TIE-45: ZERODUR® adhesive bonding recommendations

0. Introduction and Disclaimer

Adhesive bonding of ZERODUR® to either ZERODUR® or low expansion thermal metal alloys like Invar® is a process used by many of our customers. Especially ZERODUR® mirrors in astronomical telescopes are bonded to supporting structures via invar pads using adhesive bonding techniques. Nevertheless, to generate adhesive bonds with long lifetimes, it is important to know what influences the achievable adhesive strength and fracture toughness of the material. This technical information is intended to give some technical recommendations on surface preparation, design, cleaning and bonding from the ZERODUR® material point of view. General information about ZERODUR® processing can be found in [1].

Disclaimer:
This technical information serves only as recommendation guideline since the longevity of bonds depend on the very special case out of a huge possible variety and on practical craftsmanship. No liability can be given for any case.

1. Preparation of ZERODUR® bonding surface

- ZERODUR® surfaces shall be ground using grinding tools consisting of a metal carrier with bonded diamond grains of a defined size distribution.
- Typically tools used for processing are of type D151 (100/120 acc. ASTM) with a maximum grain size of 150 µm and D64 (230/270 acc. ASTM) with a maximum grain size of 64 µm.
- The surface shall be ground homogeneously flat without any steps, burrs, grooves or deep scratches that could act as stress concentrators (the surface should not be processed by cutting only).
- Depending on the viscosity of the adhesive the flatness or shape deviation tolerance of the bonding partners should be in the range 20 µm or 50µm.
- To reduce the microcrack depth, the final grinding step shall remove at least a material thickness larger than the grain size of the preceding grinding step. In workshop practice a layer of three to four times the grain size dimensions of the preceding step is removed.
- The adhesive strength depends on the total surface available on the bonding area, therefore a ground surface leads in general to a higher adhesive strength compared to a polished surface.
- D151 and finer ground surfaces of ZERODUR® have bending strength significantly higher than 10 MPa (see reference [2] and [3]). Even higher strengths can be reached by acid etching or optical grade lapping and polishing with intermediate subsurface damage removal. These processes remove residual microcracks. However the roughness should be kept at a certain minimum level to improve the adhesive strength.

- Acid etching is done with e.g. a mixture of HF(38%) + HCl(37%) + H_2O in a ratio of 2:1:1,5 to a depth at least the size of the last grinding tool grain size. For D64 the minimum material removal shall be 65 to 100 µm [4].

2. ZERODUR® surface strength in general

- The strength of glass and glass-ceramics is not a material property like the Young's modulus e.g. It is dependent on
  - the microcrack depth below the surface which is tension stressed by the load applied,
  - the area of the surface exposed to tensile stress,
  - the rate of stress increase and
  - the environmental media.
- A piece of glass breaks when two conditions coincide. The first is the presence of tensile stress at the surface and the second is the presence of a flaw with a minimum depth, the size of which depends on the tensile stress at the location of the flaw.
- Milling and grinding of glass introduces microcracks to the surfaces.
- Empirically one found with ZERODUR® that the microcrack or subsurface damage depth is similar to the maximum grain size of the sieve fraction or somewhat smaller for larger grain sizes above ~100µm.
- Decreasing grains sizes result in less deep microcracks and in higher characteristic strength values (given at 63% fracture probability).
- At fracture probabilities of 10% and less the strength values of surfaces fabricated with D64 and D151 tools do not show a significant difference [3].
- As an example it was shown that a homogeneous isotropic load of 41 MPa (stress increase rate 2 MPa/s) on a 1 m^2 ZERODUR® surface (without any pre-damage), ground with bound diamond grain tool D151, leads to a failure probability of less then 0.001 (0,1%) 95 % confidence level range. This is a stress amount that is 4 times higher than the conservative rule of 10 MPa at a moderate surface treatment. [2, 3].
- Etching or optical polishing of ground surfaces significantly improves the characteristic strength of the glass by eliminating the microcracks. The grain size of the preceding grinding process does not play any decisive role anymore, when the etched off layer thickness is significantly higher than the subsurface crack depths. Etching broadens the strength probability distribution thus leading to a smaller slope in the cumulative failure probability plot [3].
- The above given statements are only valid if the surfaces are not damaged by handling etc.

