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Study of creep/relaxation mechanisms in thin freestanding nanocrystalline palladium films through the lab-on-chip technology

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A nanomechanical lab-on-chip set-up has been used to study the creep/relaxation response of thin palladium films with temperature. The basic idea is to use residual stresses present in a silicon nitride thin beam to load the test film after etching the underneath sacrificial layer. The main advantage of this experimental method is that we can simultaneously perform thousands of creep/relaxation tests without monopolizing any external actuating/loading equipment and without using any time consuming calibration procedures. A signature of the dominant relaxation mechanism is given by the activation volume which has been determined for different levels of plastic deformation and different temperatures. The activation volume is equal to $\sim 15\text{--}40\text{ b}^3$ at room temperature and tends to decrease with increasing plastic deformation. The activation volume decreases when relaxation takes place at 50°C down to $\sim 7\text{--}20\text{ b}^3$. These variations of the activation volume indicate the competition between two different thermally activated deformation mechanisms in the temperature range between 20°C and 50°C . © 2013 American Institute of Physics. [<http://dx.doi.org/10.1063/1.4775398>]

I. INTRODUCTION

Time dependent relaxation/creep mechanisms are amplified in nanocrystalline materials compared to traditional microcrystalline systems.¹ This fact originates from the switch in deformation mechanisms from dislocation mediated plasticity, involving forest hardening, to more thermally activated mechanisms associated to the predominant contribution of the grain boundaries. These mechanisms involve the nucleation of dislocations at grain boundary ledges, diffusion along grain boundary, grain growth, and grain boundary sliding. The strain rate sensitivity of fcc metals has been shown to increase when the grain size decreases; while for bcc metals, it is sometimes reduced.² For instance, the recent literature has revealed large rate sensitivity exponents m , defined as $\partial \ln \sigma / \partial \ln \dot{\epsilon}$ with σ the stress and $\dot{\epsilon}$ the strain rate, with values on the order of 0.01–0.03 for nanocrystalline (nc) Ni,³ 0.06 for nc Pd,⁴ 0.07 for nc Al,⁵ 0.03–0.15 for nc Au,⁶ 0.04–0.05 for nc Cu,^{7,8} 0.03–0.20 for nc Au-Cu alloys.⁹ These values are typically one order of magnitude larger than in the microcrystalline counterparts. The increased rate sensitivity of nanocrystalline fcc metals results in delayed shear localization.⁶ On the contrary in bcc metals, the literature reveals small rate sensitivity exponents, for instance, values on the order of 0.01 have been reported for nc Fe¹⁰ and 0.02 for nc Ta.¹¹

In thin metallic films (“thin” meaning thinner than approximately $1\text{ }\mu\text{m}$), the grain size is, often on the order of the film thickness and, in addition, the effect of the free

surface provides other degrees of freedom to favor, for instance, diffusion mechanisms.¹² Recently, many authors have indeed shown a significant rate sensitivity, even at room temperature, in Al,¹³ Au,⁶ and Ni films.³ Up to now, the literature has essentially focused on the relaxation mechanisms occurring under moderate to low strain rate conditions—e.g., the lowest strain rate in Ref. 6 is $6 \times 10^{-6}\text{ s}^{-1}$, but the long term “slow” relaxation mechanisms in thin films and nanocrystalline systems have not received much attention. The analysis of relaxation in thin films, especially when freestanding, does not only offer a system of fundamental interest due to the inherent simplicity of the microstructure (often one grain in thickness), sharp crystallographic textures, and the magnified effect of the free surfaces, but it has important applications. Creep in freestanding films affects the operation of several MEMS devices such as RF-MEMS switches.¹⁴ Also, thin coatings involving large internal stresses could relax over long periods of time and lead to modifications of the overall operating conditions of the systems. The mechanical testing of freestanding thin films is an experimental challenge. In order to measure the relaxation behavior in thin films over long periods of time, the concept of lab-on-chip testing of freestanding films, initially developed in the context of the analysis of static properties,^{15–17} has been diverted from its original idea in order to address relaxation/creep.¹⁸ The technique is applied here on thin freestanding nanocrystalline Pd films because Pd is a material of interest for many applications including hydrogen storage. Indeed, Pd shows a particular affinity to hydrogen. Nowadays, Pd is used for hydrogen gas separation in micro fuel cells¹⁹ as well as MEMS-based hydrogen gas sensors,²⁰ sometimes in the form of thin freestanding membranes. The

