O-Ring	Rubber	yes	vacuum
O-Ring	PTFE	yes	vacuum
O-Ring	FKM	yes	vacuum/high
O-Ring	FFKM	yes	vacuum/high
Wire	Indium	no	high/UHV
Wire	Lead	no	high/UHV
Wire	Gold	no	UHV/XHV
Wire	Silver	no	UHV/XHV

Table 1b: Hard on Hard

Type	Material	Multiple use	Pressure level
Flat/Profile	Alu	no	UHV/XHV
Flat/Profile	Copper	no	UHV/XHV
Ring	Steel/Alu[1]	yes	UHV/XHV
Flat/Profile	SST	yes	UHV/XHV

[1] A combination with steel spring and cover ring of aluminium (HELICOFLEX®)

Theoretically gas penetration can occur from outside into the evacuated vacuum system with two different mechanisms. Gas can penetrate through the gasket material directly or can pass between surfaces of gasket and the seat of the flange.

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The sealing process is expressed by:

$$C = 1.93 * 10^4 \sqrt{\frac{T}{M} \frac{2\pi\, A^2}{\ln(\, r_0/r_i\,)\, 8.12}}\; K e^{-3\frac{P}{R}} \qquad (1)$$

- C is the conductance (of these two ways) in cm^3/sec
- T and M are temperatures in K
- r_0/r_i are outside and inside gasket radius in cm
- K is a factor of surface roughness or surface condition (scratches, dent or waviness…) [7]
- P tightening pressure in kg/cm^2
- R is the sealing factor in kg/cm^2 [7]
- Is the peak to valley value of the surface roughness

ALTERNATIV TEST PROCEDURES

As mention before deformations at the Conflat knife edges had been detected, all chambers were dismounted and were checked visually with the result of the "orange skin" on many knife edges. It was tried to seal the chambers with alternative sealing technologies, like soft copper gaskets, taper sealed (TS) gaskets (hart and soft) and at least aluminium gaskets. But no technology worked. Thus all the vacuum chambers were sent back for refinishing.

After rework of all vessels we tried to seal the chamber a second time with soft annealed copper gaskets and with hard and soft TS gaskets, but we had the same result as before reworking of chambers.

At least aluminium gaskets were chosen. Only the aluminium gasket had sealed the chamber reliably, but this technology was not accepted for the XFEL vacuum-system.

After a lot of different type of gaskets with variable hardness conditions had been tested, DESY made the decision to rebuild all the 50 vacuum chambers for E-XFEL.

HARDNESS CHECK

To find the reason for the "orange skin" one Type B chamber the hardness was checked at DNV GL [8] in Hamburg (see Fig. 3). This first test showed a discriminated value to raw material certification. The results varied between 145 and maximum 154 HB.

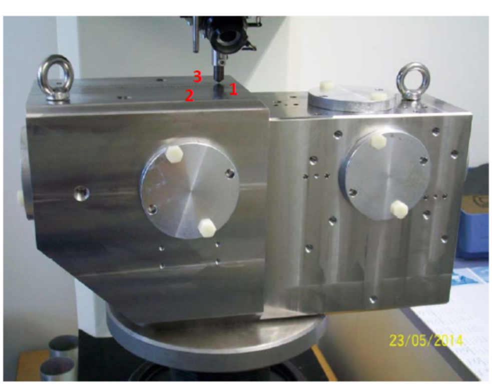

Figure 3: Show of the hardness test of one type B vacuum chamber.

In the second step all the vacuum chambers were tested by mobile hardness measuring. By measuring test parts and comparing them with mobile hardness there was only 0.5 to 1 HB difference. Due to the mobile measurements with a Krautkramer MIC 20 testing device at DESY at most chambers a smaller hardness than certified was detected.

One type A/C chamber with 164 HB had the highest value. The minimum value was 122 HB. The average of 20 chambers type A/C was 135 HB. The type B vessels didn't show different results. The minimum value was 129 HB and one chamber had a maximum of 151 HB. The average of type B was 137 HB.

