06

The effect of heat treatment on the physical and mechanical properties of thin-film membrane-type Al structures of various shapes

© N.A. Djuzhev, E.E. Gusev, I.V. Kushnarev, V.A. Bespavol

National Research University of Electronic Technology, Zelenograd, Moscow, Russia
E-mail: bubbledouble@mail.ru

Received June 24, 2024
Revised September 5, 2024
Accepted September 5, 2024

An approach to modifying mechanical properties through the processes of thermal recrystallization of thin-film aluminum structures made in the form of square, pentagonal and circular membranes fabricated by magnetron deposition has been implemented and explained for the first time. The experiment was performed according to the planar silicon technology. Modification of mechanical properties was revealed after heat treatment in vacuum at 450°C for 1 h (for example, critical rupture pressure of a circular membrane decreased from 4.9 to 4.0 atm, average variation in the critical rupture pressure decreased from ± 1.5 to ± 0.9 atm, biaxial modulus of elasticity decreased by 24 GPa); the variations were induced by changes in the material structural properties (grain size increased almost 2 times, roughness increased from 76 ± 4 to 4480 ± 90 nm). Additional heat treatment allowed creation of more reliable Al-membranes.

Keywords: mechanical properties, grain size, thin films, membranes.

DOI: 10.61011/TPL.2025.01.60141.20034

The importance of this study is confirmed by the high frequency of microdevicesfailures due to low mechanical reliability of microcomponents [1]. One of the most common external factors affecting mechanical properties is the exposure to heating [2]. Heating of the material can occur either intentionally during the device fabrication, or involuntarily at the stage of the device active operation under harsh conditions. The problem of lack of information on exact mechanical properties of materials makes it difficult for developers of new nano— and microsystem technology devices to select a proper relationship between the service life of microcircuits containing multilayer structures and upper ranges of the device operating parameters.

A number of factors are known that affect the mechanical properties: the effect of reduction in the thickness of a single layer in a multilayer structure (the overall thickness being preserved) [3], variation in the atomic ratio between the alloy elements [4], reduction in surface defects [5], doping the film material with atoms of another substance, e. g. silicon [6], increase in the film–substrate binding energy, radiation effect on the material [7] (including that in X-ray lithography or in space), effect of the grain orientation and size [8], cyclic load effect.

This study examines the effect of heat treatment and test object shape on mechanical properties of Al often used for interconnections of the integrated circuit elements or as sensor membranes.

Studies are known which are devoted to the mechanical properties adjustment via heat treatment both for other materials Ni–Co [2] or Cu [9] and for Al [10]. Under the temperature impact, the elastic modulus changes [11], and the films become denser. In the titanium nitride and aluminum nitride films, the shape of mechanical stress depth distribution changes from the sharp „bell" to mild „„bell" [12], while average residual stresses decrease slightly (in absolute value) from -5.84 to -4.98 GPa [12].

In this work, thin films were created by magnetron deposition on the Si(100) substrate at the temperature of 180°C; the working chamber vacuum was 0.2 mbar (setup MAGNA). The aluminum film deposition rate was 50 nm/min. In this way, circular, square and pentagonal membranes of equal areas were formed on one plate by the group method. The circular membrane diameter was 1095 μm. The aluminum membrane thickness was 2200 ± 100 nm. This diameter was chosen based on the necessary relationships between the applied pressure, membrane thickness and characteristics of the metering devices. The selected membrane thickness, on the one hand, allowed formation of a fairly uniform film; on the other hand, it allowed one to refrain from passing to the range of nanoscale effects. Fig. 1, a presents the dimensions of a set of fabricated crystals in comparison with a coin and, in addition, a schematic image of the test structure. Fig. 1, b presents SEM images of the aluminum-film membrane and membrane local region.

Fig. 2 demonstrates the dependence of the square membrane deflection on excess pressure. Comparing (in the absence of external pressure) the square membrane surface reliefs before and after heat treatment (top view), one can see that the membrane/substrate interface relief has changed significantly, which indicates variations in internal mechanical stresses.

