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Unknown properties of aluminum nano-layer in unglue assemblage of ZERODUR parts

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Abstract: In the course of technological operations and the following running, the aluminum nanolayer providing reliable unglue bonding the ZERODUR parts demonstrates a complex of previously unknown properties: - compensation of small geometrical deviations in joined parts, due to aluminum plasticity the requirements to geometry of joined parts can be lowered from $N = 0.5$, $\Delta N = 0.1$ (where N is the deviation of surface geometry from standard expressed in the Newton interferential rings, ΔN – local error or deviation of the type "well" or "nib"), which are necessary for joining, if one applies the methods of optical contact or deep optical contact, to $N = 2\text{-}5$, $\Delta N = 1$; - Chemical interaction with polished ZERODUR surface at relatively low temperatures (400 to 600ºC); - Minimum level of internal mechanical strains in joined parts; - Properties of elastically-brittle durable material. Using the above generalization of the obtained results, we have offered new technological schemes for production of ZERODUR parts with a complex shape.

Keywords: Aluminum Nano-Layer, Unglue Bonding, ZERODUR

1. Introduction

ZERODUR manufactured by the Schott Corporation is glass ceramic widely used in different branches of industry and science. Due to its unique properties, it is widely used in manufacturing main mirrors of telescopes, interferometer parts, laser gyroscopes, etc. It has decisive advantages for application in modern LCD, lithography and production of various precise optical devices.

Among the most important advantages of this material are its zero thermal expansion, good processability and very low surface roughness. From the chemical viewpoint, ZERODUR consists of oxides Li2O–SiO2–Al2O3.

Technology for preparation of this material lies in melting the charge inside a glass-founding oven at a temperature of 1600°C. After homogenization, this fusion is discharged into a special mould, and after cooling it, homogeneity of the prepared glass is tested. Then, the glass is repeatedly heated up to the temperatures of 900 - 1000ºC for crystallization. In order to stimulate the formation of the nucleation center, titanium oxide is added to the starting charge. This ceramics has both crystalline (80-percent lithium aluminum silicate crystal and 20-percent high silica amorphous glass phases. After exposure within the above temperature range, the obtained blank is cooled.

However, this material has its temperature limits in technological processing and application: it changes its properties irreversibly at temperatures above 600ºC, which is caused by changes in crystallite sizes and relationship between crystalline and amorphous phases. It results in enhancing the value of its thermal expansion coefficient. So, technologies for processing any parts made of ZERODUR as well as methods for joining them are limited by this temperature factor. Therefore, for instance, annealing the precise mirrors from ZERODUR for laser gyroscopes to improve the state of their surface is performed at a temperature of 450°C [1].

At the same time, it is known that to ensure high durability and reliability for diffusion bonding the parts from glass, crystals and glass=crystal materials, the temperature factor is decisive. To solve the problem of joining ZERODUR, the following technologies were developed:

- optical and deep optical contact;
- special chemical processing of the combined surfaces aimed at improving quality of deep optical contact [2, 3].

A common deficiency of the known technologies is the necessity to precisely process the combined surfaces that limits the maximum dimensions of joined parts and increases considerably expenses of their production. As usual, the diameter of ZERODUR parts joined in accordance with this technology is close to 30–50 mm, and the maximum one is 250 mm.

Difficulties in joining the large-scale parts (2 to 4 m in diameter) from ZERODUR do not allow manufacturing aided mirrors of a sandwich type with the internal honeycomb layer for telescopes, which could provide a record degree of their aiding (up to 92-95%).

Besides, as we ascertained, chemical processing of the combined surfaces, although it enables the enhancement of durability of the joint, has a glaring defect – parts should be joined immediately after this processing. When storing, chemically processed layers lose their advantages, and joining of the details becomes more complex.

