Single-sample combinatory for composition dependent TEM and nanoindentation studies of Al-Mg binary films

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Our aim was to effectively study the concentration dependent properties of two component thin layer systems. We worked out a high throughput method, the so called micro-combinatory [1]. It is based on the "one-sample concept" i.e. a single gradient sample may condense the whole binary system of components A and B with the composition A_xB_{1-x} (x=0...1) and geometry that adapts to the current analytical measurement technique eg. TEM [2]. The all-in-one feature is very efficient; a single sample is required to handle, no need for laborious preparation, replacement and study of a series of TEM specimens. A specific advantage of μ-combinatory is that phases of various compositions are formed and investigated side by side, in a single TEM grid that provides superior reproducibility and a straight comparison. Furthermore, the consistency, and known concentration profile of μ-combinatorial samples are an invitation to high throughput automated TEM.

The deposition of gradient samples is implemented by moving a narrow slot over the TEM grid meanwhile the power of the magnetron sources A and B are regulated in sync with the movement of the slot. The outline of μ-combinatorial deposition and a Mg-Al TEM sample with a composition gradient of 15 μm/% are shown in Fig.1 a and b, respectively.

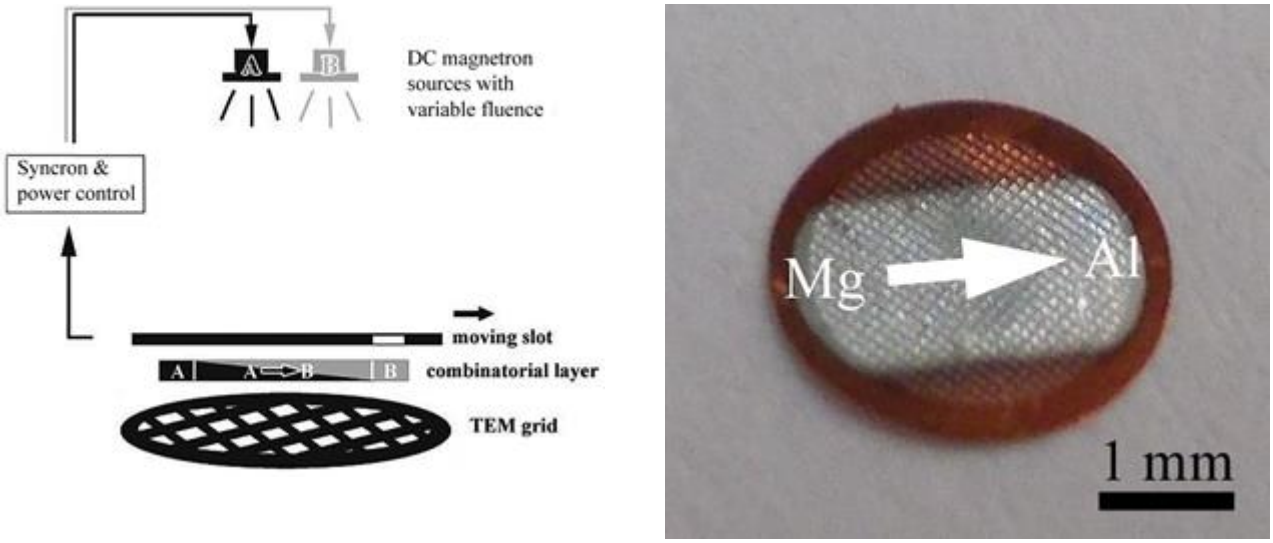

Figure 1. (a) Set-up of micro-combinatorial deposition for TEM samples. For nanoindentation the sample size is scaled up to 25mm. (b) Photo of a combinatorial Mg-Al sample on a 300-mesh TEM grid that exhibits a 1.5 mm long gradient section embedded between pure Al and Mg domains.

Research of thin Al-Mg is strongly motivated by technological applications and understanding of the mechanical properties. For both a demonstration of the effectiveness of the method and the technological importance gradient Al-Mg binary films were studied by TEM and nanoindentation and this revealed the correlations of composition, structure and mechanical properties.

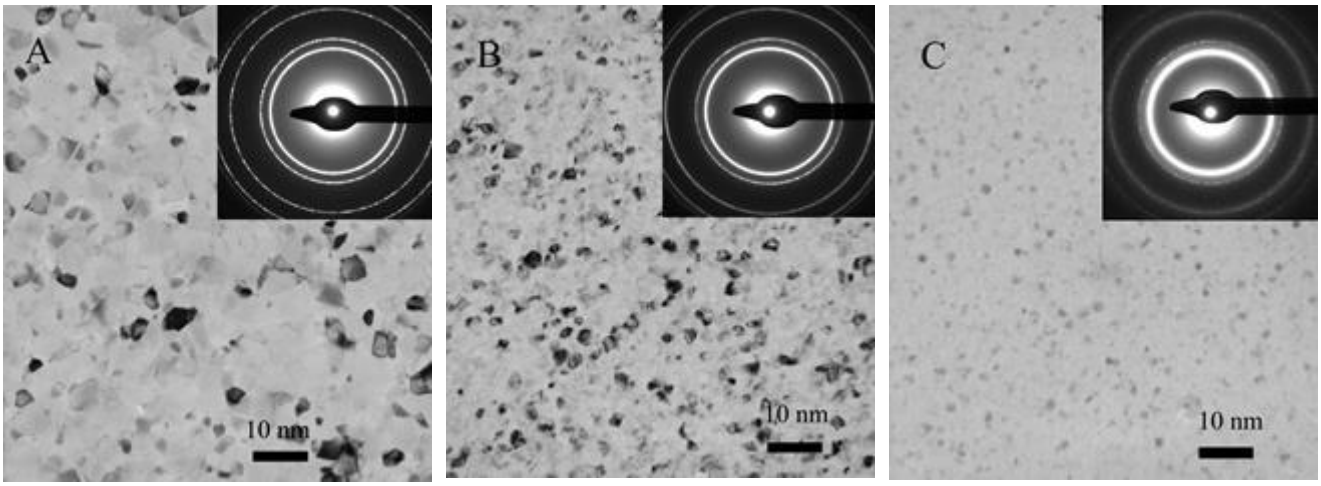

Figure 2. BF TEM images with SAED insets of (a) 1%, (b) 10% and (c) 30% Mg/Al showing remarkably different grain size and structure revealed from a single μ-combinatorial sample.

The exploration of the concentration dependent structure of Al-Mg contributes to the understanding of the deformation mechanism and the fine tuning of the mechanical properties. The present study focuses on the 1...30% Mg composition range. Fig.2 a, b and c shows, for instance, the significant difference in microstructures of Al-Mg containing 1, 10 and 30% Mg, respectively. Nanoindentation measurements have revealed that similarly to the differences in microstructure, the nanohardness and the deformation mechanism are also totally different at the three compositions.

The micro-combinatorial method is developed in a cooperation of RITP and Holocom.

Acknowledgements

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References

[1] G. Sáfrán Hung. Patent, P 15 00500 (2015)

[2] G. Sáfrán, "One-sample concept" micro-combinatory for high throughput TEM of binary films

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