First, the critical membrane rupture pressure was determined on the test bench by the method of supplying excess air pressure. The test bench components are described in [8]. Standard deviation (SD) of the critical rupture

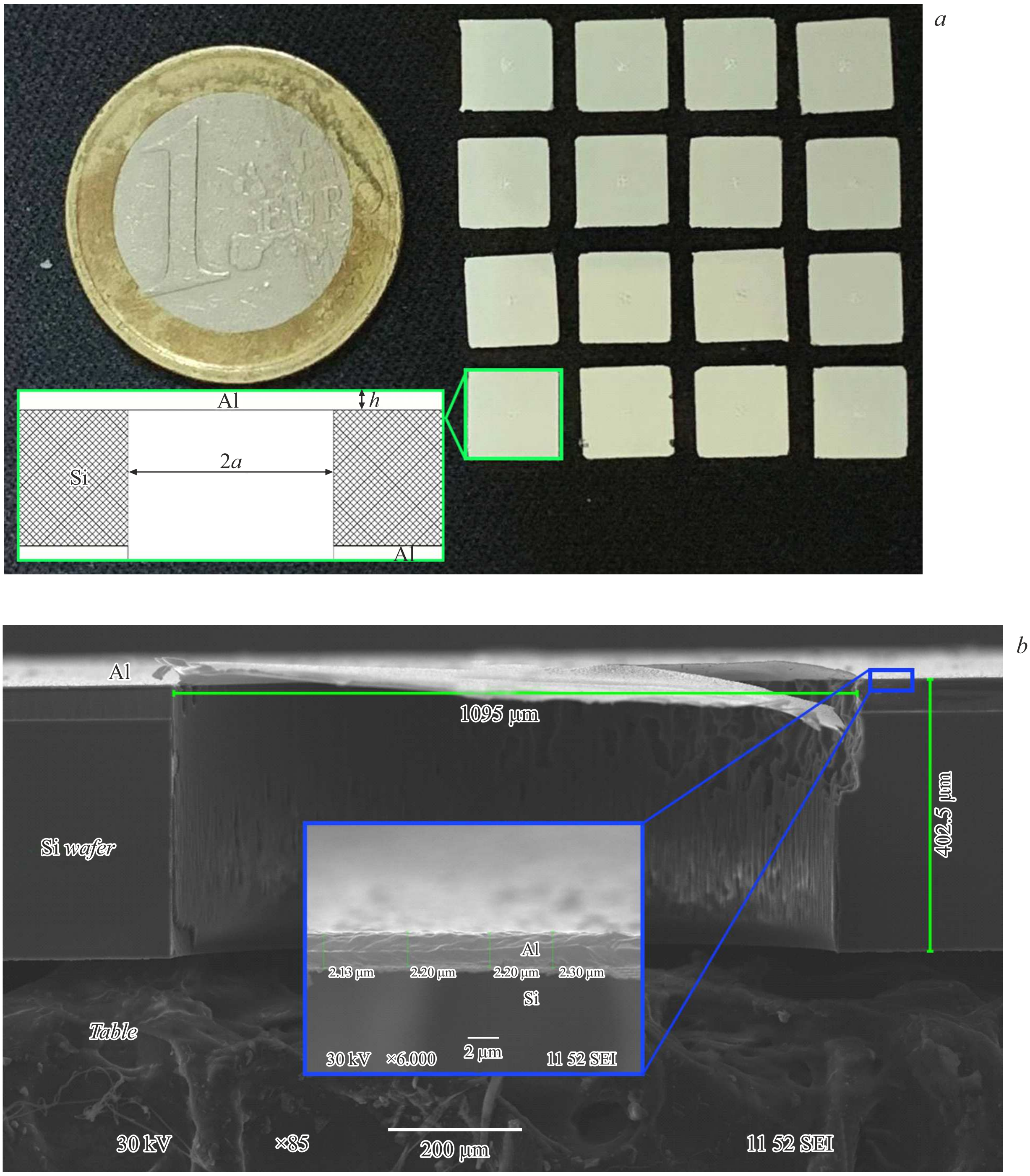

Figure 1. Structures to be studied. *a* — structure schematic diagram (side view); *b* — SEM image of the aluminum film.