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results presented here provide information about the relaxation mechanisms in thin freestanding Pd films based on an original test method. Sections II and III describe the experimental setup and data reduction scheme. Section IV is dedicated to the presentation of the results and to the discussion before concluding in Sec. V.

II. EXPERIMENTAL SETUP

A suite of MEMS-based microstructures has been recently developed to extract the mechanical properties of submicrometer thin films.^{15,16} The basic idea is to make use of the residual stress in the “actuator layer” (here silicon nitride with about 1 GPa internal stress) to load a “specimen layer,” in this case Pd, under uni-axial tension (Figure 1). Indeed, when removing the underneath “sacrificial layer” (here silicon dioxide) by etching, the system actuator-sample is released from the substrate and is free to deform under the action of residual stresses. The actuator pulls on the sample until force equilibrium is reached. The measurement of the displacement between moving cursors (attached to the actuator) and reference cursors (attached to the substrate) gives access to the strain and to the stress if the initial elastic strain associated to the internal stress and Young’s modulus of the actuator are known.²¹ The “specimen layer” is a Pd film deposited by e-beam evaporation. Lift-off photolithography is used to pattern the layer. Specimens have a “dogbone” shape to ensure a good anchoring between the “specimen layer” and the “actuator layer” and to guarantee a uniform stress along the Pd sample beam. The width of the specimen beams varies between 1 and 10 μm and the length ranges between a few μm up to 1 mm. The thickness of the Pd layer is measured by profilometry and it is equal to ~ 350 nm. The internal stress is determined by profilometry using the Stoney equation²² and is equal to about 390 ± 20 MPa. It has been shown previously by Idrissi *et al.*^{23,24} that these Pd films involve, on average, 1.5 grains over the film thickness direction with in-plane grain size equal to ~ 25 nm (columnar-like shape). Transmission electronic microscopy (TEM) has also revealed the presence of nanotwins inside a fraction of the grains.^{23,25} The “actuator layer” is made of low pressure chemical vapour deposited (LPCVD) silicon nitride (Si_3N_4). The layer is patterned in the shape of long beams by photolithography followed by dry etching (SF_6 plasma). The length of the actuator beam varies between a few hundreds of μm to several mm in order to generate different stress levels. The width is equal to either

10 μm or 15 μm . The thickness of the Si_3N_4 beams is measured by ellipsometry and it is equal to 115 nm. Approximately 860 MPa of residual stress was measured by profilometry in Si_3N_4 and an internal strain of -0.0031 ± 0.0001 was measured with independent test structures.¹⁸ Cursors are designed on both sides of the actuator beam and are referred to as the “moving cursors,” whereas the cursors connected to the anchors (Si substrate) are called the “reference cursors.”

The “sacrificial layer” is a thermally grown silicon dioxide (SiO_2). The oxidation of the Si wafer was made at 950 $^\circ\text{C}$ under O_2 and H_2 flows. The thickness of the sacrificial layer is 500 nm. The actuation is performed by wet etching the sacrificial layer in 73% pure fluoridric acid (HF) solution.

