Qualitative and Quantitative analysis of Ti_2AlC MAX-phase films by Electron Spectroscopic Imaging Series

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Keywords: Ti_2AlC, Max phases, electron spectroscopic imaging

Electron spectroscopic imaging (ESI) series are a powerful tool to perform a qualitative and quantitative analysis in nanometer scale investigations. This method has several advantages compared to other techniques. It has a shorter acquisition time than in the STEM EELS, it can record information simultaneously over large areas and the energy-loss spectra can subsequently be extracted for any given area in the images [1,2].

Ti_2AlC belongs to the so called of MAX-phases which have promising features because they combine unique properties which usually found in either metals or ceramics [3-5]. A high impurities single bulk materials (impurities $\leq$ 4 vol pct) of Ti_2AlC has successfully been synthesized. Therefore a number of bulk properties are known for this phase [6]. The deposition of Ti_2AlC thin film was reported by Wilhemsson et.all [7] and Walter et.all [8].

In this work, a Ti_2AlC film was deposited by magnetron sputtering depositions from segmented compounds target. The phase identifications using X-ray diffraction (XRD) shows that the deposited film consists mainly of the Ti_2AlC phase, with a small amount of TiC. STEM HAADF images show that there is a porous layer above the interfaces. Additionally STEM HAADF line scans show the concentration gradient for titanium and aluminum near the porous area (figure 1.a and b). EFTEM elemental maps indicate carbon enrichment on the porous areas (figure 1.c-f). Since the investigated areas are very small, the ESI series technique was applied to qualitatively and quantitatively identify the existing phases in the porous area.

Spectra of the near-edge structure (ELNES) of the carbon K edge were extracted from series of spectrum images taken from areas near the pores and also other parts of the film. The spectra show a different ELNES for two different areas (figure 2.a and b), which indicates both areas consist of different phases. Carbon-K edge ELNES spectra of TiC (2.c) and Ti_2AlC (2.d) obtained by TEM imaging mode also show a good agreement with extracted spectra from ESI series.

We also took ESI series in the energy loss range between the carbon-K and titanium-L edges. From these spectra, the concentration ratio between Ti and C was determined. The results of quantitative analysis of the Ti/C ratio for another part of the sample is twice larger than the area near the pores, indicating that Ti_2AlC and TiC coexist in these regions.

This result not only shows both quantitatively and quantitatively analysis of Ti_2AlC film can be done by a series of electron spectroscopic imaging but also leads to understanding of the deposition process of the film.

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9. Support of these investigations by the DAAD and the DFG is gratefully acknowledged.

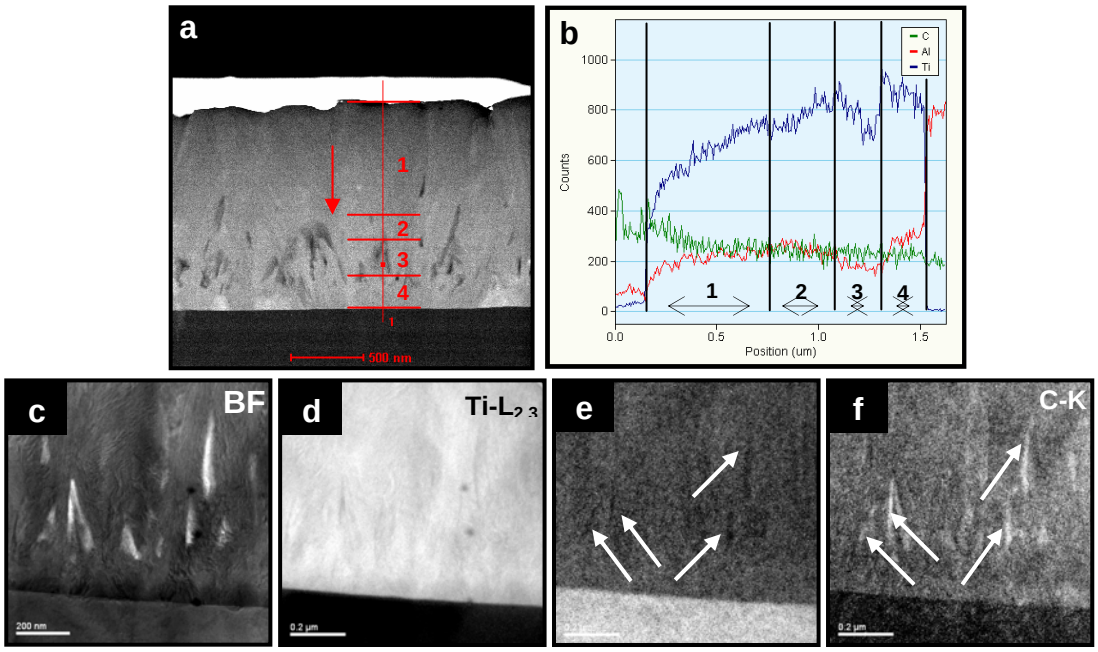

Figure 1. (a) STEM HAADF line scan and (b) intensity profile spectra taken from top of the film to the substrate. The spectrum shows the variation of the intensity of titanium and aluminum divides the film onto four areas. (c) EFTEM bright field image and (d-f), which shows the enrichment of the carbon contents in the pores.

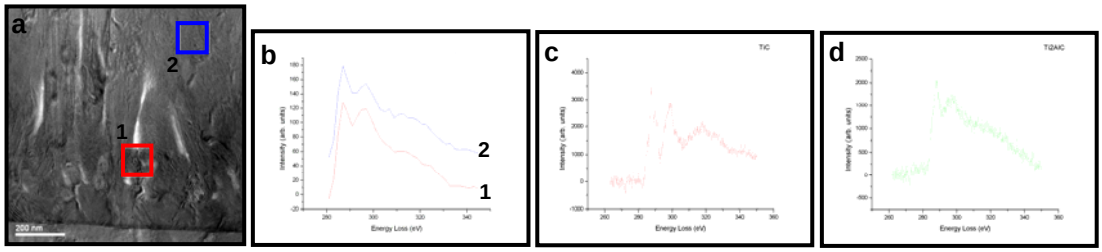

Figure 2. (a) Bright field image of two areas from which spectrum imaging series were taken. (b) Spectra which were extracted from the series of 36 ESI images with ∂E=2 eV and an increment of 2 eV by integrating over the rectangular areas indicated in (a). ELNES carbon K edge of (c) TiC and (d) Ti_2AlC, which were acquired in the EELS imaging mode, show a good agreement with spectra extracted from ESI series.