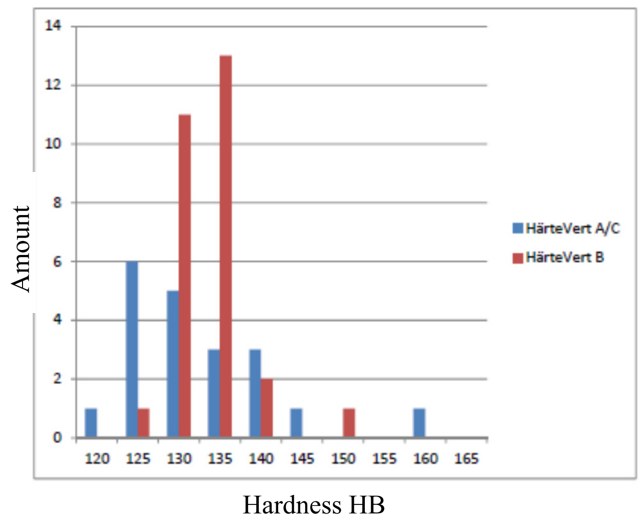

Diagram 1: Show of hardness comparison between type A/C and B.

Moreover the hardness of the raw material was checked, too. The results were in the same bounders of the chambers. Minimum value was 141 HB and maximum was 152 HB. The measurements have been done at DNV GL in Hamburg (see Diagram 1).

MATERIAL ANALYSIS AND INSPECTIONS

This chapter describes same of the test procedures done for checking other material properties. The specific parameter of non-metal parts, embattling into the raw material, determine the grain of purity, and critical grain size had been checked by using different methods and inspections. The results were compared to the material certification and did not fulfil the DESY material specification.

In a following step a part of the knife edge was cut out and was inspected by a 3D microscope. In the Figure 4 the damages of the knife edge is shown by 10 x magnifying.

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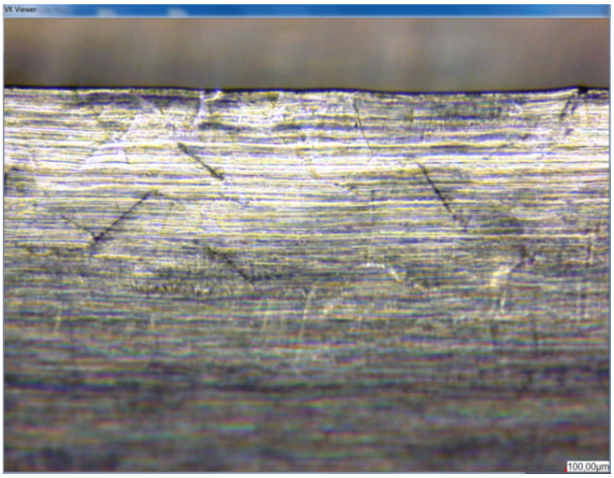

Figure 4: 3D picture of microscope inspected the knife edge of CF flange (rectangle to the 20 ° knife position).

In the Figure 4 crossing lines are burred approx. 0.3 to 0.4 mm deep into the material. The structure of expansion and appearance looks like the stainless steel material grain structure. This visual inspection shows perfectly the "orange skin" surface structure. These massive damages on the CF knife edges are serious problems and have to be understood, therefore more inspection for better understanding these problems were done.

Furthermore copper gaskets (NW 50) were inspected and tested. On the Figure 5 a cross section cut of a tested gasket is shown. The two different cuts are from two different CF knife edges. On the left side the special DESY knife edge is shown and on the right the ISO /T 3669-2 geometry is shown. The difference is the variation of the knife edge angle. DESY had defined a 72° knife edge and in ISO/TS 3669-2 a 90° angle is preferred.

These testes were made at BAM [9] in Berlin because they have adequate equipment for micro hardness soft material testing and preparing.

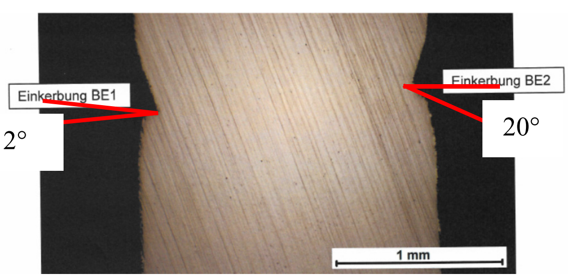

Figure 5: 3D picture of microscope inspected the knife edge of a copper gasket (rectangle to the 20 ° knife position).

