Thin Solid Films 604 (2016) 85–89

Contents lists available at ScienceDirect

Thin Solid Films

journal homepage: www.elsevier.com/locate/tsf

Elastic properties of nanolaminar Cr_2AlC films and beams determined by in-situ scanning electron microscope bending tests

Rolf Grieseler [a,*], Felix Theska [a], Thomas Stürzel [a], Bernd Hähnlein [b], Mike Stubenrauch [c], Marcus Hopfeld [a], Thomas Kups [a], Jörg Pezoldt [b], Peter Schaaf [a]

[a] Chair Materials for Electronics, Institute of Materials Engineering and Institute of Micro and Nanotechnologies MacroNano®, TU Ilmenau, Gustav-Kirchhoff-Str. 5, 98693 Ilmenau, Germany
[b] Chair Nanotechnology, TU Ilmenau, Institute of Micro and Nanotechnologies MacroNano®, Gustav-Kirchhoff-Str. 7, 98693 Ilmenau, Germany
[c] Chair Biomechatronics, TU Ilmenau, Institute of Micro and Nanotechnologies MacroNano®, Max-Planck-Ring 12, 98693 Ilmenau, Germany

ARTICLE INFO

Article history:
Received 17 September 2015
Received in revised form 4 March 2016
Accepted 11 March 2016
Available online 14 March 2016

Keywords:
MAX phase
Cr_2AlC
In-situ testing
Thin film
MEMS
Young's modulus

ABSTRACT

The mechanical properties of Cr_2AlC MAX phase structures were investigated by in-situ bending tests. Freestanding structures such as cantilevers and doubly clamped beams of Cr_2AlC were produced. The structures exhibit a Young's modulus of 184 GPa which is close to the value obtained by vibrational measurements. The in-situ bending test allows the determination of the mechanical properties with a lower variance of the measurement results compared to the vibrational measurement. The results are a good starting point for the development of microelectromechanical structures based on MAX phases.

1. Introduction

For the application of materials in microelectromechanical systems (MEMS), the knowledge of their mechanical properties is indispensable. Commonly, the mechanical properties of thin films as the starting point for the MEMS production are well known [1–4] and studied by a variety of methods such as X-ray diffraction (XRD) [5–7] or other types of radiation [8] as well as mechanical testing, e.g. the determination of the wafer bow [9] or nano-indentation [10,11].

More challenging is the determination of the mechanical properties such as intrinsic stress or the Young's modulus of MEMS structures as they are commonly different from the thin film the MEMS structures are made of. A typical approach is the use of indicator structures showing the presence of internal stress [12,13]. A typical method to determine the mechanical properties is to cut cantilevers out of the surface of the investigated material using a Focused Ion Beam (FIB) and subsequently to determine the bending behavior of these cantilevers in-situ [14–16] to simulate the mechanical behavior of a MEMS structure. In order to determine the Young's modulus, the vibrational properties of cantilevers and beams can be used measuring their Eigenmodes [17–19]. Furthermore, a bending test performed on existing MEMS structures is a common method of identifying the mechanical properties within certain limitations [20–22].

In this work, the mechanical properties of nanolaminar $M_{n+1}AX_n$ (short: MAX, with commonly $n = 1; 2; 3$) microstructures are investigated. MAX phases are an interesting material class, as they show good mechanical, thermal and chemical stability comparable to ceramics as well as good electrical conductivity comparable to metals [23–26]. Therefore, MAX phases can be a suitable material for applications in harsh environments and at elevated temperatures as conductors, connectors or other microstructural devices. The prototype of the 211 MAX phase is Cr_2AlC. There are already investigations regarding this MAX phase, both for Bulk as well as for thin film materials. The most common way is the determination of the Young's modulus using the indentation modulus [27,28]. Typically MAX phase thin films are produced using plasma based processes such as magnetron sputtering from the compound target [27,29,30] or from elemental targets at higher temperatures [31,32]. Among others MAX phase thin films could also be obtained depositing multilayers of the compound's elements and subsequent annealing [18,33,34]. First investigations using other MAX phases such as Ti_2AlC were carried out using microcantilevers produced by Focused Ion Beam sputter etching from bulk material [35–37]. In this work an alternative nondestructive determination method of the mechanical properties of thin film materials directly in the fabricated microelectromechanical structures is demonstrated. It is based by combining micromechanical systems

