

## In-situ Observations of Self-Propagating High-Temperature Synthesis of AlCo Multi-layer Foils by Dynamic Transmission Electron Microscopy

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The dynamic transmission electron microscope (DTEM) combines the high spatial resolution of a conventional TEM with nano-second temporal resolution (Figure 1). [1,2]. This unique combination of spatial and temporal resolution enables us to observe the reaction front of self-propagating high-temperature synthesis (SHS) reactions in reactive multilayer foils (RMLFs) [3]. Although these reactions are irreversible, the single-shot imaging capability of the DTEM allows their progress to be recorded with 15ns electron pulses at various times after reaction initiation to elucidate the structural transformations taking place during the reaction [3].

Intermetallic compounds, especially metal aluminides show potential for many structural applications due to their favorable physical and mechanical properties [4]. The SHS reaction is a promising technique for processing these materials with tunable properties at relatively low cost, and nanolaminates in the metal aluminide systems that have a high exothermic enthalpy of formation, are good candidates for SHS [5]. Many theories have been proposed for quantifying SHS reactions, e.g., determining reaction kinetics, the phase sequence, and initiation temperature, yet direct observation of the reactions on the nano-scale and with the required temporal resolution have been extremely limited [6].

Here we have observed the SHS reaction of equiatomic Al-Co nanolaminates. The 160nm-thick foil was comprised of 8 bilayers of 20nm. The reaction was initiated inside the DTEM using a single, 10ns laser pulse with a wavelength of 532nm and a fluence of 200 mJ cm<sup>-2</sup>. The absorbed laser energy heats the multilayer stack and induces interdiffusion of Al and Co layers in the center of laser-irradiated area (~50 μm in diameter). The local temperature at the reaction front rises due to the heat of mixing and the formation of intermetallic CoAl (B2) phase, which stimulates interdiffusion of the layers in the surrounding regions and propagation of the reaction [6]. The reaction zone propagated to a diameter of approximately 200 microns before quenching itself, indicating that the reaction is not self-sustaining and requires both the heat from the absorbed laser light and heat from mixing of the layers to propagate the reaction. At distances exceeding twice the 1/e<sup>2</sup> diameter of the laser spot (~95μm), the reaction stalls due to the lower local temperatures and diminished interdiffusion of the layers.

Figure 2 shows a sequence in the reaction taken at several time intervals with each image representing a 15ns snapshot in time. The initial velocity of the propagating reaction zone is estimated to be 15 ± 2 m/s. Figure 2b shows the propagating reaction 1.7μs after initiation. The laser hot spot is approximately 30 microns away from the reaction front, and out of view. Figure

2c shows the reacted foil. The largest grains appear closest the initiation site, in the lower left of the image. The foils are imaged in plan view and the contrast in the reaction front in 2b is mass-thickness contrast caused by mass diffusion of the foil constituents. The thickness modulations are short-lived, spanning about 4-6 microns and can be seen to disappear quickly in Figure 2b (lower left corner) as larger grains nucleate and grow. These results, along with other studies of foils with different compositions and layer thicknesses provide an overall view of the reaction process and will be described in detail in this presentation [7].

#### References:

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- [7] This work was performed under the auspices of the U.S. Department of Energy, Office of Basic Energy Sciences, Division of Materials Sciences and Engineering, under contract No. DE-AC52-07NA27344 under the auspices of the U.S. Department of Energy, Office of Basic Energy Sciences, Division of Materials Sciences and Engineering, under contract No. DE-AC52-07NA27344.

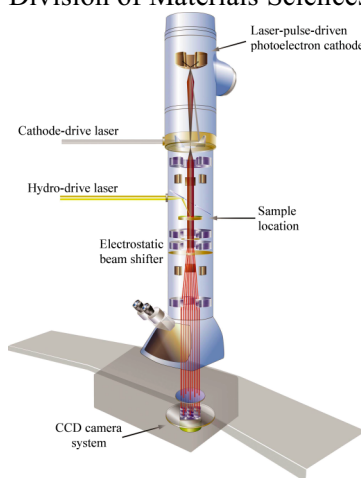


Figure 1. Schematic representation of the DTEM

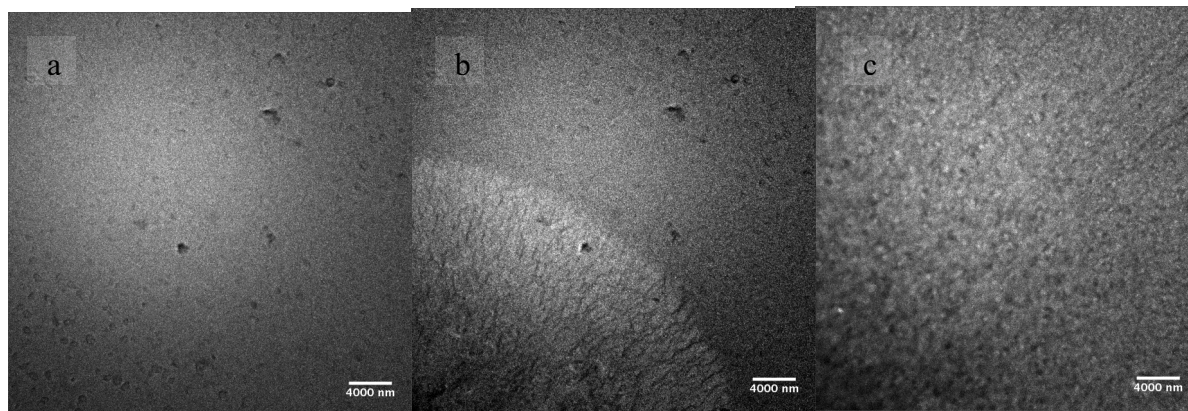


Figure 2: Propagation of the SHS reaction front in AlCo nanolaminates. a) before reaction b) 1.7  $\mu$ s after initiation c) after reaction completion