3. Geometry / shape of the ZERODUR® bonding area

- Due to the fact that ZERODUR® is a brittle material in comparison to metal, the main design target should be to minimize tensile load concentration areas in the design.
- The load of application forces in general has to be distributed over a larger area. Point like and line-like stresses should be avoided because they might lead to fracture or chips depending on the height of the load.
- Tensile stresses acting only on one side of the bonding area ("opening stress modes") shall be avoided in construction as far as possible. Shear mode stresses on the bond are less critical.
- The ideal shape of the bonding area is circular. If circular bonding areas are not possible, the rectangular ZERODUR® bonding area and also the counterpart pad to be bonded shall have a generously rounded edge geometry (e.g. R2 according to ISO 2768)

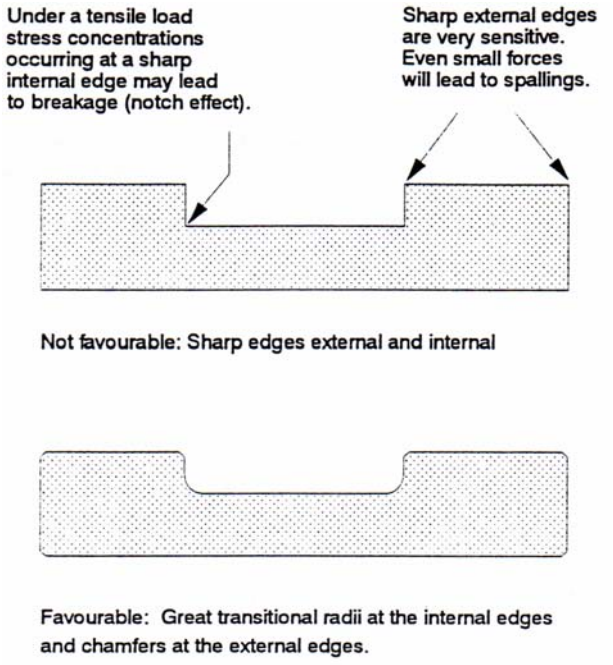

Figure 1: Favorable edge design of ZERODUR® parts [1].

- Within pockets notches and sharp internal edges have to be avoided by assigning largest possible transition radii radii (e.g. ~R2 according to ISO 2768 at bottom edges and even larger radii at inner quadratic structures) between inner bottom and wall edges.
- Remaining edges in general should be furnished with small chamfers. Preferably obtuse angles should be assigned to external profiles.
- The counterpart pad to be bonded shall be chamfered at the edges to prevent stress concentration.

4. Cleaning of ZERODUR® bonding area

- In general a small amount of particles on the surface of the bonding areas is not critical. It is in many cases not necessary to perform the cleaning and bonding in a clean room environment. More critical are surface layers of oil and grease or other organic or inorganic compounds.
- Oil and grease can be removed with alkaline cleaners containing tensides by application of an ultrasonic bath or mechanically with a soft brush. Afterwards the parts have to be rinsed with water, finally with deionised water.
- More resistant mineral oils can be removed using acetone or ethylmethylketone (MEK) in high purity (analytical grade).
- It is very important to prevent a surface pollution by silicone. Silicone can only be removed by using additional plasma cleaning.
- After cleaning the surfaces may be dried with ionized oil-free nitrogen or air.