---

* Corresponding author.
E-mail address: rolf.grieseler@tu-ilmenau.de (R. Grieseler).

http://dx.doi.org/10.1016/j.tsf.2016.03.026
0040-6090/

processing and in-situ micromanipulator testing. The investigated beam structures can afterwards be used as indicators for internal stresses resulting from the production process by heat treatment or even a gradient of intrinsic properties of the materials due to the multilayer character of the precursor thin film. Tensional stresses for example can lead to an optically interpretable bending of the beams [12,38].

2. Experiments

First, MAX phase thin films of Cr_2AlC were prepared using magnetron sputtering of multilayers, each up to 10 nm as maximum, from elemental targets as described in [18]. Prior to that deposition, a 50 nm dry thermal silicon oxide barrier was grown on the silicon substrate. This diffusion layer suppresses the interaction between the silicon and the deposited multilayer system during phase formation by heat treatment. The multilayer system was subsequently annealed at 650 °C for 30 s using a rapid thermal processing system (RTP, Jet First, Joint Industrial Processors for Electronics) with a heating rate of 15 K/s in argon atmosphere. This process forms a MAX phase thin film with a total thickness of approximately 500 nm. The phase formation was confirmed by X-ray diffraction (XRD) and transmission electron microscopy (TEM), both not shown in this paper. From these thin films, doubly clamped beams as well as cantilevers were prepared using a standard lithography as also demonstrated in [18]. The final freestanding structures are shown in Fig. 1.

The mechanical behavior was investigated using a crossbeam SEM/FIB device (Auriga 60, Zeiss) equipped with a piezo driven three-axes micromanipulator (Kleindiek® MMA3-EM). The measurement was carried out close to the middle of the beams width as well as on the half-length of the cantilever. To estimate the maximum displacement of the structures a finite element simulation was applied as

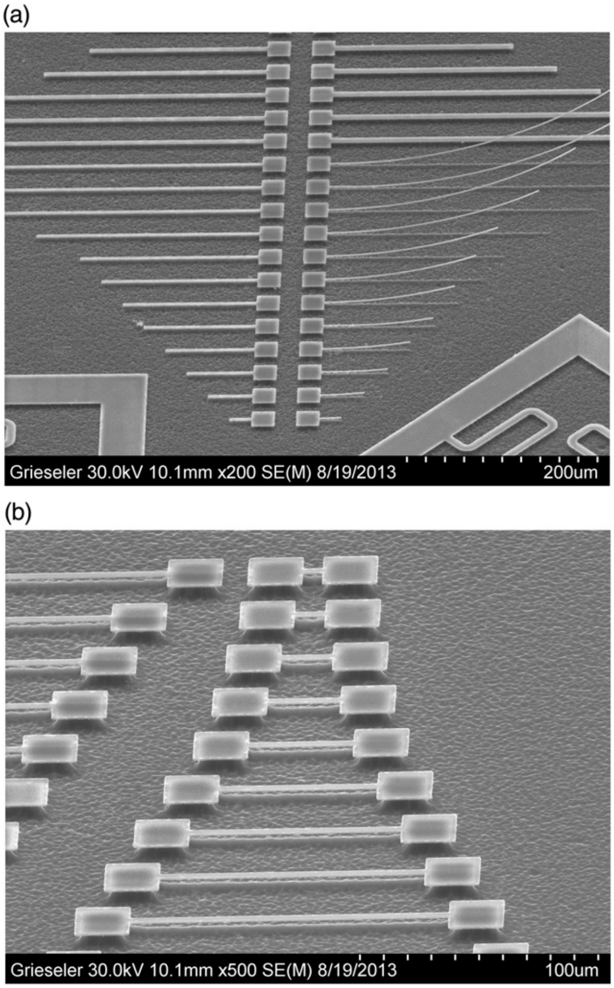

Fig. 1. Freestanding doubly clamped cantilevers a) and beams b) made of Cr_2AlC from Cr–C–Al multilayers annealed at 650 °C for 30 s with a heating rate of 15 K/s.

