**Apparatus for simultaneous stress and weight measurement
during film drying in an environmentally controlled chamber**

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The drying behavior of coatings is essential for the development of formulations in order to obtain reliable and defect free finishes. There are two major measures of interest: the development of the stress responsible for cracking and the drying rate that gives insight into the morphological structure. The cantilever deflection method is the predominant way of determining stresses under defined drying conditions such as temperature and humidity. However, both measures of interest are currently obtained using two different coatings when dried in a chamber, or a single coating with simultaneous measurements that can only be dried under ambient conditions. In this paper, we present an apparatus design based on the cantilever deflection method that allows simultaneous measurement of the stress and drying rate in an environmentally controlled chamber.

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I. INTRODUCTION

Modern coatings are found in a vast variety of products and markets. Whether it be in the automotive industry as varnishes, protective paints for wood, marine anti-fouling coatings or printing of conductive pastes or batteries.¹⁻⁶ In the strive to create environmentally friendly waterborne formulations, absent of volatile organic compounds, the suspension formulation becomes even more critical as they are prone to defects. Drying of these coatings can lead to cracking or other defects caused by stress formation within the coating.⁷⁻⁹ The drying process can be divided in different stages. At first, the suspension dries and shrinks with the characteristics of the bulk fluid at a constant rate with negligible stress increase.¹⁰ Second, as the particles have compacted, the fluid recedes into the coating thus forming menisci. Chiu et al. were the first to experimentally show that stresses that occur during drying are caused by the capillary tension induced menisci.¹¹ This understanding helps modifying coating formulations to reduce drying defects.

There are different stress measurement techniques available with varying levels of additional measure integration. These include setting the drying conditions, measuring the drying rate, and visually observing the drying by means of a camera, among others. The drawback of some of the employed techniques is that measurement results stem from different coatings. Drawing conclusions from these examinations is only valid if the obtained films are nearly identical, which is the case for easily spreadable and coatable materials such as low viscosity polymer blends. Application of thick, elastic pastes, on the other hand, often leads to local variations in thickness that greatly affect drying rates and stress build up, making a comparison between different films suspect. In this work, we present an apparatus design that allows us to simultaneously record the stress and drying rate of the same coating at specified drying conditions. The next section briefly describes different stress measurement techniques before introducing our new apparatus design.

II. STRESS MEASUREMENT TECHNIQUES

Stress measurement techniques have been evolving since the early work on the tension of metallic films by Stoney in 1909.¹² Since then, most techniques developed have been based on the deflection of a substrate. An optical technique was presented by Raju and co-workers.¹³ The measuring principle is based on a linear change in birefringence caused by a stress-dependent increase from optically isotropic to optically anisotropic properties of the fixed substrate. They claim to

obtain a more accurate stress value due to the absence of a stress relaxing deflection of the substrate. A direct comparison of their set-up with bending techniques were difficult due to a lack of adjustable drying conditions. An *in situ* drying rate was not determined.

Other techniques correlate a substrate's deflection evoked by shrinkage of an adhering coating. The result is an average stress of the coating. Details are described later in [section III F](#). In contrast, von der Ehe and Johannsmann¹⁴ developed the membrane technique that allows spatial resolution of the stresses. The sample dries on a semitransparent membrane that mirrors an array of dots placed underneath. A camera detects distortions of the image, which are related to the local vertical deflection and subsequently used to compute the spatial stress.¹⁵ Their technique only measures stresses, other measures of interest have to be determined separately. Interferometry makes use of generated Newton's rings of a monochromatic light source to determine the curvature of a material.¹¹ A sample is coated on the substrate and placed on an optical flat (glass). As it deflects, a thin air film is generated. Part of the monochromatic light ray coming from the bottom is reflected (ray 1) at the glass-air interface. The other part of the incident beam (ray 2) refracts and is subsequently reflected at the interface of the air film and substrate accompanied by a phase shift. Interference of ray 1 and ray 2 lead to constructive and destructive interference patterns with a distance of half the light's wavelength known as Newton's rings allowing the determination of the deflection at a given length. Suspending the optically flat substrate from a scale allows simultaneous measurement of weight loss and shrinkage as carried out by Chiu et al. (1993b)¹¹ and Guo et al. (1999)¹⁶. Bauer and co-workers (2009)¹⁷ extended this method by using a transparent substrate, which allows the film saturation and drying front to be tracked. Drying conditions were ambient and not adjustable.