After the release, the sample goes into a critical point drying chamber in order to avoid stiction with the underneath substrate. Then, it is introduced in a scanning electron microscope (SEM) for observation and measurements. The SEM used for the measurements of the in-plane displacements and width of test structures is a FEGSEM Zeiss Ultra 55. Experiments were performed at room temperature (~ 20 $^\circ\text{C}$) and at $T = 50$ $^\circ\text{C}$. For experiments at temperatures higher than room temperature, as soon as the structure is released and dried, an observation is made by SEM to determine the initial position of the cursors; then the sample is introduced into an oven for a defined period of time; after this, the sample is introduced again into the SEM. After the SEM observation and displacement measurements, the sample returns to the oven for an additional period of time. The sample is then checked regularly after defined time intervals. During the time between the moment of removing the sample from the oven and its re-introduction into the oven after the SEM measurements, the temperature of the sample is not controlled anymore. When the structure is heated, a small thermal expansion of both the actuator and test material takes place, leading to a relaxation of the stress, which must be taken into account in the data reduction. At room temperature, displacement measurements were performed after 2 h, 5 days, 1 month, 3 and 4 months after the release. At $T = 50$ $^\circ\text{C}$, displacement measurements have been performed 1 h, 6 h, 1 day, and 1 month after the release.

III. DATA REDUCTION AND RESULTS

For each structure, the displacements u of the “moving cursors” relative to the “reference cursors” are measured at different periods of time, which provide the evolution of $u(t)$ with time, see Figure 2(a) at $T = 20$ $^\circ\text{C}$ and 2(b) at $T = 50$ $^\circ\text{C}$. The time interval can vary from a few hours to several months depending on the experiment temperature (room temperature or higher). From the displacements, the mechanical strain inside the beam after release can be calculated as

$$\varepsilon(t) = \ln\left(\frac{L_0 - u(t)}{L_0}\right) - \varepsilon_{\text{mis}}, \quad (1)$$

where L_0 is the specimen length before the chemical release and ε_{mis} is the mismatch strain in the specimen resulting from the deposition and subsequent fabrication steps. The stress in the specimen beam is given by

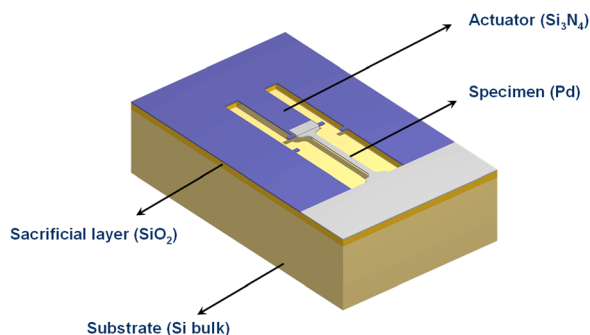


FIG. 1. Schematic view of a typical MEMS-based uniaxial tensile structure.

