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Precipitation Hardening via Chemically Ordered $L1_2$ Precipitates in Al-Sc-Zr Alloys— New Insights Using Combined STEM and EDS Study

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Al-Sc-Zr alloys have the potential for elevated-temperature strength resulting from thermally-stable $L1_2$ precipitates [1]. However, low solubilities of Sc and Zr limit precipitate volume fractions [2], enabling dislocation bypass via climb. One method to increase precipitate volume fraction involves using rapid cooling techniques to increase supersaturation. Rapid cooling utilizing melt spinning (10^7 k/s solidification rate) can increase solute supersaturation by a factor of ~8 from Al-0.06Sc-0.06Zr at% to Al-0.5Sc-0.4Zr at%. Melt-spun ribbon underwent two-step heat treatment (250°C for 40 min and 350°C for 1h). The ambient temperature hardness of peak-aged ribbon increased over the aged Al-0.06Sc-0.06Zr at% by 450 MPa (67%). Ribbon samples cut into 3mm discs using discs punch and thinned with mechanical polishing. Mechanical polished samples were cleaned with ethanol and electropolished using a Methalthin instrument with a 1:4 ratio mixture of reagent grade nitric acid/reagent grade methanol at 10 to 12 V.

Experimentally, many details of these phases, such as--composition of the precipitates, atomic occupancy --have not yet been discovered. In this work, we are studying the crystallographic and chemical nature of $L1_2$ precipitates. For the present work, high resolution STEM imaging and energy dispersive spectroscopy (EDS) chemical measurements have been performed from the same area.
The crystal structures and elemental maps by EDS of Al-Sc-Zr alloys with varying compositions were observed in an aberration corrected FEI Titan Themis microscope at Michigan Technological University. The spatial resolution of the STEM system was 0.08 nm. EDS chemical mapping was performed in the same instrument using a SuperX^{TM} EDS detector.

The best way to determine the $L1_2$-type chemical ordering in precipitates is tilting the grain along [001] zone axis. The Z- contrast HAADF STEM [3] image shows that the corner and face centered positions are occupied by different atoms (Figure 1A). The quantitative EDS analysis shows that the precipitate has 92 at. % Al, 6 at. % Sc, and 3 at. % Zr. The corresponding elemental EDS maps confirms that the corner and face centered positions are occupied by Sc/Zr and Al, respectively (Figure 1B-D). Interestingly, in about 20% of the cases, Al sites are occupied by Sc atoms (Figure 1E-F). This kind of site mixing is a direct consequence of arrested transformation from a random mixture of atoms to a chemically ordered phase during rapid cooling process.

Another important feature of the Zr/Sc site is the relative position of Sc and Zr atoms. Figure 2 shows that in some cases, Sc and Zr are separated with respect to each other in a given atomic site. The difference in atomic sizes between Sc and Zr may have contributed to this relative separation. So, this work determines the chemically ordered $L1_2$ phase formation in Al-Sc-Zr alloy system by high resolution STEM and EDS mapping. The site mixing and relative position of two atoms in a given site are indicative of complexity of alloying process when two or more immiscible atoms are used to form a chemically ordered phase at nanoscale [4].

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[4] The Office of Naval Research sponsor this work (N000141612878). The electron microscopy research was performed at the Applied Chemical and Morphological Analysis Laboratory, at Michigan Technological University. Electron microscopy facility is supported by NSF MRI 1429232.