In the following Figures 6 and 7 the gradients of hardness curves within the deformed areas are shown. Figure 6 relates to the ISO/TS 3669-2 knife edge angle of 90°. In Figure 7 the DESY 72° knife edge geometry is presented. The numbers present the micro hardness range on the contact zones of the flange to chamber connection with normal copper gaskets (85 HB). For the ISO/TS 3669-2 geometry the maximum value is 122 HV 0.01. But for the DESY geometry this value raised up to 148 HV 0.01.

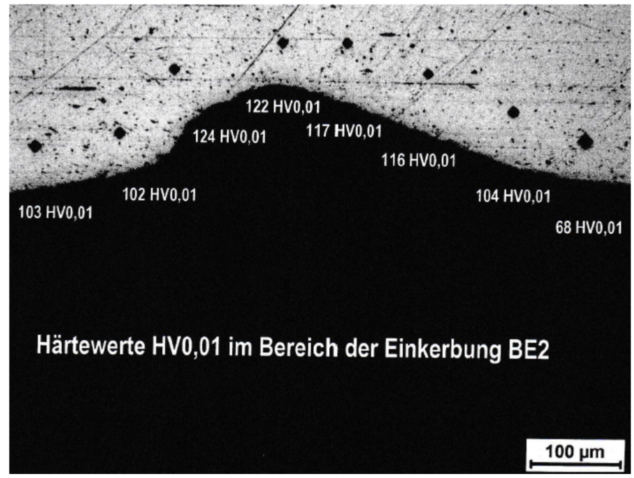

Figure 6: Polished cut image of ISO/TS 3996-2 90° knife edge.

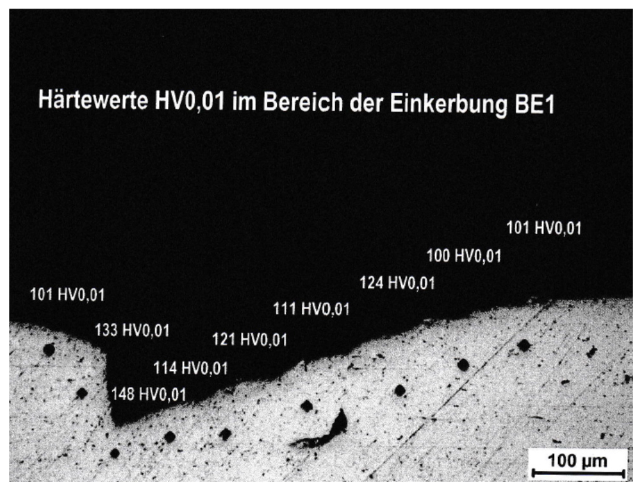

Figure 7: Polished cut image of DESY 72° knife edge contour.

The results of these two different knife edge geometries gave a big breakthrough to understand the orange skin "phenomenon", because these results compared to the measured hardness of the stainless steel blocks have printed out one critical aspect. Due to the assembling process the copper gasket material hardens. The average of the hardness type A/C and B compare to these results quite similar. Even the hardness of copper gaskets is sometimes higher than the average of the booths vessel versions.

FEM ANAYSIS

In a second step a FEM simulation [10] has to elaborate the critical areas of the two different CF flange knife edges. This FEM analysis compares two versions of knife edges geometries are used for UHV flange applications; the ISO/TS 3669-2 standard and the DESY Conflat knife edge geometry (see Figs. 8-10).

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90 ° knife edge 72 ° knife edge

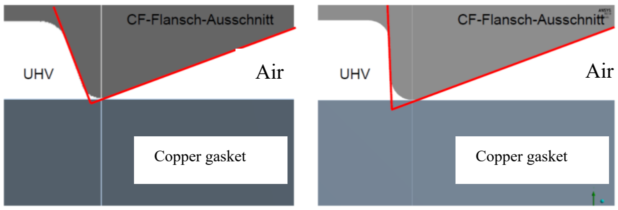

Figure 8: ISO/TS 3669-2 (left) DESY knife edge (right).