pressure P_{cr} was calculated. Then the dependence of the membrane deflection on excess pressure was measured, and the samples were heat-treated in vacuum at $T = 450^\circ$C for 1 h. The selected treatment temperature matched the recrystallization temperature of pure aluminum [13]. After that, measurement and calculation of parameters were repeated (see the Table).

As shown in the Table, thermal treatment makes the membrane material less durable but more reliable with regard to using it in devices based on thin-film membranes (accelerometers, pressure sensors). The source of variations in the mechanical properties is modification of structural properties, namely, the membrane material grain size and roughness. It is known that the grain growth mechanism depends on the temperature and time of exposure [14]. During the heat treatment, grains begin growing starting from the recrystallization temperature; due to this, the inter-grain distance decreases, grain boundary energy decreases,

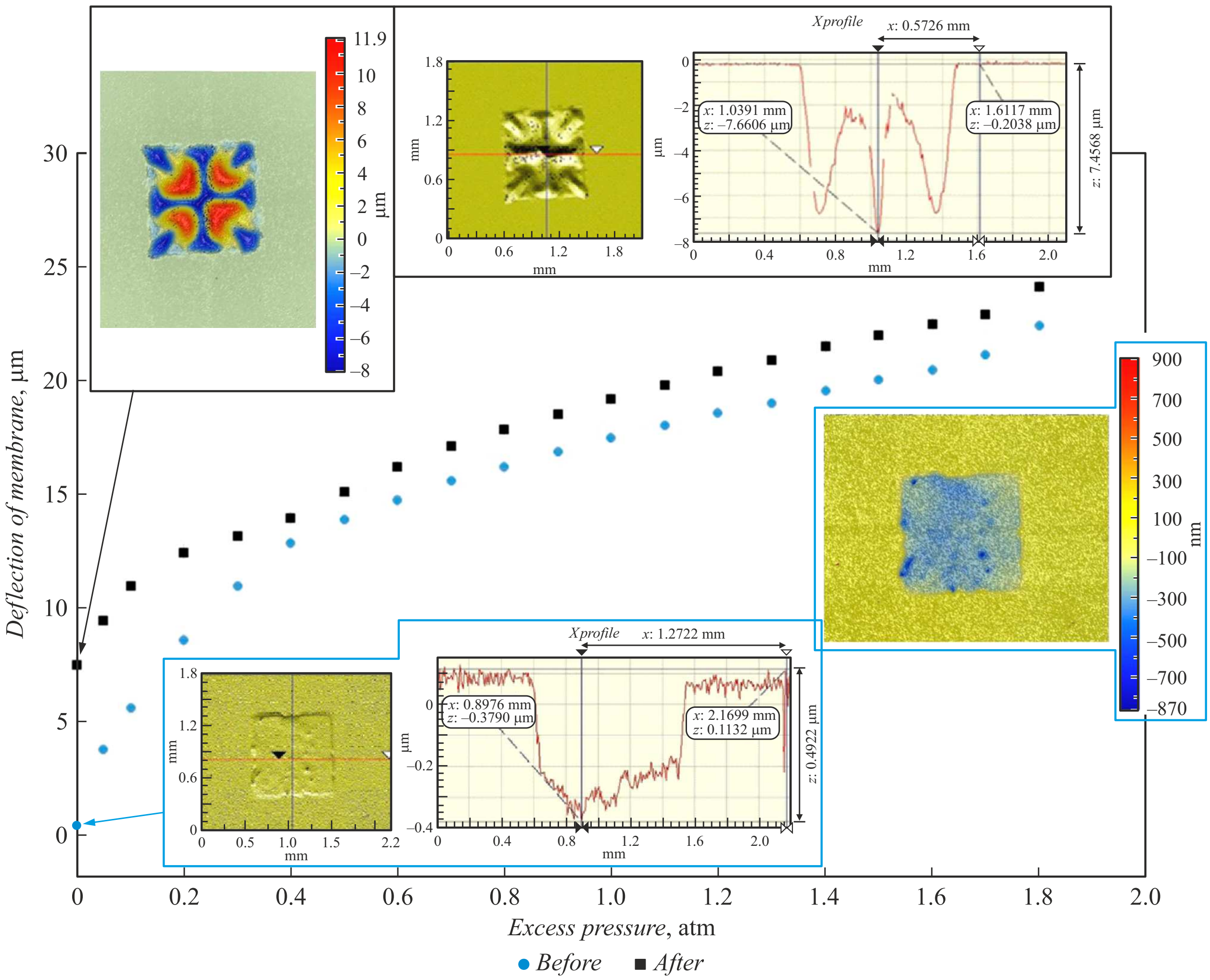

Figure 2. Square membrane deflection versus excess pressure before and after heat treatment.

Variations in the membrane physical and mechanical properties after heat treatment

Parameter	Circle		Pentagon		Square	
	before heat treatment	after heat treatment	before heat treatment	after heat treatment	before heat treatment	after heat treatment
Critical pressure P_{cr}, atm	4.9	4	4.3	3.1	4	2.8
Average variation in the critical rupture pressure, atm	±1.5	±0.9	±0.74	±0.7	±0.78	±0.59
SD P_{cr}	0.751	0.441	0.404	0.332	0.269	0.139
biaxial modulus of elasticity $E/(1-\mu)$, GPa	92	68	101	73	112	85
Grain size, nm	594	1126	535	1024	573	1011
Rouphness, nm	72	4560	80	4485	77	4390

atoms diffuse through the grain boundaries from the area with higher concentration to that with lower concentration, the number of grains decreases because grains merge together, and, finally, the grain height comes up to the film thickness.

The indicated reasons for variations in the membrane material properties after heat treatment allow explaining Fig. 3 that schematically presents the mechanical stress distribution over the membrane and results of analyzing the grain size in the ImageJ program before and after