Drying stresses often anisotropically propagate in stress fronts, which are difficult to track. However, the integrated stress can be determined by the predominant stress measurement technique: the cantilever deflection method.¹⁸ One end of the beam is clamped while the other end is free to bend as sketched in [Figure 1](#). With this technique, however, it is only possible to measure an overall stress. This includes stresses caused by other effects such as an uneven coating or lateral drying caused by the narrow cantilever.^{19–21}

The adhering and shrinking coating exerts stress on the substrate causing it to deflect. A photo diode (position sensing detector, PSD) records the distance a reflected laser beam has traveled due to the curvature of the cantilever. This technique facilitates stress measurement at specified drying conditions by placing the clamped cantilever in an environmentally controlled chamber.

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FIG. 1. Sketch of the cantilever deflection technique. The laser beam is reflected at the bottom of the substrate and recorded by the position sensing detector (PSD). Upon shrinkage induced bending of the cantilever, the laser travels from point 1 to 1' on the PSD.

Since the cantilever deflection can be small (a few micrometers), rapid and precise placement inside a drying chamber would be required for simultaneous measurements. To date, this has not been possible. Instead, most researchers coat a second cantilever and insert it in the same chamber in order to obtain the drying rate under the specified conditions. This second cantilever is then placed on a suspended balance pan connected to a weight scale outside the chamber.^{19,21–27} However, this assumes identical coatings as well as spatially identical drying conditions inside the chamber that are challenging to obtain. So far, obtaining a true simultaneous measurement of stress and drying rate of the same coating was associated with trade-offs. Fu et al. (2015)²⁸ obtained the simultaneous measurement by placing the clamped cantilever directly on a weight scale with the drawback of drying at ambient conditions. Kiennemann and colleagues (2005)²⁹ obtained the drying rate at different temperatures by placing the sample on a heated plate sitting on a balance but without information about the occurring stresses.

Our design of the cantilever deflection set-up allows the simultaneous measurement of stress development and drying rate of the same coating in a temperature and humidity controlled environment. Such a chamber enables the development of ideal drying conditions. In the following section, we will describe the design and peripheral devices in detail.

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FIG. 2. Photograph of the stress measurement apparatus on top of an optical table. The cantilever fixture is inside the chamber, suspended on a rod from the analytical balance. The calibration tool is removed before a stress measurement.

III. DESIGN OF STRESS MEASUREMENT SETUP

A. Apparatus setup

The stress measurement set-up is build on a vibration reducing, passively isolated optical table topped with a breadboard (Nexus, Thorlabs, USA). The essential parts of the apparatus are build on top of the breadboard, as shown in [Figure 2](#), with supporting parts beneath. An aluminum frame is used to secure two horizontal plates (approximately) 700 mm and 450 mm above the breadboard. An analytical balance (Sartorius Cubis MSA 224S, Sartorius Lab Instruments GmbH & Co.KG, Göttingen, Germany) is placed on top of the upper plate, which is machined with a 50x50 mm opening. Through that hole, a rod reaching into the drying chamber is connected with the suspension weighing mechanism of the scale. The attachment mechanism is described in [section III D](#). The lower plate is machined with a hole of 50 mm. Mounted on top is the

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FIG. 3. Front view of the aluminum drying chamber (dark grey) covered with polycarbonate insulation sheets (blue). The cantilever with fixture suspended from a rod is placed centrally. A humidity sensor registers the water content of the air inlet on either side of the chamber. Temperature sensors track and control the heating mats (orange).

insulated drying chamber equipped with air feed tubes on the sides and described in more detail in [section III B](#). An opening in the roof of the chamber allows the insertion of the calibration tool and is replaced by a lid during measurement and an optional USB-camera. On top of the large breadboard (bottom of [Figure 2](#)) a laser (TLS001, 635nm, Thorlabs, USA) is mounted on the left and the lateral position sensing detector (PDP90A, Thorlabs, USA) on the right. Both are attached to posts to adjust their height and in-plane adjustments are made via moving stages equipped with micrometer screws. For safety precautions as well as exclusion of other light sources, the laser and PSD are enclosed by a door and anodized aluminum sheets. The set-up is leveled by manually changing the pressure in the vibration isolators.

B. Drying chamber

A schematic showing the interior of the drying chamber is shown in [Figure 3](#). Before air enters through the tubes on either side of the chamber, it can be conditioned to the desired humidity. This is accomplished using the manual air control unit shown in [Figure 4](#). At first, dry compressed air

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FIG. 4. (a) Manual air control unit. The pressure regulator limits the compressed air pressure. Airflow is split and passes through two flowmeters. One stream is directed through a heatable bubbler to obtain water saturated air (b). Both streams are mixed and flow into the chamber.