Both structures were implicated to FEM simulation process. Various FEM simulations were processed to find optimal simulation attempt. At first the implicit method quadratic formulated finite elements was used. All attempts run into solution errors caused by highly distorted elements. The second approach was to use the explicit solving method taking account of high plastic deformations during the solving process. Again, the solution runs into solver errors due to element shape warnings. The last and successful trail was to use finite elements with mathematical linear formulation, which are more robust against higher deformations. After several evaluations the FEM the program run reliably.

90 ° knife edge 72 ° knife edge

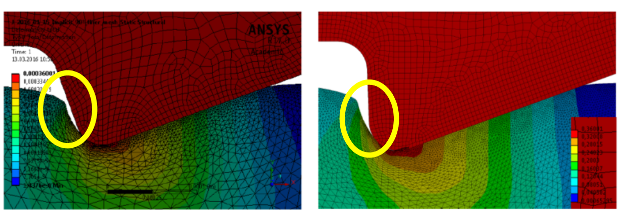

Figure 9: On the left the ISO/TS 3669-2 knife edge is presented and on the right DESY geometry is shown.

Results:

- Both geometries have maximum forces on the radius of the knife edge
- The contact areas are worked out
- A gap onto the vacuum side is shown (marks), related to direction of the knife edge drifting and deforming the gasket

The next Figures 11 and 12 show the knife edges of the flanges under pressure during assembly process. The critical zones are worked out. Differences between the two versions are illustrated. At the DESY geometry the forces on the knife edge are a little bit lower that the ISO geometry. But the steadiness of the DESY knife edge is lower.

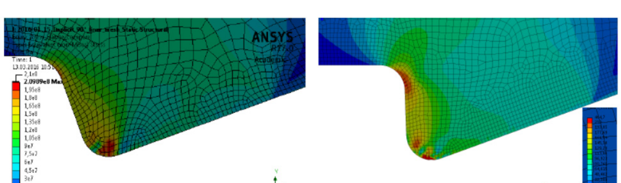

Figure 10: On these two pictures the Mises-stress of both knife edges are compared.

Results:

- Maximum on both knife edge radius form 210 N/mm²
- Bending of knife edges and friction due to flowage bending of knife edge at the ground radius, higher stress at 72 ° version

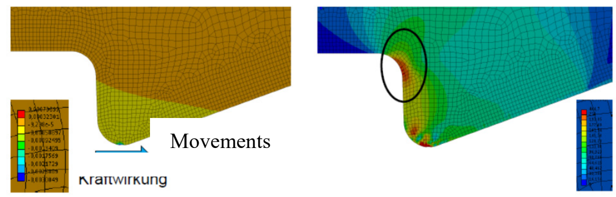

Figure 11: These pictures are shown the direction of forces and the value due to the assembling of flanges.

Results:

- Forces of friction stay against movements
- Both geometries have become forces of same direction
- Forces from vacuum to air side directions

Figure 12 documents the elongation of copper gaskets during assembling. Both copper gaskets show separate deformability behaviour. The plastic deformation to the vacuum side is 0.12 mm and to atmosphere side approx. 0.36 mm. Due to the fact that the allocations of copper material are unequal, the system moves radially to the atmosphere side.

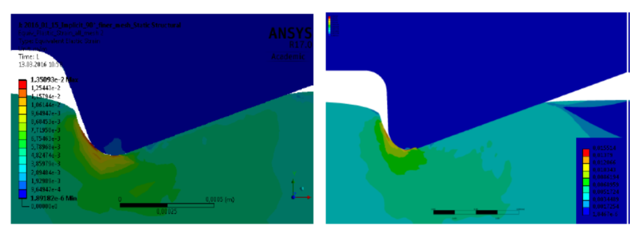

Figure 12: These pictures are shown the elongations of the gaskets in both versions.