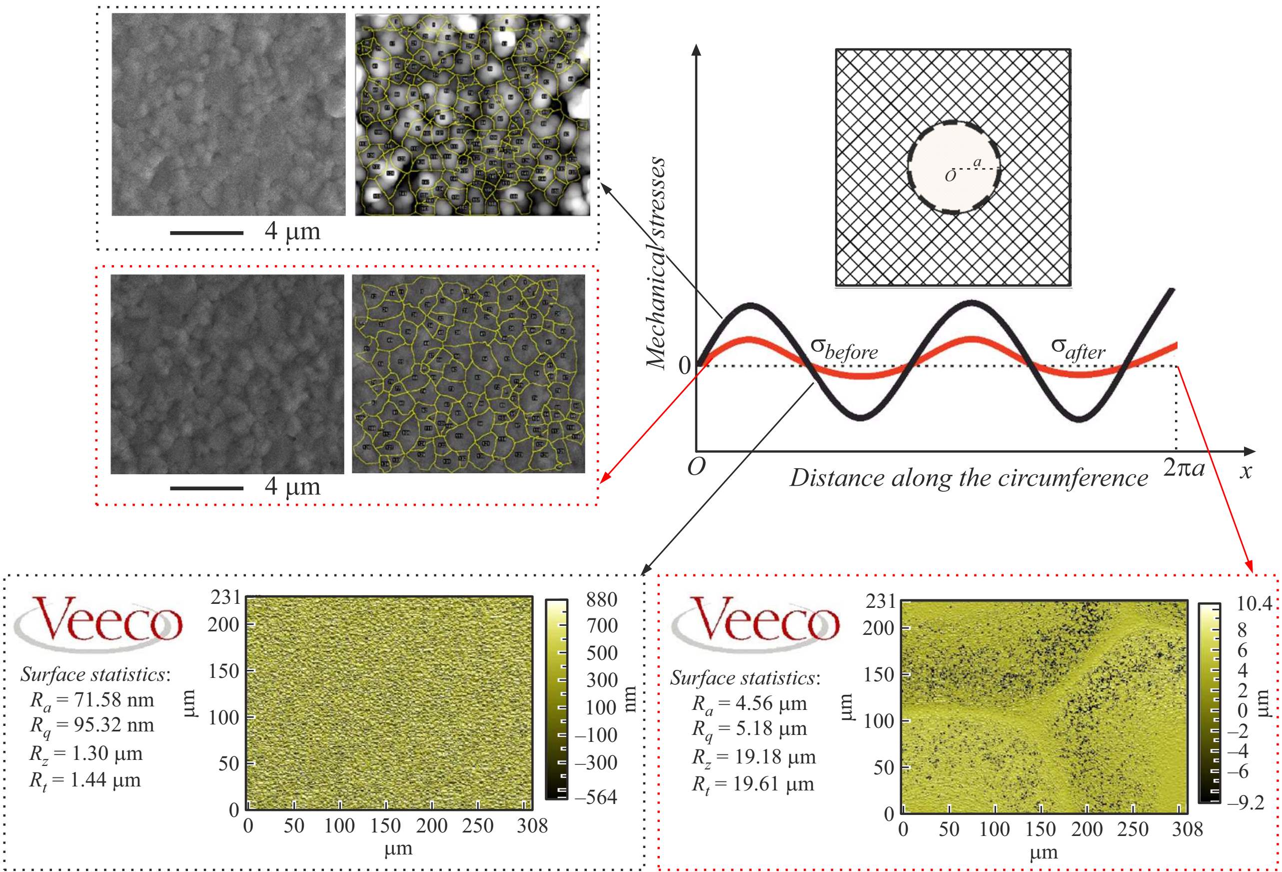

Figure 3. Explanations for the effect of variations in mechanical properties: schematic pattern of the mechanical stress distribution over the membrane before and after heat treatment, results of the grain size analysis in the ImageJ code, measurements of surface roughness before and after heat treatment.

heat treatment; Fig. 3 also illustrates the comparison of data on the aluminum surface roughness before and after heat treatment. The maximum stresses are known to arise at the membrane/substrate interface [3]. The source of the decrease in standard deviation of the critical rupture pressure is the post-treatment decrease in the mechanical stresses spread along the membrane/substrate interface. The decrease in the critical rupture pressure (mechanical strength) may be explained by an increase in grain size according to the direct Hall–Petch relation for Al- films with grain size larger than 50 nm.

The obtained results were compared with data from other publications. In [15], mechanical strength of the circular membrane was 1.64 times higher than that of the square one. Experimental value of $E/(1-\mu)$ for aluminum also correlate well with the results of other researchers. The $E/(1-\mu)$ value is 106 GPa at the Poissonś ratio of the thin-film aluminum material $\mu = 0.34$ and Youngś modulus $E = 70$ GPa [7,10]. The grain size in thin films increases with increasing heat-treatment temperature [16]; another source of the grain size increase is the film thickness increase [9]. Formation of the membrane due to a local cavity in the substrate can significantly enhance the effect of heat treatment even compared to the case of glass substrates [16] because of thermal conductivity of the material and ambient air. In the process of heat treatment, an increase in temperature stimulates an increase in the surface roughness [17].

Funding

The study was supported by the RF Ministry of Science and Higher Education (project № 075-15-2021-1350 of October 5, 2021, internal No. 15.SIN.21.0004).