105 passes the pressure regulator and is split into two flows with individually adjustable volumetric
106 flow rate as depicted in Figure 4a. The pressure regulator is used to protect the flowmeter from
107 overpressure from the centrally provided pressurized air. Each flowmeter has a range of 0-10
108 l/min. One of the air streams flows through a bubbler placed on a heating plate as photographed in
109 Figure 4b. The bubbler can be heated via a water bath to raise the absolute air saturation as needed.
110 This is necessary when the established absolute water saturation of air is insufficient at the obtained
111 wet bulb temperature. The water saturated air stream and dry air stream are then mixed and enter
112 the drying chamber while passing the vertically arranged heating elements (orange in Figure 3).
113 An insulation layer (blue) around the aluminum chamber prevents excessive heat loss. The sheets
114 are made of polycarbonate with a thickness of 15 mm and can resist a constant temperature of
115 120 °C. The drying temperature is regulated with a PID controller with the sensor (not shown)
116 centered in longitudinal direction and approximately 10 mm above the cantilever. The silicon
117 heating mats (HORST GmbH, Lorsch, Germany) are protected from overheating by means of a
118 safety shut-off if a specified temperature is exceeded. Their nominal temperature is 200 °C but the
119 safety shut off was set to 170 °C. The PID settings have to be chosen accordingly. With a step wise
120 temperature increase, uniform chamber temperatures up to 90 °C can be reached. The humidity,
121 registered by the RH sensor (dark blue circle), can be manually adjusted by means of changing
122 the flow rates of saturated and dry air streams on the flowmeters. Due to poor insulation of the

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FIG. 5. (a) Coating support machined from a steel slab with recess for the fixture and cantilever (the grid is 10 mm). (b) Power supply for adjusting the coating speed through voltage. Turning the switch starts dragging the coating blade. Image in (a) is reproduced from Fischer and Koos³⁰ with permission from John Wiley & Sons Inc.

humid air tubing and, thus, water condensation inside the tube, the attainable relative humidity inside the chamber at 40 °C is limited to 50%. Care has to be taken with thermal bridges at the air inlet. Condensed water may drain and damage the equipment. An additional temperature sensor (red circle) allows temperature gradients inside the chamber to be measured. In the middle of the chamber, the suspended fixture with the clamped cantilever is connected to the analytical balance. In order to further prevent heat losses as well as to maintain the specified drying conditions, the 50 mm hole through which the laser (red line) passes is closed with a 1.5 mm thick cuvette quartz glass (Hellma GmbH, Germany).

C. Coating procedure

A prepared sample is coated onto the cantilever outside the chamber. For that, we machined a steel slab with a recess for the cantilever as well as the fixture as depicted in Figure 5a. To ensure an equal height for the coating support and the fixture, their surfaces were sanded together. Rails were added such that the coating blade only moved along one axis. Below the cantilever we added a support topped with a weak magnetic strip as shown in Figure 5b. This method maintains a constant contact of the cantilever between coating procedures and aids in attaining repeatable film

thickness. The coating blade (ZUA 2000, Zehntner GmbH, Sissach, Switzerland) with the desired gap height is dragged by a string along the cantilever. The coating speed is adjusted by means of supplying a voltage (up to 12 V) to a small motor, such that a coating speed of up to 0.29 m/s can be achieved. The speed can be further increased by mounting a larger reel than the current one, which measures 47.74 mm in diameter. Afterwards, the fixture is inserted in the chamber as described in the next section.

D. Inserting and attaching procedure

After the cantilever has been coated, the fixture is quickly inserted into the drying chamber. The simultaneous stress and suspended weight measurement poses several challenges. First, the space to access the chamber is very limited. Large openings as well as a long insertion period would significantly prolong the time to establish equilibrium conditions upon closing the door. Second, the position and orientation of the cantilever needs to be as constant as possible between measurements to increase repeatability of the measurements. The insertion tool, as well as the attachment mechanism used to enable weight tracking have to be designed such that the fixture is lifted and attached with the correct orientation. The three relevant degrees of freedom are roll, pitch, and yaw. The design of our developed tool is sketched in [Figure 6](#). The guide rail bracket (gray) is placed on the rails mounted in the chamber and allows linear movement of the tool into and out of the chamber. A lifting lever (green) lifts the fixture against the lower support (blue). The tilting support (purple) is connected to the accessible turning wheel outside the chamber via the belt drive principle. Fitted centering pins attached to the tilting support maintain the yaw with good precision during lifting and insertion. The interplay with the fixture and attaching mechanism is depicted in [Figure 7](#).