Earlier, we obtained the result that preliminary deposition of an aluminum layer onto the polished surface of one of ZERODUR parts enables to join them at temperatures from 400 to 600°C [4]. Stability of this joint was brought nearer to the bulk ZERODUR one [5-7]. This result causes surprise since the parts in this case are connected by the plastic aluminum layer that does not possess high durability. The aim of this work was to sum up the results of research on the unknown properties of the aluminum nanolayer, which occur in unglue joining of the ZERODUR parts.

2. Results of Experiments and Discussion

Placed in a vacuum, the nano-dimensional aluminum layer around 100 nm in thickness can make up for geometrical deviations on the surface of processed parts due to its high plasticity and, at the same time, can ensure reliable joining of these ZERODUR parts. Our experiments proved that, owing to aluminum plasticity, requirements for geometry of joined surfaces, which are necessary for joining by using the methods of optical contact or deep optical contact, can be reduced from N = 0.5, ΔN = 0.1 (where N is the deviation of surface geometry from a standard expressed in the Newton interferential rings, and ΔN is a local deviation – small well or projection) to N =2-5, ΔN = 1. Making up the mirror 90º prisms for external reflection in accordance with the scheme (Fig. 1) confirmed that technological precision of angular parameters of joined parts is kept in assembled (using the aluminum nanolayer) prisms within 10 angular seconds (and the best sample had the error of only 1 angular second, which coincided with the experimental error). It means that the nano-dimensional layer of aluminum enables technologically the requisite precision even in serial production.

The bonding aluminum layer has high optical reflection properties. We confirmed the availability of free electrons in this layer, using the method of surface plasmon resonance [8]. Thus, our investigations showed that the aluminum nanolayer used for joining the parts from ZERODUR can be also employed as the optically reflecting and electrically conducting layer, which can be very useful in developing new construction solutions.

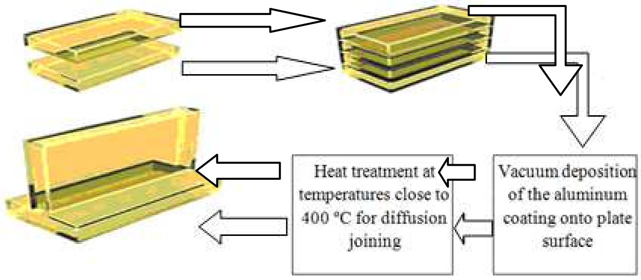


Figure1. *Technology of manufacturing the mirror prisms of external reflection from ZERODUR of collecting construction*

Internal mechanical strains in joined parts had only small values (of the order of 0.1 MPa) that is very important for keeping prism precision (constancy of the angle 90º) under extreme conditions (thermal shock when transferring from room temperature to that of liquid nitrogen, and mechanical shock at the level of 200 g) as well as long storage. Under the latter condition, the precision of mutual positions of parts was even improved to some extent (Fig. 2), which can be probably explained by a micro-creep phenomenon [9].

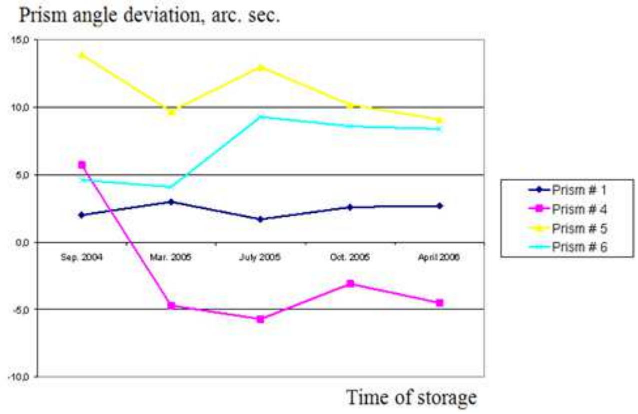


Figure2. *Influence of storage after thermal and mechanical shocks on deviations at an angle of 90° for mirror prisms of external reflection of collecting construction. The prisms were made from plane polished ZERODUR plates and joined with the help of the developed technology of diffusion joining via aluminum nanolayer [6].*

At the same time, short-term mechanical tests for a drift within the range of elasticity (mechanical stresses were put along the joining aluminum joint) showed that joined samples are ideally elastic material within the whole range up to its destruction.