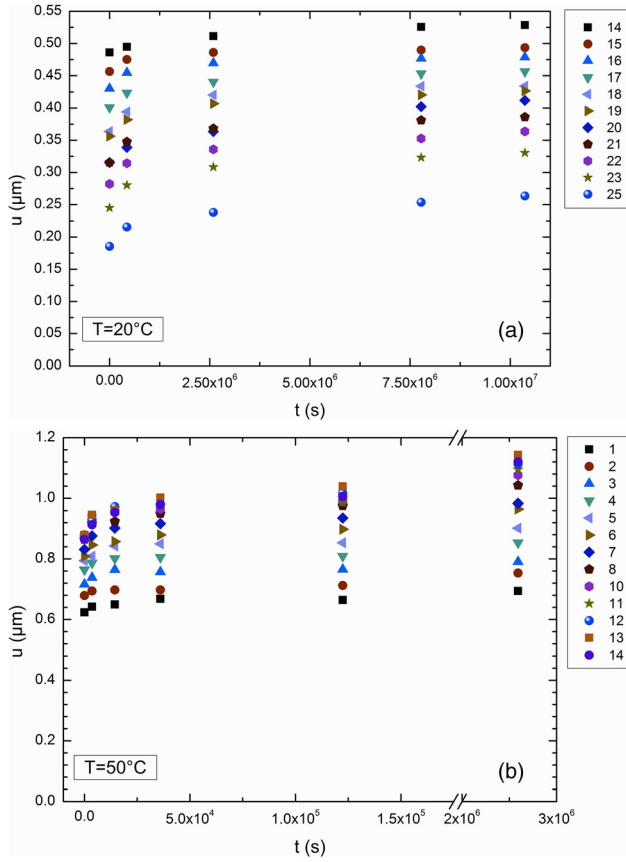


FIG. 2. Displacement versus time at (a) room temperature, (b) $T = 50^\circ\text{C}$. Each point represents the state of one MEMS based microstructure at a given time. The associated number is the reference of the specimen in the series of test structure, with decreasing specimen length (and thus increasing deformation) as the number increases (#1 is a specimen of length $250\ \mu\text{m}$ and #25 has a length equal to $5\ \mu\text{m}$). Each symbol represents the state of the same MEMS-based microstructure at different periods of time.

$$\sigma(t) = E_a \frac{S_a}{S} \left[\ln \left(\frac{L_{a0} - u}{L_{a0}} \right) - \varepsilon_{a,mis} \right], \quad (2)$$

where L_{a0} is the actuator length before chemical release, E_a the Young's modulus of the actuator, S the current cross sectional area of the beam, and $\varepsilon_{a,mis}$ the mismatch strain in the actuator resulting from the deposition and subsequent fabrication steps. These two equations enable the determination of the stress-strain response of the specimen material, see Figure 3. The room temperature relaxation tests analyzed in this paper are performed on structures with $10\ \mu\text{m}$ wide actuators and $2\ \mu\text{m}$ wide specimens, while $15\ \mu\text{m}$ wide actuators are used for the tests at $T = 50^\circ\text{C}$. The use of several test microstructures with different actuator to sample length ratios gives access to different levels of stress in the test material. The method proposed here is different than previously reported creep or relaxation techniques as both the stress and the strain vary during the loading. It is neither a true relaxation test nor a true creep test. It is similar to a relaxation test performed on an elementary structure made of a spring in series with the test specimen. More details concerning the fabrication and measurement method are given by Coulombier *et al.* in Ref. 21, as well as the procedure to estimate ε and σ when the relaxation takes place at another temperature, here at 50°C .

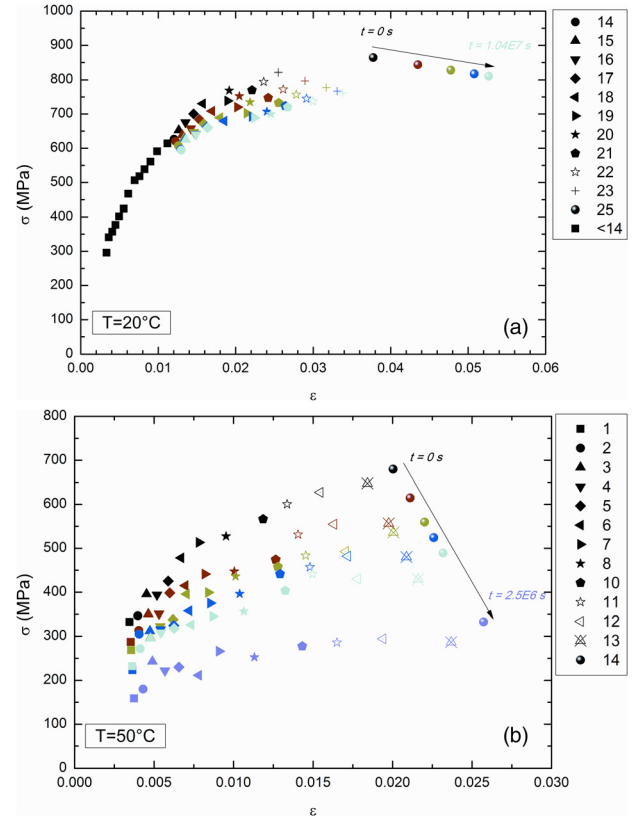


FIG. 3. Stress versus strain at (a) room temperature, (b) at $T = 50^\circ\text{C}$. Each symbol represents the state of different MEMS based microstructures at the same time.