The attaching mechanism is equipped with a neodymium magnet strong enough to hold the fixture with the clamped cantilever, preventing roll and pitch, but weak enough that the fixture can be later released without damaging the film. Furthermore, the magnet has an attached centering needle that ensures minimum yaw and correct x, y, and z positioning between each measurement. The needle neatly fits in a small hole drilled in the magnet bracket, which in turn is mounted on the cantilever fixture. The entire insertion and attaching mechanism can be described as follows. After placing the coated fixture on the lifting tool and opening the chamber door, the tool is placed on the guide rails and pushed inside the chamber until it stops at a defined distance. The fixture

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FIG. 6. Tool for lifting the cantilever fixture at a precise position and orientation in order to connect to the suspended weighing mechanism. The lifting lever (green) lifts the cantilever above the lower support (blue) and the tilting support controls the angle of the fixture.

is lifted until it is stopped and is maintained at the elevated position. At this point, the tilting support and fixture is tilted in forward position, and aligned so that the centering needle penetrates the hole in the magnet bracket. Afterwards, the tool is retracted from the chamber while turning the wheel, which brings the cantilever into a horizontal position. At a nearly horizontal position, the magnet lifts the fixture off, after which the tool is lowered, fully retracted from the chamber, and the door closed. With some practice, this procedure takes less than 15 seconds and ensures an accurate and repeatable positioning of the cantilever. Given that the PSD, the suspension weighing mechanism with needle and the fixture with cantilever are properly aligned, there should only be little variation in the PSD's x-position. Otherwise the alignment of the individual parts has to be checked. Another source of error could be an uneven coating leading to a change in the roll direction and thus a change in the laser's x-position on the detector.

E. Data acquisition

The aim of the data acquisition (DAQ) is a fully integrated system to prevent transcription errors in further data processing. A sketch of the devices used is shown in [Figure 8](#). A DAQ board

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FIG. 7. Attaching mechanism with lifting tool. The centering pins and needle ensure repeatable orientation of the cantilever. A magnet connects the cantilever clamp with the rod, which in turn connects to the suspension weighing mechanism of the analytical balance.

(NI USB–6218, National Instruments, USA) collects the signals from the temperature transducers, the relative humidity sensor as well as three voltage signals from the PSD corresponding to the x-position, y-position, and the sum. The film weight is obtained via a USB connection with a Sartorius query protocol that upon query sends the numerical value. All signals are processed and displayed in LabView during the measurement. The sampling rate can be set to at least 500 ms. After each experiment, the film thickness obtained from the stylus micrometer indicator is requested through a USB input tool (Digimatic USB, Mitutoyo, Japan) and appended to the measurement data with LabView in order to obtain a single file containing all necessary information. Further data processing such as stress calculation can be done in any suitable program. Similarly, the calibration tool is connected via USB and a desired amount of measurement points per deflection can be recorded.

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FIG. 8. Digitally integrated and acquired data. Measures include chamber sensors as well as pre-processing (calibration) and post processing (film thickness). Data is received via DAQ board or direct USB to the computer. Data is recorded with LabView.

F. Calibration

Calibration of the set-up is an essential part for obtaining the stresses during drying. The beam bending theory is based on Stoney's work for uniaxial film stress.¹² While the length of the cantilever is generally much longer than wide, the cantilever covering coating is much thinner than the wide. Therefore, the stress contribution in width cannot be neglected, leading to a biaxial in-plane stress.³¹ Later, Corcoran developed a correlation that relates the deflection/ curvature of a beam or plate with the stress in a coating causing that deflection.³² The adequate equation is applied by all techniques determining stresses through curvature of a substrate. For the cantilever deflection d , the stress σ is given by

$$\sigma = \frac{d \cdot E_s t_s^3}{3 t_c L^2 \cdot (t_s + t_c) (1 - \nu_s)} + \frac{d \cdot E_c (t_s + t_c)}{L^2 \cdot (1 - \nu_c)} \quad (1)$$

where subscripts s and c represent substrate and coating, respectively. The substrate currently used in our design is a steel cantilever with thickness $t_s = 0.196$ mm, the Young's modulus $E_s = 210$ GPa, Poisson's ratio $\nu_s = 0.3$, and the cantilever's free length $L = 40$ mm.