And so, the following question arose: why does such plastic material as aluminum in a joining nanolayer behave like the durable and brittle one?

The results of investigations [10] by means of electron microscopy (Fig. 3) provided the following explanation.

Dislocations inside the aluminum nanolayer close upon 100 nm in length are oriented along the normal to the joined planes of flat parts.

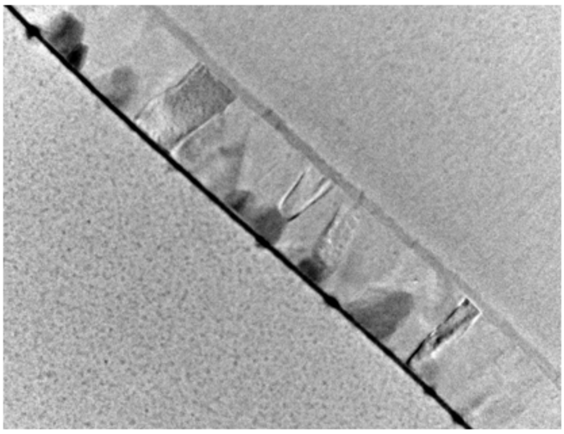


Figure3. *Topology of the transverse section of the aluminum nanolayer in the diffusion joint of ZERODUR parts [10].*

The interface between the part surface and the aluminum layer is characterized by the creation of an additional layer 10 to 15 nm in thickness. This layer consists of aluminum oxide and allows to chemically fix the above dislocations that prevents their motion along the interface till the moment when applied stresses are above the destruction threshold. It follows therefrom that the dislocations can only hog elastically. Therefore, the aluminum layer behaves like an ideally elastic material till its destruction. The enhanced durability of the aluminum nanolayer can be also explained by its structure (Fig. 3): its grain size is comparable with the layer thickness, and in accordance with the known relation

$$\sigma_f = \sigma_o + \kappa d^{-1/2}$$

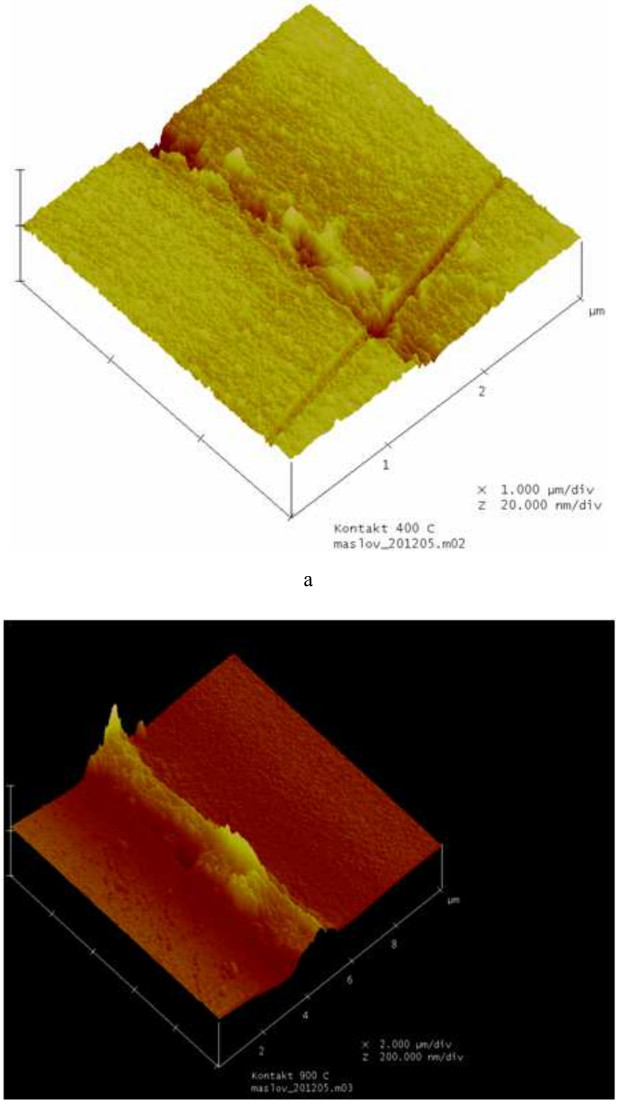
where σ_o is the destructing stress for a single crystal;

κ – some constant;

d – length of the dislocation gliding plane.

