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Experimental Stress Analysis

Tensile stress of thin ceramic layers measured by Fizeau interferometry

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Abstract: We introduce a method to measure stress of thin ceramic slurry layers, while they are drying on a substrate with known parameters. The method exploits the bending of the substrate, which is measured bye Fizeau interferometry.

Keywords: Tensile stress, Thin coating, Fizeau interferometry

1. Introduction

Ceramic material is widely used presently, because of its low weight, very low electrical and thermal conductivity and high tenacity. But there is specific risk in the manufacturing process of large ceramic objects: during drying, there is high tensile stress in the ceramic slurry, and cracks can occur. So the drying process is critically and has to be controlled. We introduce a method to measure this stress.

2. Corcoran equation

The field of engineering mechanics developed solutions for the stress distribution and deformation of thin plates. Especially Timoshenko [1] has shown solutions for many different geometries and loadings. In case of a homogeneous coating the induced stresses are planar isotropic. Therefore, the resulting bending moments M are also isotropic. For this specific case, Timoshenko has shown that a thin flat plate shows a spherical bending with bending radius R.

In case of a thin coating (compared to the plate thickness), the neutral axis is in the middle of the plate. The point of application of the applied force due to the stress in the layer can also be assumed to be in the middle of the layer because it is very thin. Under these

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presumptions, an equation for the stress in the coating is obtained, called the Corcoran-equation [2].

$$\sigma = \frac{E_s t_s^3}{6(1-\nu_s)Rt_f^2[1+\frac{t_s}{t_f}]} + \frac{E_f(t_s+t_f)}{2R(1-\nu_f)}, \quad (1)$$

where σ is tension, E_s is Young's modulus of substrate, E_f is Young's modulus of ceramic layer, t_s is thickness of substrate, t_f is thickness of ceramic layer, R is bending, ν_s is Poisson ration of substrate and ν_f is Poisson ration of ceramic layer.

Using the Corcoran-equation allows to calculate the stress in the coating when knowing the material parameters and the bending radius of the substrate. The first term of the Corcoran-equation describes the actual stress in the coating during the experiment. It only depends on substrate parameters except of the coating thickness. But due to the bending of the substrate plate the stress would be higher without this relaxation. The second term describes the amount of this relaxation. It only depends on coating parameters except of the substrate thickness.

Because of the Young's modulus of coating much lower than of substrate, and because the coating is thinner than the substrate, the second term of Corcoran equation is negligible.

3. Fizeau interferometry

We measure the small bending (typically about 6 μm) of the smooth substrate by a Fizeau interferometer (Fig. 1). The partially coherent light from the broadband light source is collimated and propagates from the bottom (Fig. 1) through the carrier glass which is shaped as a wedge. One part is reflected from the top flat side of the carrier glass wedge, the second part is reflected from the surface of the bended substrate then propagates back through the wedge. If the difference of these optical paths is smaller than coherence length of light source, we observe by the interference fringes as a camera image. Shape and number of fringes directly corresponds to the optical path difference between the carrier glass and the substrate.

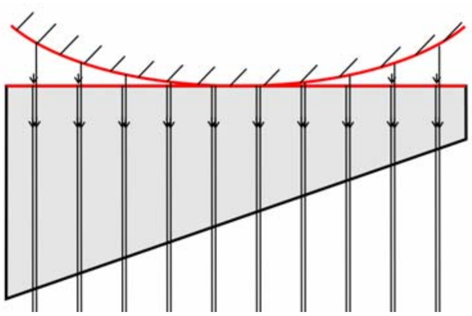

Fig. 1. Principle of Fizeau interferometry

While the ceramic layer is drying, the interference fringes are moving according to the substrate bending. The shape of the substrate is changing from a flat to a curved surface (sphere, respectively only a section of a sphere). When the ceramic layer is dry, interference fringes stop their movement and the measurement is finished.

4. Experimental procedure

A standard semiconductor laser diode is used as a light source. In the mode of stimulated emission (laser mode), the coherence length is too long and we observe additional interference fringes created inside the substrate. Therefore, the laser diode is tuned and stabilized in spontaneous emission mode, where the spectrum is broader and the coherence length is shorter. The coherence length must be shorter than the thickness of used substrate. It is important that the coherence length defines the measurement range of the bending. In our experiments, the coherence length is not longer than 30 µm. We use standard CCD camera with the resolution 1024x768 pixels and 25 Hz frame rate. The experimental setup is shown in Fig. 2.