The stress levels being quite large; the elastic strains are significant ($\sim 1\%$). As the elastic strain is not affected by thermal activation, the analysis must be performed on the plastic strain only; which is obtained as

$$\varepsilon_i^p(t) = \varepsilon_i(t) - \frac{\sigma_i(t)}{E}, \quad (3)$$

where i denotes the number used to index the test structure and E is the Young's modulus of the test material. The Young's modulus of the Pd films has been measured by nanoindentation and is equal to $120\ \text{GPa}$.²⁶ The stress evolution, $\sigma_i(t)$, is plotted in Figure 4 while the variation of plastic strain with time, $\varepsilon_i^p(t)$, is presented in Figure 5.

The effective activation volume, V , can then be calculated as²⁷

$$V = MkT d \ln \dot{\varepsilon}^p / d\sigma, \quad (4)$$

through numerical derivation. The activation volume is the physical volume over which mechanical work is performed to contribute to cross the energy barrier and trigger a transformation strain corresponding to the physical mechanism of deformation. Its measurement provides a signature of the dominant deformation mechanism. M is a factor which connects the macroscopic tensile stress to the microscopic resolved shear stress assuming the shear stress is the relevant driving force for the mechanism under investigation. A value of $\sqrt{3}$ for M is generally reported in the literature^{27–29} for textured thin films. However, in this work, the activation

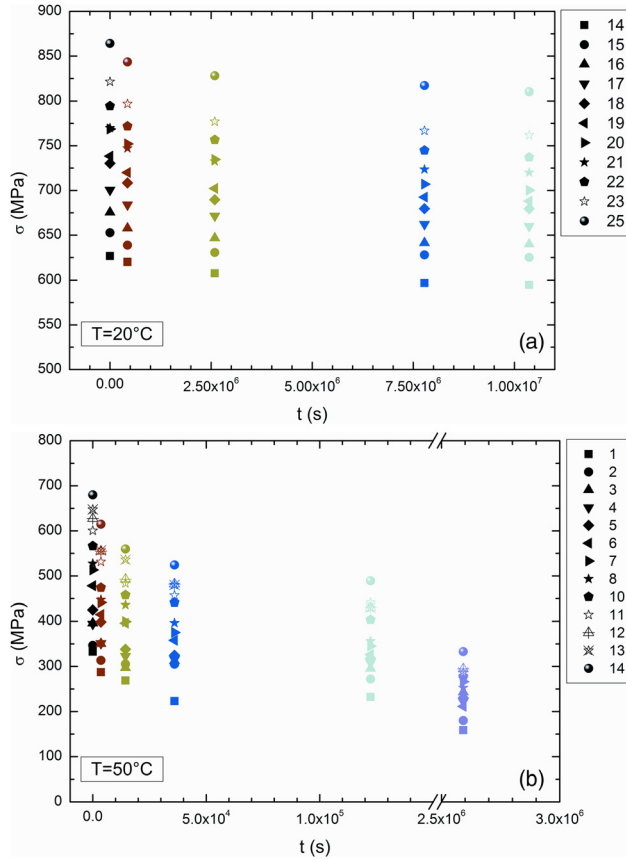


FIG. 4. Plastic strain versus time at (a) room temperature, (b) $T = 50^\circ\text{C}$. Each point represents the state of one MEMS based microstructure at a given time. Each symbol represents the state of the same MEMS based microstructure at different periods of time.

volume is reported in a normalized form as V/M (Figure 6). Typically, for diffusion, V is lower than b^3 , when dislocations nucleate at grain boundary $V \approx 2 - 20b^3$, while the recovery of dislocations in a forest by depinning involves $V > 100b^3$, varying with dislocation density.²⁷

IV. DISCUSSION

By fitting a power law equation of the type $\sigma = K\epsilon^n$ on the stress-strain curve just two hours after the release of the structures (Figure 3), the strain hardening exponent, n , is equal to 0.40 ± 0.02 which is a very large value for typical nanocrystalline materials. The large strain hardening capacity of the Pd films has been the subject of former investigations^{23,26} and results from a combination of kinematic hardening and of the contribution of nanotwins to the pinning and production of dislocations. Figure 4 shows that the stress levels applied to the specimen at 50°C are much smaller than at room temperature due to the relaxation coming from the thermal expansion of both the actuator and test specimen.

