TEM investigations of thin films of ($M_{n-1}AX_n$)-phases deposited onto Si, MgO and stainless steel substrates

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$M_{n+1}AX_n$ (MAX) phases are a group of ternary carbides or nitrides with M being an early transition metal (mainly of the groups IVB and VB), A being an A group element (mostly IIIA and IVA) and X being either carbon or nitrogen. The prototypic compound Ti_3SiC_2 had already been synthesized in the 1960s [1], but not until 1996 its remarkable properties have been discovered [2]. MAX phases combine metallic properties such as good electrical and thermal conductivity with ceramic properties like thermal stability and resistance against oxidation [3]. These properties give rise to a potential application of MAX phases in thin films – for instance as wear and corrosion resistant coatings of electrical contacts. The growth of crystalline MAX phase thin films however is not trivial. The deposition of such films by magnetron sputtering has been reported [4-6]. One work concerning the succesful pulsed laser deposition of MAX phase thin films has been published by Hu et al. [7] but was argued over in the scientific community [8,9]. Another work describing the pulsed laser deposition from a Ti_3SiC_2 MAX phase target only reported the formation of crystalline TiC_x films with incorporated amorphous silicon [10].

In this work, pulsed laser deposition with a Quantel Brilliant Nd:YAG laser (λ = 1064 nm, τ = 6 ns, f = 20 Hz, Φ = 6 J/cm^2) was used to grow thin films from pre-synthesized Ti_3SiC_2- and Cr_2AlC-MAX-phase formulated ablation targets on Si(100), oxidized Si(100), MgO(100) and stainless steel substrates with and without a 200 V Argon ion beam directed at the substrate surface during deposition. The depositions were carried out in a substrate temperature range from room temperature to 650 °C at a chamber pressure of 10^{-5} Pa without ion beam and at 10^{-2} Pa Argon background pressure when the ion beam was applied.

Transmission electron microscopy investigations including high resolution, electron diffraction and chemical analysis techniques were performed to identify phase separations and interdiffusion between substrate and MAX-layer and the content and distribution of crystalline and amorphous phases in the layer.

Figure 1 shows a high resolution TEM (HRTEM) image and an electron diffraction pattern of a Ti_3SiC_2-MAX-phase layer on along [100] oriented MgO Substrate. The layer is polycrystalline but textured and partly amorphous. The crystalline particles grow in column-like structures.

Samples based on Cr_2AlC as MAX-phase show at the interface between the substrate and the layer an additional phase with a pyramidal shape, as found in Figure 2. EDX investigations show a higher Al content at interface region then in the layer. The lattice plane distances measured by electron diffraction patterns and by Fast Fourier Transformation of HRTEM images fit to the Cr_2Al phase.

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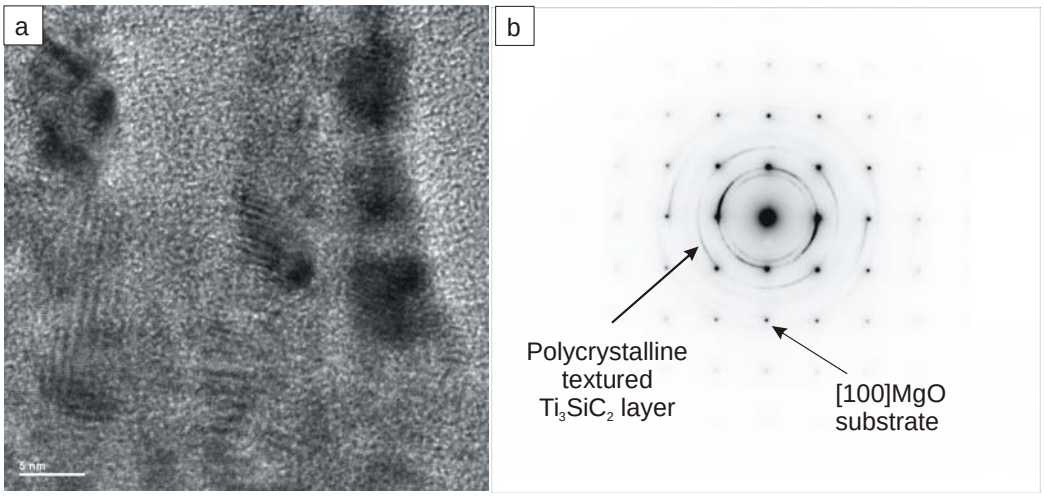

Figure 1. HRTEM image (a) and electron diffraction pattern (b) of a polycrystalline and textured Ti_3SiC_2-MAX-phase layer on [100] MgO Substrate

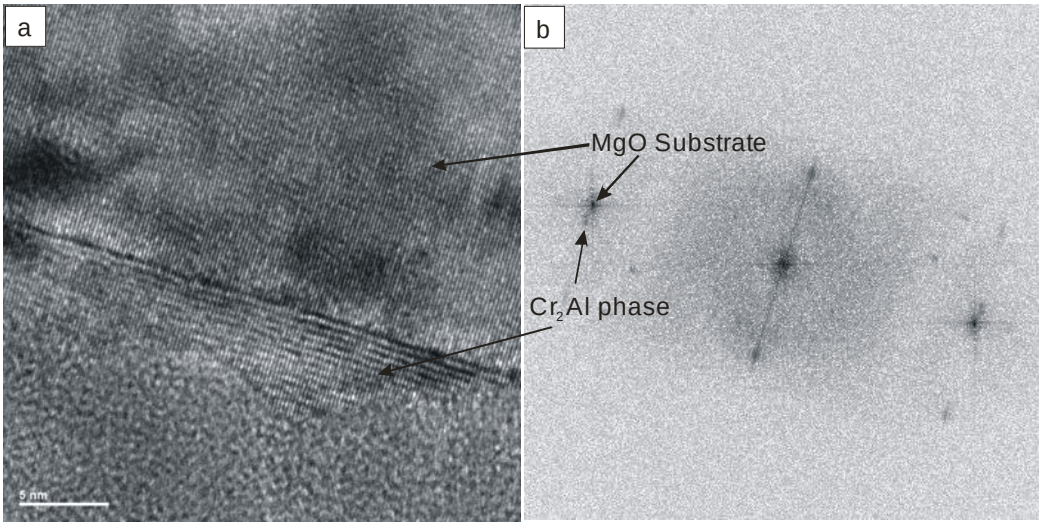

Figure 2. HRTEM image (a) and FFT (b) of the additional phase at the interface between the Cr_2AlC based layer and the MgO substrate which was identified as Cr_2Al.

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