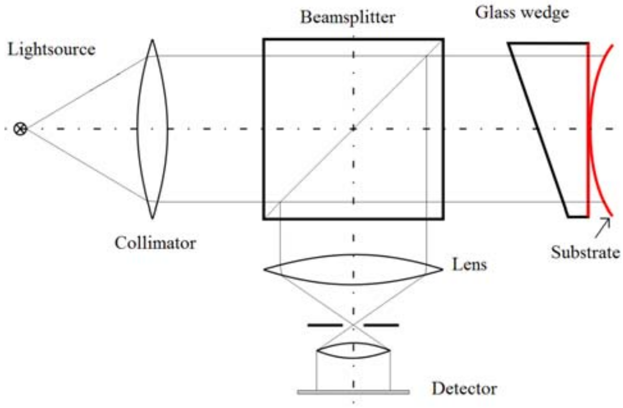

Fig. 2. Experimental setup of Fizeau interferometer

The ceramic slurry used in this experiment is model slurry, only composed of ceramic powder, dispersing agent and distilled water. The powder is ground aluminum with 1.3 µm particle size (CT 12000 SG, Almatis, Germany). The solid content of the slurry is 30 weight t%. The dispersing agent (Darvan C-N, R.T. Vanderbilt Company Inc., USA) is added (2 wt%) to prevent agglomeration.

A very homogeneous coating thickness is needed. It is difficult to achieve a complete and homogeneous coverage of the substrate, at the same time preventing slurry from flowing over the edge of the substrate. This is crucial because the small air gap between the optical flat and the substrate otherwise acts as a capillary and sucks the slurry under the substrate thereby inhibiting any measurement. The solution is coating with a scalpel and a stencil. The stencil impedes slurry from being spilled and also determines the coating thickness, whereas the scalpel guarantees a homogeneous coating. The samples are placed on the glass wedge directly after the coating. Then the measurement is started immediately. The interference fringes (ring-shaped) are observed by the camera and stored in the computer (Fig. 3).

The drying stress is calculated according to Corcoran's equation. Only the first term is used for the calculation. This describes the actual stress in the coating. The stress without bending of the substrate should include the second term. But the second term is negligible, as mentioned above.

5. Results

The evolution of the drying stress during the measurement is shown in Fig. 4. As expected, there are no significant stresses at the beginning of the drying process. This is in accordance to the expectations. First, an excess of solvent is present in the ceramic slurry. With ongoing drying the excess is decreasing. As soon as the particles in the slurry are getting so close that they touch each other a network forms, the remaining solvent has to recede more and more into the pores. This gives rise to capillary forces which cause tensile stresses in the coating.

This can be seen from the rising stress in Fig. 4 which leads to a stress peak. Because of the increasingly smaller pores which are still to be filled, the capillary forces continue to rise. The drying stress maximum is reached when the solvent is evaporated from most of the pores. In the shown example a maximum drying stress of 0.86 MPa occurs. As soon as all solvent is evaporated, the drying process is finished. Depending on the addition of additives, a fraction of the stress can remain in the dried layer. It is frozen in during drying and can not relax in the dried state where no rearrangement can take place any more. In our example, no additives were added and a complete stress relaxation can be observed.

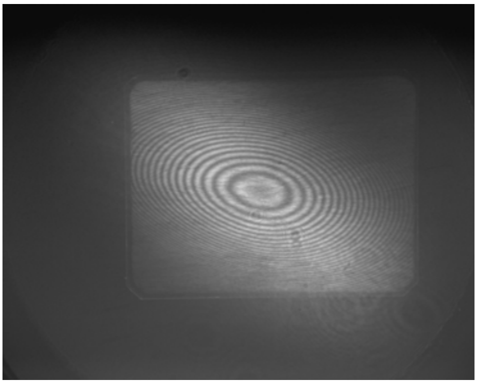

Fig. 3. Interference pattern during measurement

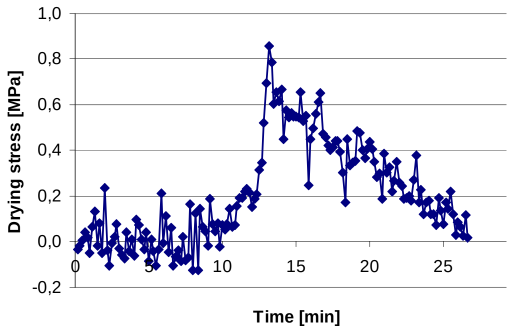

Fig. 4. Drying stress evolution during drying of a particular slurry sample

6. Conclusion

We demonstrate that our method is able to measure and evaluate tensile stress in thin drying ceramic layers on a substrate. For this purpose, we used model ceramic slurry without any additives. The next step will be introducing a prototype sensor and starting measurements of ceramic slurry with various additives.

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