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A Novel Technique for *In-Situ* TEM Characterization of Precipitates in Alloys

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Thickness of specimen preparation is particularly crucial to TEM investigation of materials. Research on precipitates of alloys is numerous. However, results of selected area electron diffraction (SAD) and energy-dispersive X-ray spectroscopy (EDS) have often been ambiguous especially for precipitates in nano-size due to the limitation of selected aperture size and the electron beam – specimen interaction volume. To obtain accurate and reliable results of precipitates in alloys, the ideal method is to isolate precipitates from the substrate/matrix. Precipitates in alloys have been investigated by replica extraction technique [1]. However, precipitate extraction by etching has been difficult due to chemical selection and low success rate. This study presents a novel technique to isolate precipitates from other phases or substrate for accurate analysis while investigating the sample in TEM.

TEM thin specimen of $Al_xCoCrFeNi$ (x=0.5, 0.7) alloy was prepared by conventional mechanical grinding/polishing, and followed by Ar^+ ion milling. A little amount of wax or paraffin was dissolved in acetone, e.g., 2 g wax in 100 mL acetone. The final ready-for-observation TEM specimen was dipped into the solution for a few seconds, and then air dried (Fig. 1(a)). The treated specimen was annealed in a heating holder in the TEM (such as the case in this study) [2, 3]. The wax or paraffin formed a thin amorphous film wrapping around the entire TEM specimen after annealing (Fig. 1(b)). Some of the precipitates separated from the matured solid solution (SS) during annealing were preserved in the thin amorphous film and isolated from substrate (Fig. 1(c)). The precipitates containing thin amorphous or paraffin film were thus investigated by an analytical TEM. In this study, paraffin coated $Al_xCoCrFeNi$ alloy thin specimen was first loaded in a Gatan heating holder (Model 652) at room temperature for TEM study. It was then heated at 900 °C for 10 min. for precipitate investigation.

TEM images of conventionally prepared specimen ($Al_{0.5}CoCrFeNi$ alloy), as shown in Figure 2, show typical large precipitates. SAD reveals a rather complex pattern due to additional diffraction information contributed from substrates surrounding the precipitate (Fig. 2(d)). Although the basic spots can be assigned to b.c.c or B2 structure, results from the dynamic diffraction show streaky patterns, making it difficult to resolve this complex eD pattern. In contrast, TEM micrographs of high entropy alloy $Al_{0.7}CoCrFeNi$ after *in-situ* annealing at 900 °C for 10 min depicts that the precipitates formed during annealing were embedded in the very thin paraffin/amorphous film (Fig. 3 (a)). The area enclosed by dotted lines reveals the newly formed carbonaceous amorphous film extended from the specimen edge. Despite the larger SA aperture size, eD pattern obtained from the single precipitate can be analyzed without ambiguity. Precipitates can be assigned correctly by combining EDS results of those precipitates (Fig. 4) with eD data, e.g., P1 f.c.c. [101] is a Ni-rich SS whereas P2 [1-10] is CrFe-based SS. This innovative method not only allows for investigation of high entropy materials and precipitates with *in-situ* TEM at different temperatures; the wax/paraffin coated TEM specimen can also be cleaned by acetone or plasma and reexamined with TEM [4].

References:

[1] P.J. Goodhew, Specimen Prep. in Materials Science, (North-Holland/Am. Elsevier, 1973), 180 p.

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[2] JC Rao *et al*, Acta Materialia. **131** (2017), p. 206;
[3] JC Rao *et al*, Materials Letters **176** (2016), p. 29.
[4] This work was supported by the Royal Netherlands Academy of Sci. (KNAW, No. 11CDP003, JTMH), National Natural Science Foundation of China (Grant No.51572054, JCR), and NanoCenter, UMD.

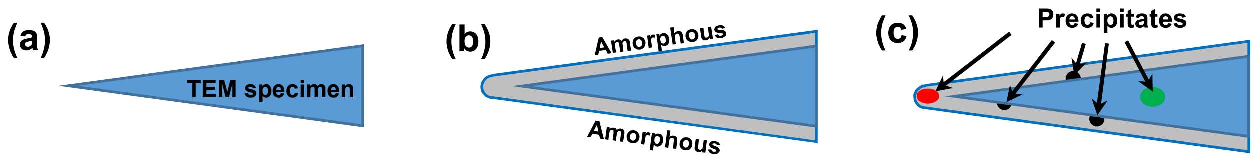


Figure 1. Schematic drawing shows the mechanism of present method: (a) cross-section of ready-for-observation wedge-shaped TEM specimen; (b) amorphous films on both side surfaces of the TEM specimen; (c) precipitates cling to the surface by amorphous film or isolated only inside amorphous film