5. Adhesive selection and bonding of ZERODUR®

- The adhesive shall be selected carefully based on the application temperature and humidity conditions. Individual testing of the adhesive is recommended if it is used under extreme environmental conditions like cryogenic temperatures or under vacuum.
- It is important that the adhesive does not change its properties or becomes brittle over the lifetime of the application under the given environmental conditions.
- ZERODUR® has only a very low UV transmittance, therefore UV curing adhesives might not work.
- Curing temperatures should not exceed 130°C if minor to prevent CTE changes in ZERODUR® have to be avoided.
- The coefficient of thermal expansion of the adhesive shall be as low as possible.
- Depending on the application the adhesive type will be selected to be elastic after curing to level thermal expansion differences between bonding partners or to be stiff after curing to achieve a maximum position accuracy.
- The adhesive shall shrink as little as possible during curing. A good indication of the shrinkage behavior is the comparison of density before and after curing in the datasheet. The differences shall be as low as possible.
- For bonding ZERODUR® to ZERODUR® the adhesive layer should be as thin as possible to minimize the stress due to shrinkage of the bond.
- ZERODUR® bonded to a material with a higher coefficient of thermal expansion, requires an adhesive with a higher elasticity and the bonding layer shall be thicker to balance the thermal stress between the bonding partners.
- The viscosity of the adhesive shall be selected to fill the gap between the bonding partners due to the shape/flatness tolerance. For a large gap a high viscosity adhesive is needed.
- The adhesive shall be applied as homogenous as possible. A wedged adhesive layer shall be prevented.

- The adhesive shall be applied in such a way that it does not squeeze out of the bonding area. Any adhesive squeezed out between the bonding surfaces might lead to high stresses at the edges during curing (see figure 2).

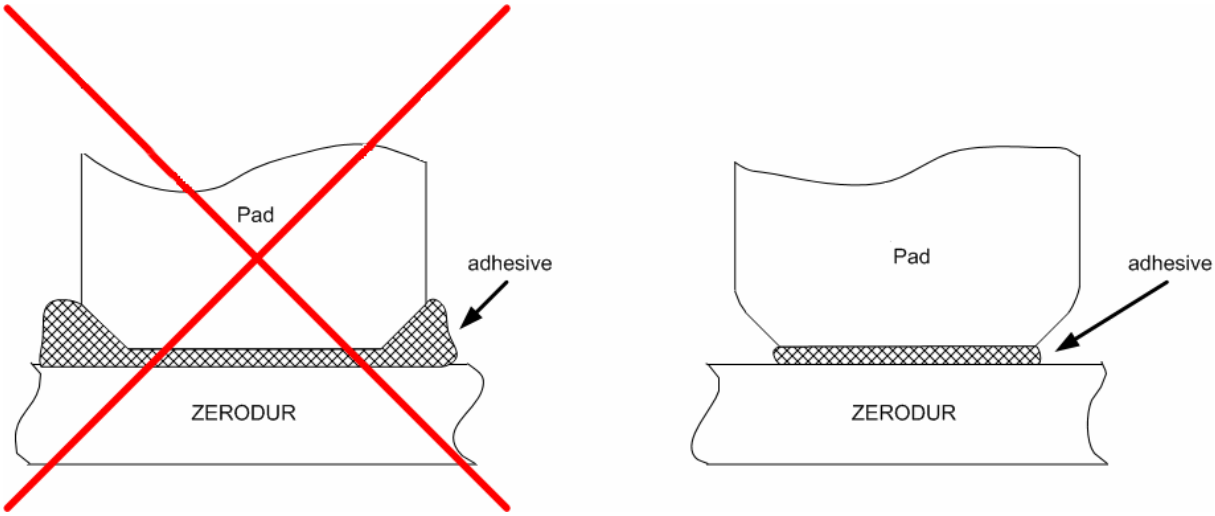

Figure 2: Adhesive application: Squeezing of adhesive out of the bonding area should be avoided (left picture). Ideally only the actual bond areas are covered with adhesive.

6. Literature

[1] SCHOTT Technical Information TIE-44: Processing of ZERODUR®

[2] Peter Hartmann, Kurt Nattermann, et. all; Strength aspects for the design of ZERODUR® glass ceramic structures; Proc. SPIE Vol 6666 Optical Materials and Structures Technologies III; William A. Goodman, Joseph L. Robichaud, Ed.; 2007

[3] Peter Hartmann, Kurt Nattermann, et. All; ZERODUR® glass ceramics- strength data for the design of structures with high mechanical stresses; Proc. SPIE Advanced Optical and Mechanical Technologies in Telescope and Instrumentation; 2008

[4] Ralf Jedamzik, ZERODUR® acid etching process in general, SCHOTT Process description 2007.

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