Results:

- Selective stress load for the 72° version
- Better stress allocation for the 90° version
- Bigger elongation zone for the 90 ° version

The following Figures 13 and 14 show a comparison with equal scaling between the two geometrical variants for the CF-flanges. In general the stresses in the sealing are nearly identical whereas the stresses in the CF-flanges are different in detail. The edge of the 72°-flange has more concentrated stress-zones compared to the 90°-flange with more homogeneous stress distribution. The stress level up to 470 N/mm² is a direct result of model-problems/a singularity in the contact zone. Stresses up to a level of 320 N/mm² are feasible. The 90°-flange has more "material connection" to the rest of the flange to withstand the forces due to tighten the flange screws.

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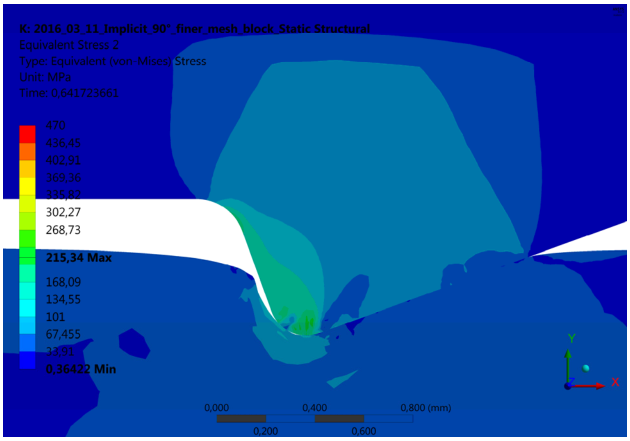

Figure 13: This picture show stress in the ISO/TS 3996-2 knife edge zone.

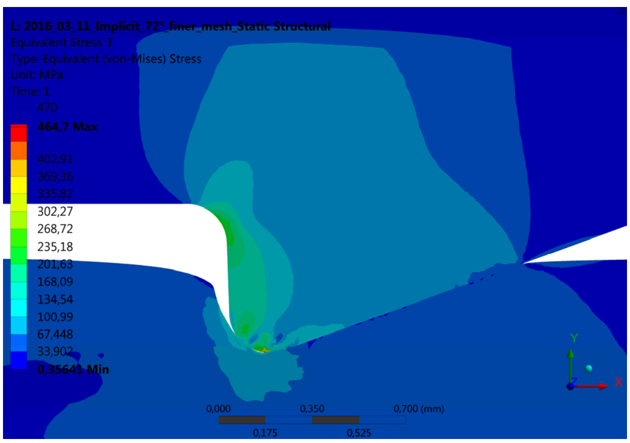

Figure 14: This picture show stress in the DESY 72 ° knife edge zone.

This paper covered only a short overview about this FEM simulation. An internal paper of these FEM simulations is under preparing at DESY [11].

RESULTS

The results are presented in this chapter. After several months ramp-up time of testing, investigations and analysis the new production went smoothly. The newly selected material had a significant higher hardness than the DESY specifications had prescribed.

The FEM simulation results gave a lot of new aspects in several iteration steps all critical forces and zones were pointed out. The FEM simulation gave excellent results for deeper understanding both common knife edge versions used at DESY.

Furthermore the deformations of the copper gaskets are not negligible. The behavior of copper during cold hardening under pressure is a serious problem especially if the minimum boundary of flange material is close to the consistency of copper gaskets after they were assembled under pressure.

All of the hardness tests, analysis and measurements have shown that the raw material certification is not enough for accepting the material. All tolerances were pointed out and had to be integrated in specification for ordering raw materials.

The sealing areas of knife edges receive a lot of power transmission and radial forces. These forces will be never noticed until micro damages on sealing area on the surfaces of flanges happen.

The advantages of Conflat vacuum connections to be used in multiple forms and they can be baked out. The range of temperature limits depends on gasket materials and coated surface of gaskets as reported by Danielson [12].

The disadvantage of Conflat flange connections is that they need much more attention and special care during manufacturing and assembly processes. In order to obtain reliable flange connections, the sealed diameter of the mating knife edges must be absolutely equal to avoid the umbrella effect or staggering of gasket, as reported by Unterlerchner [13]. In the following Figure 15 the umbrella effect is documented. This negative effect produces untitled vacuum leaks.


Figure 15: This picture show a gasket can be twist during assembling.