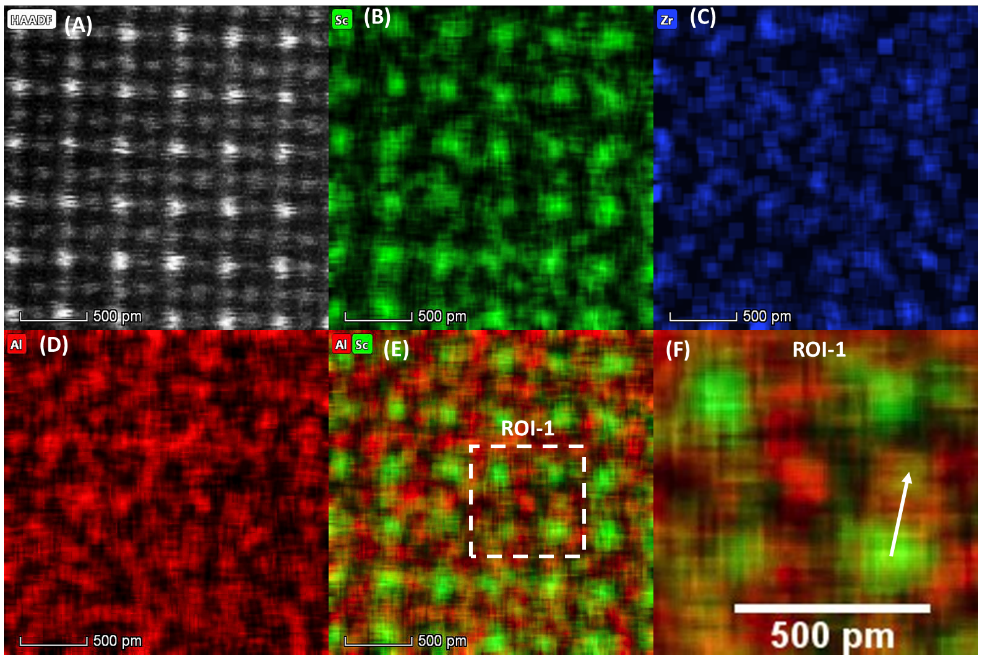

Figure 1. (A) Variation in STEM contrast as observed along [001] zone axis in $L1_2$ precipitates. The corresponding EDS maps of Sc (B), Zr (C), and Al (D) are shown in green, blue, and red. (E, F) Combined maps of Sc and Al shows an Al site is occupied by Sc atom (marked with arrow).

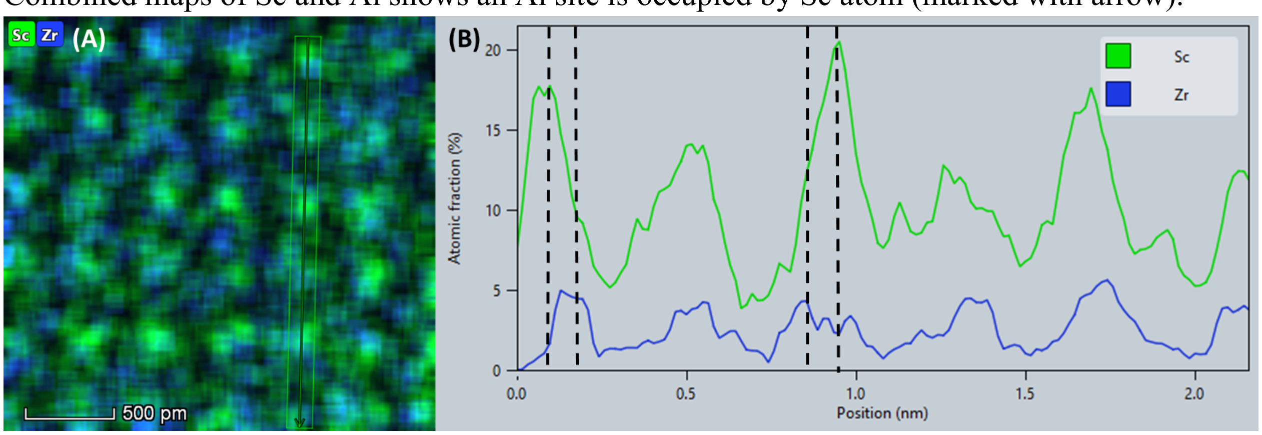

Figure 2. (A) Combined maps of Sc and Al shows relative displacement of Sc and Zr atoms in a given site. (B)The displacement is clearly visible from the linescan along the box in figure A.

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