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# Thin Solid Films



# Fatigue behavior of thin Au and Al films on polycarbonate and polymethylmethacrylate for micro-optical components

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# ABSTRACT

The thermal and mechanical fatigue behavior of thin metal films on polymer substrates has been investigated and compared for different combinations of materials, which are typical for micro-optical components: gold or aluminum film deposited on PolyCarbonate (PC) or PolyMethylMethAcrylate (PMMA) substrate. Mechanical fatigue testing has been carried out using an experimental setup, which allows for testing in an equi-biaxial loading condition, mimicking the strain state of the film during thermal cycling. Using scanning electron microscopy, fatigue damage morphologies for the different film/substrate combinations have been found to be quite different for both thermal and mechanical cycling. Furthermore, our results indicate a somewhat lower resistance of the films deposited onto PMMA as compared to PC to both thermal and mechanical fatigue. Under mechanical loading, Au/PC specimens show a longer time to failure as compared to the Al/PC specimens.

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# 1. Introduction

Thin metal films are commonly used in current technologies for the fabrication of micro-optical devices. Structures composed of nano-scaled metal films deposited on polymer substrates, which offer manufacturing flexibility and possible cost effective production, are currently implementing mirroring and waveguiding functionalities [1–3].

One reliability issue in such devices is the damage evolution of the metal film as a result of mechanical and/or thermal cyclic loading conditions. This is owing to fatigue damage that leads to degradation of the optical properties, e.g. the reflectivity of a mirror component in a device. It is well known that cyclic temperature changes result also in a mechanical loading conditions with high stresses in the film if the film and substrate have different thermal expansion coefficients [4]. As a result, the films may show, depending on the combinations of materials properties, damage such as voids [5], cracks [6], and roughening or hillock formation [7–9] even after only one or two cycles. For films on polymer substrates, it has been shown that cyclic mechanical loading conditions with typical ranges between 0.1% and 1% strain lead to the formation of damage such as cracks, surface roughening and extrusions of metallic films [10-13]. This is in contrast to monotonic loading, where cracking of metal films on compliant substrates does only occur when large strains (>10%) are reached [14,15]. Therefore it can be concluded

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<sup>1</sup> Present address: GKSS-Research Centre Geesthacht, Institute of Materials Research, Materials Mechanics, Max-Planck-Strasse 1, 21502 Geesthacht, Germany. nents is given by the ISO 9022-2 norm, which recommends a thermal test consisting of 5 cycles between -40 and +55 °C [16]. This test takes several hours and does only characterize the low cycle fatigue behavior of the components under rather extreme conditions. If the component survives this test, a certain safety for devices in many applications can be guaranteed. However, the test does not allow the prediction of the lifetime of micro-optical components.

As described and validated in a previous article [17], replacing thermal by mechanical cycling allows not only a significant acceleration of the test, but also to separate the thermally and mechanically affected deformation mechanisms. The method, described in [17], makes use of a dedicated experimental setup, which allows for testing in equi-biaxial loading condition, mimicking the strain state during thermal loading.

In this work, we have applied the method to investigate the fatigue behavior of different film/substrate material combinations in terms of lifetime as well as fatigue damage morphology. It will be shown that it is possible to describe the lifetime of the films by a Coffin–Manson type of relationship, where the number of cycles to failure is related by a power law function to the strain amplitude.

## 2. Experimental methods

## 2.1. Materials

We have investigated the thermal and mechanical fatigue behavior of gold and aluminum thin films deposited onto polymer substrates,

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that cycling in mechanical loading plays a major and detrimental role, which is even enforced when the temperature is changed. A straightforward assessment of the reliability of optical compo-

i.e. PolyCarbonate (PC) and PolyMethylMethAcrylate (PMMA). Thermal cycling tests have been performed on micro-optical elements, with film and substrate thickness of 400 nm and 1.28 mm, respectively. These components with 3D microstructures are produced by hot-embossing of the polymer and physical vapor deposition of the film [3,18]. Deposition of the metallic films has by done in an Univex 450 deposition chamber with electron beam vaporizer. Deposition has been done in two steps: in the first step, the sample has been tilted by 20° and 280 nm of metal has been evaporated on the polymer substrate. Then the chamber has been opened and the sample has been rotated by 180°, and tilted to 0°. During the second step, a layer of 200 nm of metal has been deposited. For both steps, an evaporation rate of 0.7-0.9 nm per seconde has been used. Using the same processes, flat samples for the mechanical fatigue tests have been produced. These specimens, designed for our experimental setup, are circular with a diameter of 70 mm, and have the same film and substrate thicknesses as the micro-optical components. For the Au films on both types of substrates, a 10 nm chromium under-layer has been deposited to enhance adhesion.

Prior to fatigue testing, the microstructure of the metal films has been investigated. According to Focused Ion Beam (FIB) and Atomic Force Microscopy investigations, the mean grain size for both types of metal films can be estimated to be of the order of 50 nm.