Figure 6(a) shows the variation of the activation volume averaged during the time of relaxation as a function of the average stress during the relaxation. The activation volume is a signature of the dislocation mobility mechanism (the Burgers vector of Pd is equal to 0.275 nm ³⁰). Figure 6 shows that the activation volume evolves inversely proportional to the stress at $T = 20^\circ\text{C}$, being in agreement with the Cottrell-Stokes

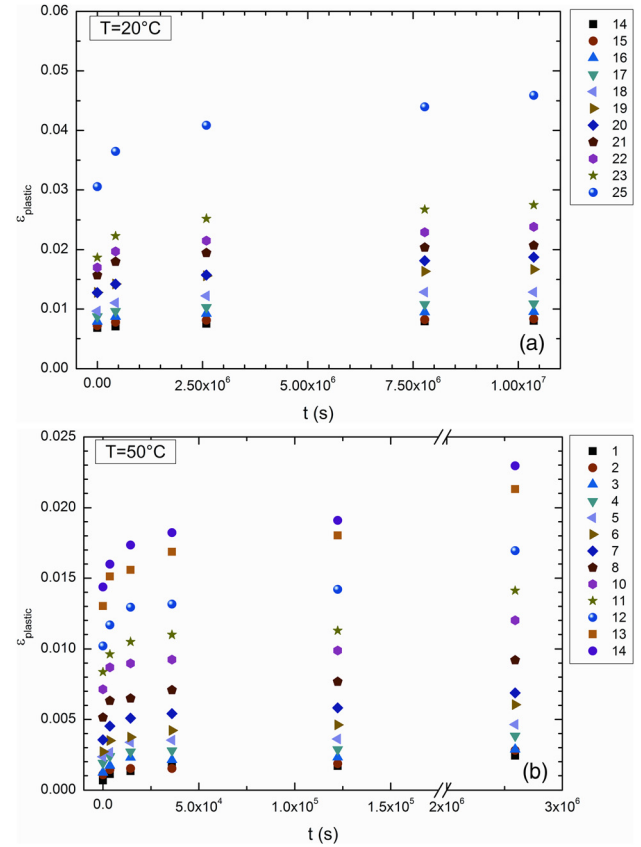


FIG. 5. Stress versus time at (a) room temperature, (b) $T = 50^\circ\text{C}$. Each point represents the state of one MEMS based microstructure at a given time. Each symbol represents the state of the same MEMS based microstructure at different periods of time.

law.^{27,31} A higher stress corresponds to a larger plastic strain, hence to a larger dislocation density and thus to a smaller distance between pinning points. Even though the grain size is very small, detailed TEM analysis shows the accumulation of full dislocations inside the grains where the typical spacing could be on the order of 5 to 10 nm. When the activation volume is between $10b^3$ and $100b^3$, the rate process typically involves cross-slip whereas smaller activation volume implies grain boundary dislocation nucleation, grain boundary sliding or grain-boundary diffusion process. At $T = 50^\circ\text{C}$, the activation volume is significantly smaller and tends to $\sim 7b^3$ at the largest stresses which indicates the transition to the activation of another mechanism, such as dislocation nucleation at grain boundaries.^{6,32,33} The change of behavior with such a small temperature difference is intriguing and the subject of more investigation. In Ref. 34, the deformation behavior of nanocrystalline Pd films was investigated at room temperature and at $T = 60^\circ\text{C}$. The films were not freestanding but deformed while sitting on a compliant polyimide substrate. The stress evolution was extracted using a synchrotron-based *in situ* tensile testing technique. This study also showed that plasticity was essentially taking place along grain boundaries at temperature slightly higher than room temperature (50°C - 60°C) and the deformation behavior of nanocrystalline Pd was still governed by dislocation mediated plasticity at large stress. The present study confirms that the rate-dependent deformation behaviour of nanocrystalline Pd with grain sizes in the range