For customer designed Conflat flange applications a few measures and tolerances have to be attended. The knife edge diameter should better within 0.1 mm, the knife edge radius not bigger than 0.2 mm and the eccentricity should be held be within 0.1 mm related to the flange middle axis. The recess for the gasket should be tolerated to within 0.05 mm related to middle axis of the flanges. The sealed surfaces must not have any marks, scratches or dumps. And the sharp tolerance of sealing surfaces should be within 0.05 mm. And finally the knife edge angles should not be smaller than 90 ° (leaned to ISO/TS 3669-2 standard).

In Figure 16 the risk of getting virtual leaks is pointed out. The pairing flanges are pressed to zero gap. These typical connections are required for XFEL warm beamline connections, because of reduced bellows connections and small gaps between the Conflat flanges.

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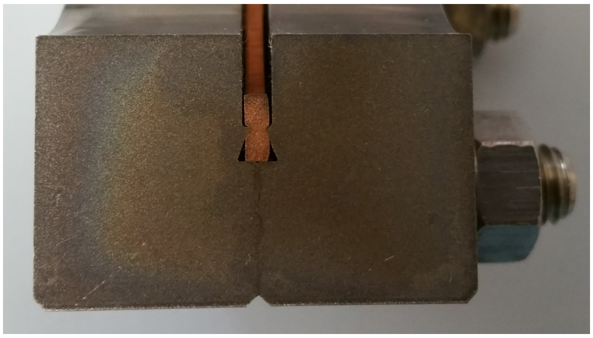

Figure 16: This picture presents a gasket connection between two Conflat flanges with different knife edges.

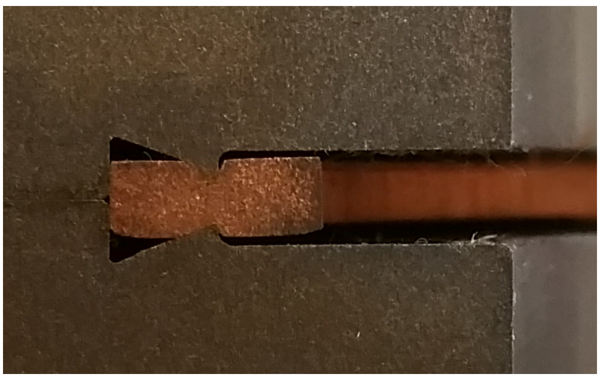

Figure 17: This picture is presenting the pairing of DESY knife edge with IST/TS 3669-2 knife edge.

Figure 17 the different knife edge geometries are presented. This picture represents very clearly the problem during the assembling process, if two different knife angles cut copper gaskets. The gasket tries to twist and the risk to get vacuum leaks increases.

Many gaskets have been badly. They are produced using vibratory grinding and have bad surface roughness. Final processes like etching or polishing are used. In that case gaskets may have micro cracks, shrink holes or the copper grain structure obtrudes. When these gaskets are used for high end applications of UHV, XHV or under cleanroom application leaks are more likely.

A few technologies are existing for bringing more hardness into the surface of high stainless steel alloys, but the penetration of these skin procedures are not deep enough for getting excellent achievements. One process is called Kolsterising® [14] and another is called "Kalt-Nitrieren" [15]. This paper does not cover more details about these two technologies but recent tests at DESY show no reliable improvements [16].

The implementation of quality management for bigger projects or serial production can be controlled more easily. The risk of failure materials gets lower and you will get more reliability.

CONCLUSIONS

The proper material for manufacturing massive vacuum chambers has to be well defined and very painstakingly and carefully inspected before starting serial productions.

The variations of mechanical specifications given in material certifications can be very large. Typical material certifications are made before the raw materials are forged, deformed and annealed. A lot of material specifications are focused on tensile and yield strength, but the hardness of this material mustn`t be ignored. Hardness is a significant property of reliable vacuum tightness, and also for reusing vacuum flange connections. If the hardness of the copper gaskets is under pressure in the order of the stainless steel hardness boundary, serious vacuum problems may appear.