#### 2.2. Thermal and mechanical fatigue testing

Micro-optical elements have been thermally cycled in a climate chamber HYGROS 15C, according to the ISO 9022-2 norm [16]. This norm refers to 5 cycles between -40 and +55 °C, at a rate of 0.7 K/s, with additional hold periods of 2.5 h at the minimum and maximum temperature.

For the mechanical testing, we make use of an experimental setup, which allows to reproduce a biaxial stress state, which is equivalent to the one created by temperature changes. This setup has been presented and validated in a previous article [17], and is described only briefly here. It is based on the ring-on-ring test, in which the specimen is supported by a ring while a concentric ring of smaller diameter loads the opposite face. It produces a homogeneous biaxial tensile strain and stress state inside the smaller ring. We have modified the conventional ring-on-ring experiment by using support and loading rings on both sides of the specimen, in order to induce alternating tensile and compressive strain in the film.

An *in situ* failure detection system, especially designed for our microoptical mirrors, includes a continuous observation of the optical dispersion of the metal film, which increases with the development of damage in the film. It uses a laser diode and photo sensors. The laser beam is introduced perpendicular to the film surface, while the sensors are facing the metal film but outside of the optical way of the reflected laser beam. An increase of the light intensity detected by these sensors is correlated to an increasing dispersion of the laser beam by the film. Therefore this signal can be used to detect the onset of damage

Table 1

Young's modulus, thermal expansion coefficient difference and strain amplitude in the metal film due to a temperature change between -40 and +55 °C, to which we refer as standard thermal test

	Au 78.5 Au on PC		Al 70.6		PC 2.3		PMMA	
E (GPa)							3.14	
			Au on PMMA		Al on PC		Al on PMMA	
$\Delta \alpha_{20 \circ c} (K^{-1})$	5.1 10 <sup>-5</sup>		5.4 10 <sup>-5</sup>		4.15 10 <sup>-5</sup>		4.45 10 <sup>-5</sup>	
	$\Delta \epsilon_t$	$\Delta \epsilon_m$	$\Delta \epsilon_t$	$\Delta \epsilon_m$	$\Delta \epsilon_t$	$\Delta \epsilon_m$	$\Delta \epsilon_t$	$\Delta \epsilon_m$
+20 → -40 °C (%)	-0.376	-0.292	-0.397	-0.296	-0.381	-0.240	-0.401	-0.260
+20 → +55 °C (%)	+0.220	+0.170	+0.232	+0.182	+0.222	+0.140	+0.234	+0.151



Fig. 1. Load versus strain for five cycles of a fatigue test of an Au/PC specimen.

formation, as well as failure in the film in terms of optical functionality. For instance, an increase of the surface roughness has been detected for the gold films prior to the occurrence of cracks and extrusions [17].

Using the setup, we have mechanically reproduced the strain amplitude created by a certain temperature change. The strain amplitude corresponding to a standard thermal cycle (ISO 9022-2) has been estimated by 2D finite element elastic calculations, using the commercial code ABAQUS. This has been necessary as the film material is much stiffer than the substrate, and the common thin film assumption to neglect the compliance of the substrate can not be applied. Table 1 summarizes the total strain amplitude (i.e. the sum of the thermal and mechanical strain amplitude),  $\Delta \varepsilon_t$ , and the mechanical strain amplitude,  $\Delta \varepsilon_{\rm m}$ , in the film related to a decrease of the temperature from +20 °C to -40 °C, and for an increase from +20 °C to +55 °C, for the different materials combinations studied. For this calculation the difference in the thermal expansion coefficients has been assumed to be constant. However, thermal expansion coefficients are temperature dependent, particularly for polymers, and, therefore, the calculated strain amplitudes can only be regarded as an estimate.

The influence of the strain range on the specimen lifetime has been studied with strain ranges of 12.5%, 25%, 50% and 100% of the total strains given in Table 1.

The mechanical tests were conducted under load control using two different servo-hydraulic testing machines SCHENCK MP312, equipped with load cells of 10 or 40 kN maximum load. The tests were performed at room temperature, with a frequency of 0.2 Hz. The strain in the specimens during the test is measured by a strain gauge, glued on the backside of the polymer substrate. As a typical example, Fig. 1 shows load and strain for five loading cycles for an Au/PC specimen, loaded with a strain amplitude corresponding to 100% of the total strain given in Table 1. As the deformation was measured by a gauge glued on the backside of the specimen, a compressive strain of the gauge corresponds to a tensile strain in the film. Nevertheless, the measured strain range can be assumed to be identical to the one for the film. The non-linearity and the hysteresis in the load/strain diagram are observed as a consequence of the visco-elastic behavior of the polymer substrate. Data acquisition is performed by using DasyLab® operating under Windows®. It reads continuously the applied force, the strain and the light intensity detected by the photo sensors.

The fatigue damage of the metal films has been characterized using a Philips FEG XL30 ESEM scanning electron microscope (SEM), and a FEI 200xP dual beam FIB work station. The surfaces of the mechanically tested specimens have been inspected after a fixed Download English Version:

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