A drawback preventing the straightforward application of Equation 1 is its second term. It

contains the coating's Young's modulus E_c at the time of measurement. As it is usually challenging to obtain, one tries to have an experimental design that allows dropping the second term. The temporal coating thickness t_c can be obtained through laser thickness measurements. However, in our current design we used a stylus micrometer indicator (ID-H530, Mitutoyo, Japan) with a precision of $\pm 1.5 \mu\text{m}$ and measured the dry film thickness. This is a good approximation since significant drying stresses predominantly arise at the compacted state. In order to drop the second term $E_s \gg E_c$ and $t_s \gg t_c$ needs to apply³². Using a steel cantilever, the first approximation applies for most coatings (particle coatings for instance^{29,33,34}). In our case, the introduced error is $\sigma_c < 10 \%$,^{28,32} for a coating thickness $t_c < 100 \mu\text{m}$.

Generally speaking, Corcoran's equation is only valid with the following assumptions:^{32,35}

- ideal adhesion between coating and cantilever;
- isotropic elastic properties of the coating and substrate;
- no plastic deformation of the coating or substrate;
- uniform biaxial stress in the coating;
- and a small cantilever deflection.

By ignoring the second term of Equation 1, the deflection is the only unknown to determine the drying stress. This is achieved by deliberately deflecting the cantilever a known distance with a micrometer head (Sylvac SA, Switzerland, Figure 9a). The U shaped tool is attached to the non-rotating spindle and only touches the cantilever at the very end as sketched in Figure 9b. A step-wise increase in deflection causes the laser beam to move on the PSD which results in a linear voltage change as shown in Figure 9c. The slope of the linear regression can then be used to calculate the cantilever's deflection at a given voltage change registered by the PSD. With the obtained deflection and dry film thickness, the temporal stress development can be computed. As can be seen from Equation 1, choosing a cantilever material with an appropriate Young's modulus, allows adapting the setup in a way such that the prerequisite assumption for small deflections is met. That is, coatings with large stress development should be measured on a substrate with higher Young's modulus such as steel. Alternatively, glass or silicon wafers can be used. However, we found that using materials different from the clamp material can cause distortions at elevated temperatures caused by stress induced through a variation in thermal expansion coefficients. Furthermore, the cantilever thickness can be chosen within technical and handling limits.

FIG. 9. (a) Micrometer head with non-rotating spindle and U-shaped tool. (b) Sketch showing the deliberate deflection of the cantilever by a known distance causing the laser beam to move on the PSD. (c) Graph of the PSD voltage response upon cantilever deflection. The slope of the regression line allows to determine the cantilever deflection at a given voltage.

IV. APPLICATION EXAMPLE/RESULTS

With the data obtained from an experiment as well as the calibration, one can directly compare changes in drying rate with the stress profile of the same coating. An exemplary result is illustrated in Figure 10. This measurement shows the drying behavior of a capillary suspension prepared with alumina particles suspended in 1-heptanol and addition of a small amount of glycerol. The drying temperature was set to 40 °C and 1% relative humidity (RH). One can see that the initial drastic increase in stress is not correlated to the drying rate. Moreover, when the film is dry and the weight constant within the scale's resolution, there is still a stress increase that results in a coating with a constant residual stress. Further examples where this set-up was employed for investigation of capillary suspensions (also with the USB camera) can be found in Fischer and Koos.^{30,36}

V. CONCLUSIONS

The drying behavior of coatings gives valuable insight into how to improve formulations in order to prevent defects such as pinholes or cracks, which are related to drying stresses. Since drying conditions, such as humidity impact the microstructural development and potential binder migration to the surface (skinning), reducing the drying rate, it is important to correlate both factors. The drying conditions will influence the drying rate, to show, for example, how the drying

FIG. 10. Simultaneous stress and weight measurement of 25 vol % alumina particles suspended in 1-heptanol with 3.1 vol % added glycerol, dried at 40 °C and 1% relative humidity over time. The left y-axis shows the film weight and the stress is shown on the right. Changes in drying rate can reliably and directly be related with the stress profile.

of multicomponent formulations under humid conditions can be beneficial for a stress reduction. Therefore, a test set-up with a controllable environment is advantageous. However, it was not previously possible to obtain the stress and drying rate simultaneously using the same coating in such a chamber. Either two specimen were dried in a chamber and results combined as if it was one, or results from simultaneous measurements, which could only be performed in ambient conditions, were extrapolated to the conditions of interest. In this work, we present an experimental set-up that allows the simultaneous observation of stress growth and drying rate of the same coating. This is of special interest for investigation of pastes where identical coatings are challenging to produce in a lab environment. The apparatus allows films to be reliably measured starting very shortly after the film spreading.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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