The knife edge with the knife radius is one critical aspect for getting better UHV vacuum connections. However the main important area is the 20 ° surface area, between the knife edge and the shoulder fit of gasket with is terminated vacuum against air. Rework of damage components are very time and costs expensive. FEM analysis gave a better understanding material stresses and forces of several gasket knife edges.

LESSONS LEARNED

Based on the bad experience within this project DESY produced these vacuum chambers a second time. Very big efforts are necessary in order to build new chambers for XFEL within a short time. Not to run into time problems for the XFEL schedule these efforts are necessary. Based on the first design new massive chambers were fabricated and installed in the tunnel.

One lesson learned was to say good bye to constructions of massive blocks like these two designs with integrated Conflat flanges. Running into serious problems in case of having to rework these chambers is one aspect for using vacuum chambers made of standard Conflat flanges with normal standard SST pipes.

For new constructions of diagnostic vacuum chambers, if it cannot be avoided to use massive blocks, the selection of raw material has to be well defined and precisely observed. A lot of specifications are not up to date and should be matched to new technologies or projects. New applications like particle clean environments are the new leading technologies for new accelerator designs. Particle clean components have to be assembled under restricted specifications and the requirements to these parts are much more complicated than in the past. Limited to new conditions like higher cleaned components in high precisions, low residual magnetism and high purity, the specifications for raw materials have to be adapted.

Moreover a lot of different suppliers and producers for Conflat flanges and copper gaskets are on the market, but are they handle out precis specifications to their products?

This worst case event capable to showed that general specifications for vacuum components may often not be precise enough for high technology applications. Many years of experience is necessary, but this cannot be enough. The following steps may help to avoid these bad experiences.

- First clarify, concentrate and be precise about all requirements to the design (incl. all environment conditions, to be bake out, movements, cleanness requirements…)

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- Clarify and require the raw material specifications (find out what is described in your organization …)
- Use standard norms, references or guidelines
- Earliest decision "Make" or "Buy"!
- One big point! Use pipes and standard Conflat flanges if you can!
- Use lessons learned from internal papers, or constructions, if you will get them
- Make a requirement matrix and estimate the technical functions, write out the results
- Before ordering raw material, check very painstakingly and carefully the material properties which will be documented in the 3.1 material raw certification!
- This 3.1 certification should be related to your delivered batch!
- Hardness for secure and reliable UHV or higher vacuum applications should be over 170 HB, better up to 185 HB (results of many different tests)
- N better 0,14 %, Cr not over 17,5 % and Ni more than 12 % (results of many different tests)
- ESR = Electro-Slag-Remelting, forged multidirectional and annealed is mandatory, full austenitic structure
- Early integration of material producers or vendors, manufactures and purchase office
- If you make your own design, build more than one prototype for serial productions.
- According to ASTM E112-13 the grain size shall be better than 3, but for machinability not better than 8!
- Independent material tests of the raw material should be done after delivering the material
- For bigger serial productions make sure that you will have adequate technical descriptions which will cover all the requirements of your scopes.
- Make Quality Audits with suitable vendors or manufactures
- Make a risk analysis and if you can use project management competences
- GOOD LUCK!

to avoid pictures like the following Fig. 18...

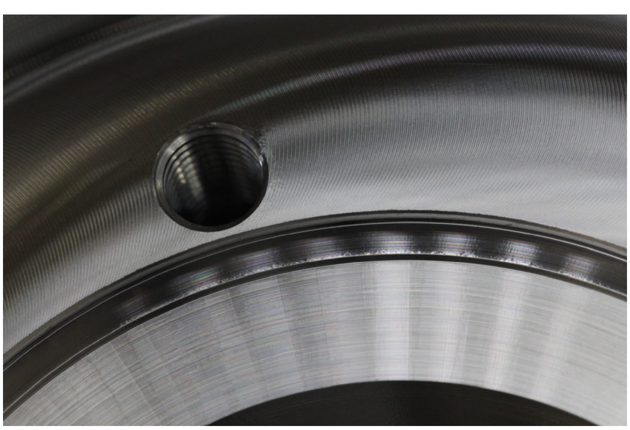

Figure 18: Last picture documents the orange skin micro damages on the scintillation screen monitor vacuum chambers for E-XFEL project.

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