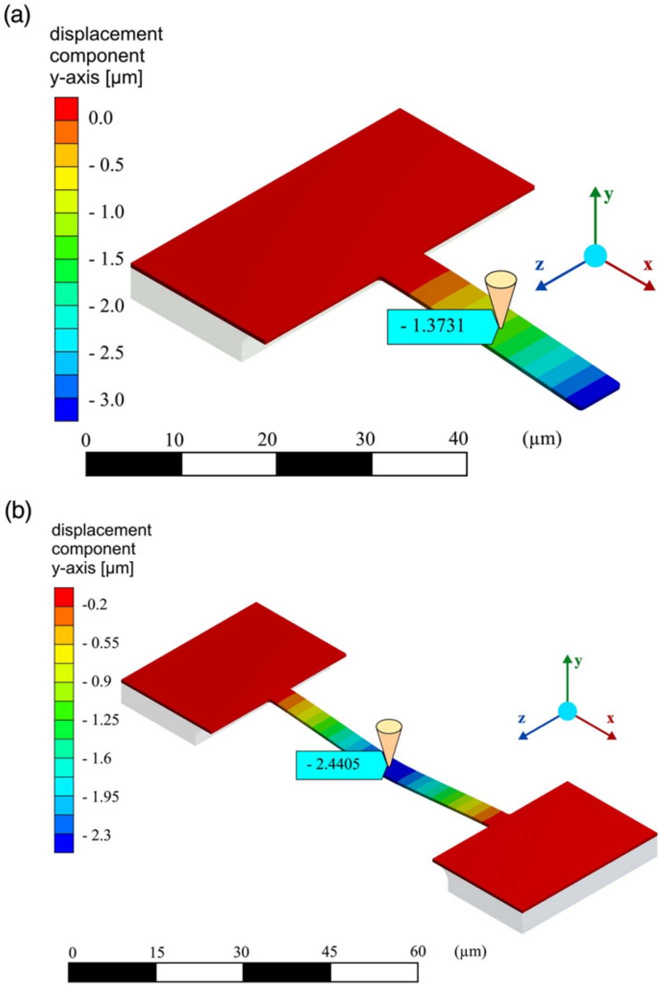

Fig. 2. Maximum displacement component in y-axis of a) a cantilever and b) a doubly clamped beam as simulated using the finite element method.

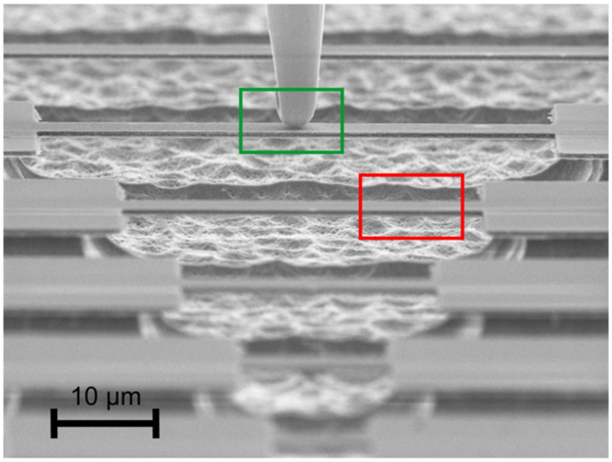

Fig. 3. Setup for the bending test at the center of a doubly clamped beams. The green square represents the displacement measurement frame for the beam and the sample. Whereas, the red square represents only the sample displaced reference frame.