

A Comparative Study of Conventional TEM and FIB Sample Preparation Techniques for High-Entropy Alloys

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High-entropy alloys (HEAs) have emerged as a transformative class of materials in the field of materials science and engineering. Unlike conventional alloys, which are typically based on one or two principal elements, HEAs are composed of multiple principal elements [1]. Since their introduction just over a decade ago, HEAs have attracted significant attention due to their exceptional mechanical strength, thermal stability, and corrosion resistance. The complexity of their microstructures offers unique challenges and opportunities for fundamental research and practical applications [2]. This study presents a comparative evaluation of conventional Transmission Electron Microscopy (TEM) sample preparation and Focused Ion Beam (FIB) sample preparation methods for HEAs, highlighting their respective advantages, limitations, and impact on microstructural interpretation [3].

In this study, a comprehensive approach to TEM sample preparation for HEAs is presented. A comparative analysis was conducted between conventional preparation techniques and FIB lamella preparation using a Helios 5UC. Conventional TEM samples were prepared using a Precision Diamond Wire Saw, Gatan Disc Grinder Model 623, Dimple Grinder Model 656, and subsequently thinned using a Precision Ion Polishing System (PIPS). An additional variant of the conventional method incorporated a final low-energy ion milling step using a Model 1040 NanoMill, aimed at improving surface quality and reducing damage layers. For comparison, FIB TEM lamella was prepared using a semi – automated process on the Helios 5UC system, followed by final thinning with the NanoMill to achieve electron transparency and minimize Ga⁺ ion damage. To evaluate and compare four different preparation routes: conventional cross section, conventional cross section with NanoMill, FIB lamella, and FIB with NanoMill. Sample thickness and quality were assessed using the Scanning Transmission Electron Microscopy detector (STEM) on the Helios 5UC and imaged in a Jeol JEM - 2100 TEM. The primary objective of this work was to identify the most effective sample preparation method for accurate microstructural characterization of HEAs.

Key words: TEM, TEM sample preparation, Electron Microscopy, HEAs, FIB, Nanomill

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