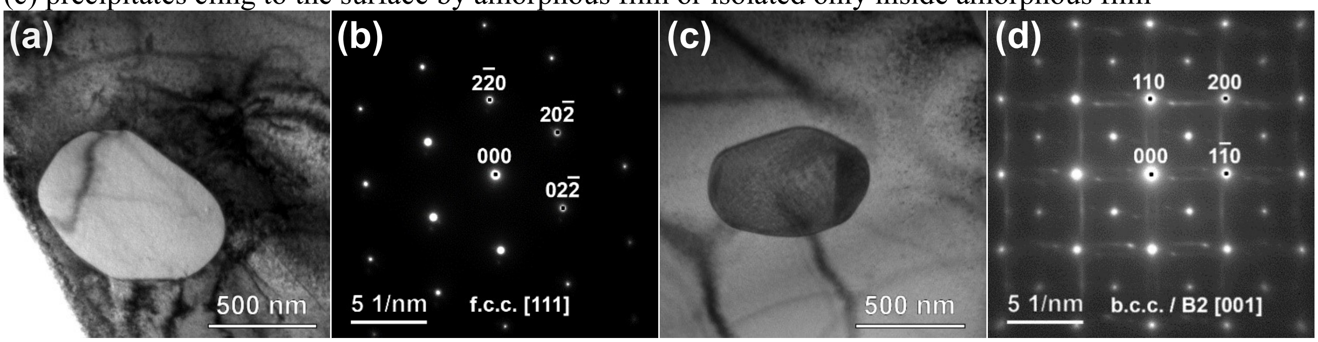


Figure 2. TEM micrographs of larger precipitates in high entropy alloy $Al_{0.5}CoCrFeNi$ reveal a large precipitate near the edge of TEM specimen (a) and the corresponding SAD pattern (b); a dark precipitate far away from the edge (c) and the corresponding SAD pattern (d).

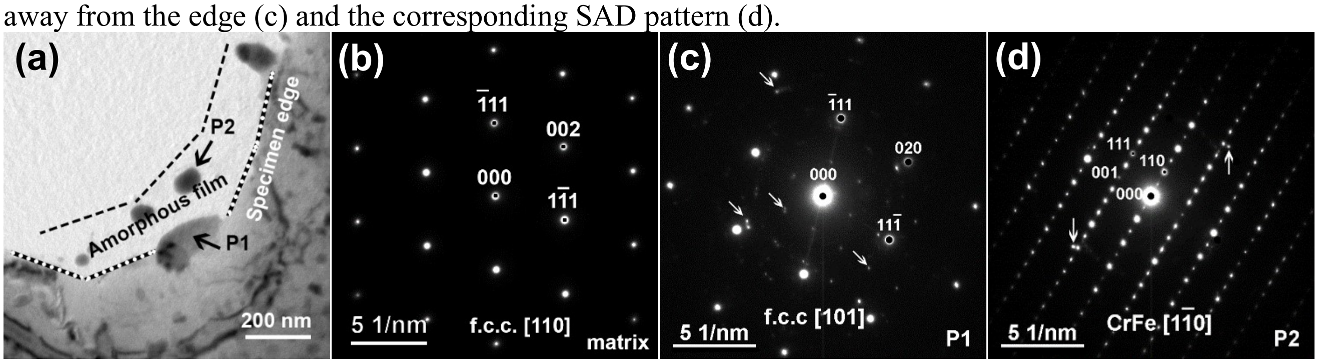
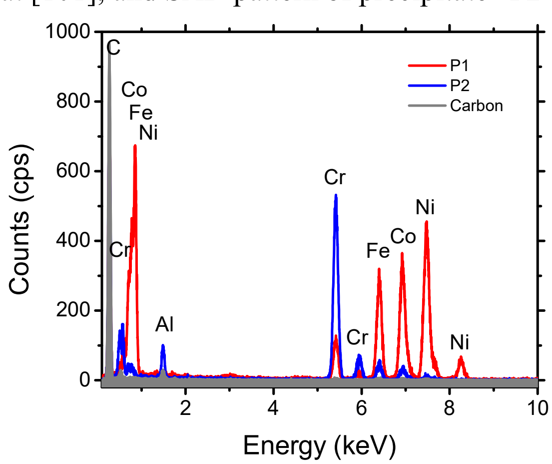


Figure 3. TEM image of high entropy alloy $Al_{0.7}CoCrFeNi$ taken after *in-situ* heating at 900 °C for 10 min. in TEM (a); SAD pattern of matrix at [110], precipitate “P1” can be identified as f.c.c Ni-rich solid-solution at [101]; and SAD pattern of precipitate “P2” showing CrFe-based solid solution at [1 -1 0].



Element	**C**	O	**Al**	Cr	**Fe**	**Co**	Ni
P1	-	-	0.78	6.67	20.71	29.84	42.01
P2	-	-	12.67	74.31	6.69	4.78	1.56
film	98.16	1.31	0.53	-	-	-	-

Figure 4. Results of EDS analysis of precipitates “P1” and “P2” as well as the amorphous carbon film.

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