Conflict of interests

The authors declare that they have no conflict of interests.

References

[1] W. Zhou, J. He, P. Peng, L. Chen, K. Cao, in *Reliability and maintenance. An overview of cases*, ed. by L. Kounis (IntechOpen, 2020). DOI: 10.5772/intechopen.86754

[2] S.J. Kim, H.W. Jung, M.W. Lee, Y.J. Kim, Y.H. Huh, J.H. Park, Mech. Adv. Mater. Struct., **26** (10), 1589 (2018). DOI: 10.1080/15376494.2018.1444217

[3] N.A. Dyuzhev, E.E. Gusev, M.A. Makhiboroda, Mech. Solids, **57** (5), 1044 (2022). DOI: 10.31857/S0572329922050063
[4] J.H. Liu, J.X. Yan, Z.L. Pei, J. Gong, C. Sun, Surf. Coat. Technol., **404**, 126476 (2020). DOI: 10.1016/j.surfcoat.2020.126476
[5] M.G. Mueller, M. Fornabaio, G. Žagar, A. Mortensen, Acta Mater., **105**, 165 (2016). DOI: 10.1016/j.actamat.2015.12.006
[6] K. Nakamura, H. Ohashi, Y. Enta, Y. Kobayashi, Y. Suzuki, M. Suemitsu, H. Nakazawa, Thin Solid Films, **736**, 138923 (2021). DOI: 10.1016/j.tsf.2021.138923
[7] N.A. Dyuzhev, E.E. Gusev, E.O. Portnova, M.A. Makhiboroda, Mech. Solids, **59** (1), 20 (2024). DOI: 10.1134/S0025654423601040
[8] N.A. Djuzhev, E.E. Gusev, I.V. Kushnarev, M.A. Makhiboroda, D.A. Dobrokhotov, V.A. Bespavol, Tech. Phys. Lett., **50** (5), 8 (2024). DOI: 10.61011/TPL.2024.05.58412.19833.
[9] S. Du, Y. Li, Adv. Mater. Sci. Eng., first published: 09 March 2015. DOI: 10.1155/2015/969580
[10] Y.Y. Lim, M. Chaudhri, Y. Enomoto, J. Mater. Res., **14** (6), 2314 (1999). DOI: 10.1557/JMR.1999.0308
[11] M. Laleh, E. Sadeghi, R.I. Revilla, Q. Chao, N. Haghdadi, A.E. Hughes, W. Xu, I. De Graeve, M. Qian, I. Gibson, M.Y. Tan, Prog. Mater. Sci., **133**, 101051 (2023). DOI: 10.1016/j.pmatsci.2022.101051, 2022
[12] Y. Yang, S. Zhao, J. Gong, X. Jiang, C. Sun, J. Mater. Sci. Technol., **27** (5), 385 (2011). DOI: 10.1016/S1005-0302(11)60079-0
[13] Y. Zhao, L. Li, Z. Lu, G. Teng, S. Liu, Z. Hu, A. He, Mater. Res. Express, **8** (4), 046515 (2021). DOI: 10.1088/2053-1591/abf3e3
[14] Z. Huda, M. Saufi, Shaifulazuar, J. Ind. Technol., **15** (2), 127 (2006).
[15] A. Berns, U. Buder, E. Obermeier, A. Wolter, A. Leder, Sensors Actuators A, **132** (1), 104 (2006). DOI: 10.1016/j.sna.2006.04.056
[16] K.M. Wibowo, M.Z. Sahdan, M.T. Asmah, H. Saim, F. Adriyanto, Suyitno, S. Hadi, IOP Conf. Ser.: Mater. Sci. Eng., **226**, 012180 (2017). DOI: 10.1088/1757-899X/226/1/012180
[17] B.N.K. Reddy, N.K. Udayashankar, Surf. Interfaces, **5**, 62 (2016). DOI: 10.1016/j.surfin.2016.09.007

Translated by EgoTranslating