The validity of this formula was confirmed many times during theoretical and experimental research on metal parts. One can see the review of respective works in [11]. In this case, the authors of these works used the grain size in the structure of metal as the parameter *d*. Disintegration of grains through thermal and mechanical processing enables a considerable increase in the level of destructive stresses. In the case of offered technology, this approach allows to use the thickness of joining aluminum nanolayer (several hundred nanometers) as the *d* parameter. Therefore, durability of such nanolayer in a diffusion joint of ZERODUR parts can exceed durability of bulk aluminum parts by 20 to 100 times since the grain sizes in the latter can reach several micrometers for thermo-mechanically (rolling, extrusion) processed parts up to several millimeters for the molten ones.

Also, it is interesting to study the influence of mechanical processing (diamond cutting, polish, burnish) the parts joined via this aluminum nanolayer. After finishing, we analyzed the surface by using atomic force microscopy (Fig. 4). Under elastic stresses, the joining aluminum nanolayer is located below polished ZERODUR parts (Fig. 4a). However, after additional oxidation of this part, due to an increase in the volume of the layer (aluminum transforms into its oxide) near the section surface, we observed the creation of nano-nibs (Fig. 4b). This processing brings about simultaneous changes in ZERODUR properties, as it was noted in the Introduction.



a

b

Figure4. *Connecting joint (a) after additional thermal processing at 900 ºC for 1 hour (b).*

Experiments helped to join parts made of amorphous glass phase and, after repeated heating up to 900-1000ºC, provide partial crystallization of glass bulk with simultaneous dissolution of the aluminum nanolayer, which causes its partial transformation into ceramic nanolayers. This scheme enables production of blanks with a complex shape from glass-crystalline materials.

The generalization of experiments allowed to discover a new complex of properties of the aluminum nanolayer as well as to use the vacuum-placed aluminum 100 nm in thickness for reliable joining the precise ZERODUR parts. Given that in this case traditional equipment and technologies of optical production can be used, there is a real possibility of creating large light-weight mirrors of a sandwich type, for example.

To confirm this possibility, we made the prototype of such a mirror 500 mm in diameter from ZERODUR in lab conditions.

In addition, the developed technology provides for a new scheme to produce complex-shaped jobs from ZERODUR: after the first stage of glass preparation, this glass should be processed to obtain parts of required shapes, then these parts should be joined with each other using the aluminum nanolayer at a temperature of 400–600ºC, and the combined structure should be repeatedly heated to the crystallization temperature of 900-1000ºC. After cooling this structure, valuable optical surfaces should be polished and finished.

3. Conclusions

Thus, in the course of technological operations and the following running, the aluminum nanolayer provided reliable bonding of the ZERODUR parts and thereby demonstrated a complex of the previously unknown properties:- compensation of small geometrical deviations in joined parts, owing to aluminum plasticity the requirements for geometry of joined parts can be lowered from $N = 0.5$, $\Delta N = 0.1$ (where N is the deviation of surface geometry from standard expressed in the Newton interferential rings, ΔN – local error or deviation of the type “well” or “nib”), which are necessary for joining, if one applies the methods of optical contact or deep optical contact, to $N = 2\text{-}5$, $\Delta N = 1$;- chemical interaction with a polished ZERODUR surface at relatively low temperatures (400 to 600ºC);- minimum level of internal mechanical strains in joined parts;- properties of elastically-brittle durable material.

Using the above generalization, we have offered new technological schemes to produce complex-shaped ZERODUR parts.

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