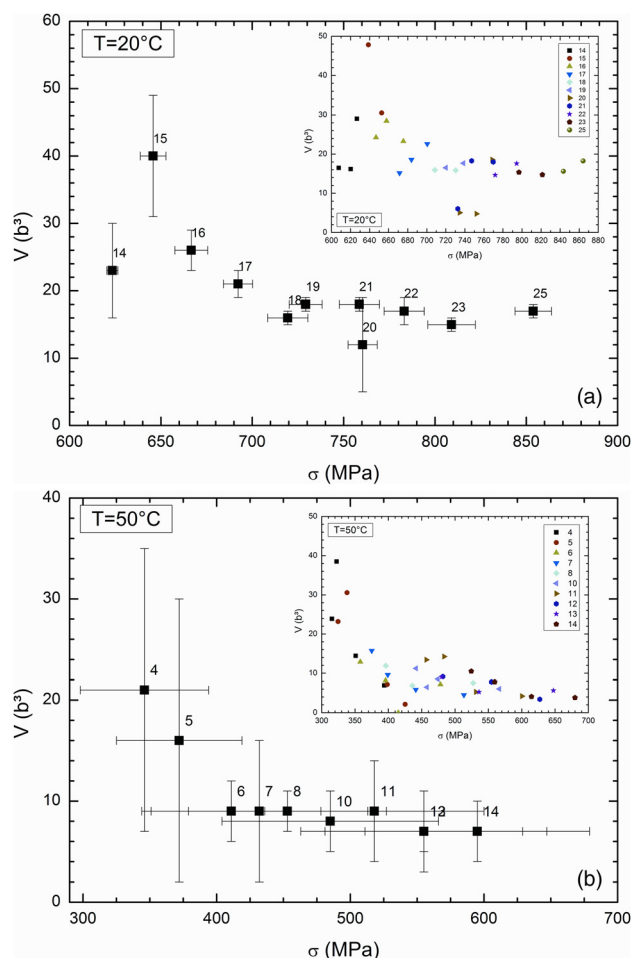


FIG. 6. Activation volume versus stress at (a) room temperature, (b) $T = 50^\circ\text{C}$. The activation volume is mean value over the entire relaxation time. Inset: The activation volume is given as a function of stress at several times of relaxation for the different test microstructures. Each symbol represents the state of one MEMS-based microstructure.

of 20 to 50 nm shows a transition of mechanism in the 20°C to 50°C temperature range. This is an important conclusion as it very much impacts the creep and relaxation behavior of Pd membranes, for instance, that would operate in this range of temperatures.

Finally, Figure 6(b) shows the variation of the activation volume calculated for each time interval during relaxation as a function of the average stress during the time interval. The scatter increases but no clear or reproducible variation of the activation volume with relaxation is observed. The average values of Figure 6(a) contain all the relevant information.

V. CONCLUSIONS

The lab-on-chip technology used in this paper allows measuring the mechanical properties of submicron palladium films without time consuming calibration procedures and without any sophisticated actuating or loading devices. The activation volume of the palladium films has been determined at two different temperatures and over a wide range of stress levels. Furthermore, the evolution of the activation volume with stress follows a Cottrell-Stokes type evolution at room temperature and at $T = 50^\circ\text{C}$. These two sets of experiments at 20°C and 50°C share a common stress range

around 600–700 MPa where the activation volume can be directly compared. For each stress value belonging to this common stress range, two different activation volumes at 20°C and 50°C indicate that the presence of two different deformation mechanisms contributing to relaxation. A transition between these two dominant mechanisms is observed with the normalized activation volume changing from $\sim 15\text{--}20b^3$ down to $\sim 5\text{--}10b^3$ when temperature increases from 20°C to 50°C and sufficient plastic pre-strain is imposed. The change of temperature from 20°C to 50°C seems to induce a transition from dislocations-dislocations dominated interactions to dislocations-grain boundaries dominated interactions. Further work is in progress to unravel the physical mechanisms underlying this change of behaviour.

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