

Direct *In situ* Observation of Tempering-induced Austenite Decomposition and Atom Probe Analyses of *k*-Carbide Precipitates in Lightweight Fe-Mn-Al-C Steels

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Tempering processing at temperatures of 500~600°C leads to the decomposition of retained austenite into Al-partitioned carbides (termed *k*-carbide) and Al-depleted ferrite, profoundly deteriorating the overall mechanical property of ferrite-based lightweight steels containing high-Al contents [1,2]. To evaluate the thermal stability of retained austenite, direct observation of austenite decomposition by *in situ* heating transmission electron microscopy (TEM) at 500°C is a promising topic. In addition, atom probe tomography (APT) can offer a pathway to three-dimensionally display the redistribution of microalloying elements in the products formed from austenite decomposition [1,2]. In particular, the current work pays attention to determine the concentrations of Al and C atoms in the products found in tempered steels through clipping them in conjunction with separate mass-spectra. Analyses were performed using a LEAP-4000HR microscope in voltage-pulsing mode at 200 kHz pulse repetition rate, 0.005 atom/pulse detection rate, 20% pulse fraction and 70 K.

Fig. 1 shows *in situ* heating TEM images at 500 °C showing morphological evolution of austenite decomposition into ferrite and *k*-carbides. High C contents of austenite with nanometer-size enables to have higher driving force for decomposition resulting in low thermal stability. The morphology of *κ*-carbides would be spheroidized with increasing the tempering temperature, sustaining the Nishiyama–Wasserman (N-W) relationship.

Fig. 2 shows 3-D reconstructed map of Mn, C, and Al exhibiting the presence of two *k*-carbides and ferrite decomposed from retained austenite. Note that Al³⁺ and Al²⁺ peaks detected at 9 and 13.5 Da in the mass-spectrum are visualized without the peaks of Al¹⁺, due to peak overlapping of ²⁷Al¹⁺ and ⁵⁴Fe²⁺.

The corresponding proxigram of cluster (as highlighted by rectangle box) and 1-D profiles along the cylinder provide the chemical gradients of elements across the phase boundaries. The C profile measured by the cylinder method and/or proxigram with one mass-spectrum can be underestimated in carbon-enriched regions and overestimated in carbon-depleted regions. The decomposition via the IVAS software represents that ¹²C₄²⁺ gives a contribution of about 40% to the peak at 24 Da in the mass-spectrum obtained inside carbide regions. In addition, peaks detected at 36 and 40 Da in the carbide spectrum can be ascribed to FeC and FeC₂, respectively. However, there are no 24.5 and 25.0 Da peaks, which can be assigned to (¹³C¹²C₃)²⁺, in spectrum found inside ferritic regions, suggesting that there is a negligible fraction of ¹²C₄²⁺ at the peak at 24 Da. This means that the concentration profile of carbon across the two phases are strongly affected by the choice of mass-spectrum. Besides the carbon peaks, the peak at 27 Da is partially assigned to ⁵⁴Fe²⁺ due to the contribution of Al¹⁺ ions [3]. In the Al-rich carbide regions, the peak decomposition algorithm in IVAS indicates that ~50% of this peak can be assigned to Al¹⁺, whereas ~20% for ferrite regions with lower Al contents. This implies that when the concentration profile of Al across the two phases using the cylinder method with one mass-spectrum, the

concentration of Al in Al-rich carbide regions can be underestimated, whilst overestimated in Al-depleted matrix. And hence, the measurement of Al contents in the two phases is considered by clipping them, and subsequently by identifying two different mass-spectra based on peak decomposition algorithm.

Although the peak decomposition algorithm can determine the concentration contribution of Al^{1+} to the peak at 27 Da, the reconstructed map does not reflect such a decomposition, implying that the distribution of Al in the 3D atom map is not divided by that of Fe.

References:

- [1] H.S. Park *et al*, *Materials & Design* **82** (2015), p. 173.
- [2] J.B. Seol *et al*, *Conference Proceedings of 7th Korea-China Joint Symposium on Advanced Steel* (2015) p. 93.
- [3] M.J. Yao *et al*, *Acta Mater.* **106** (2016) p. 229

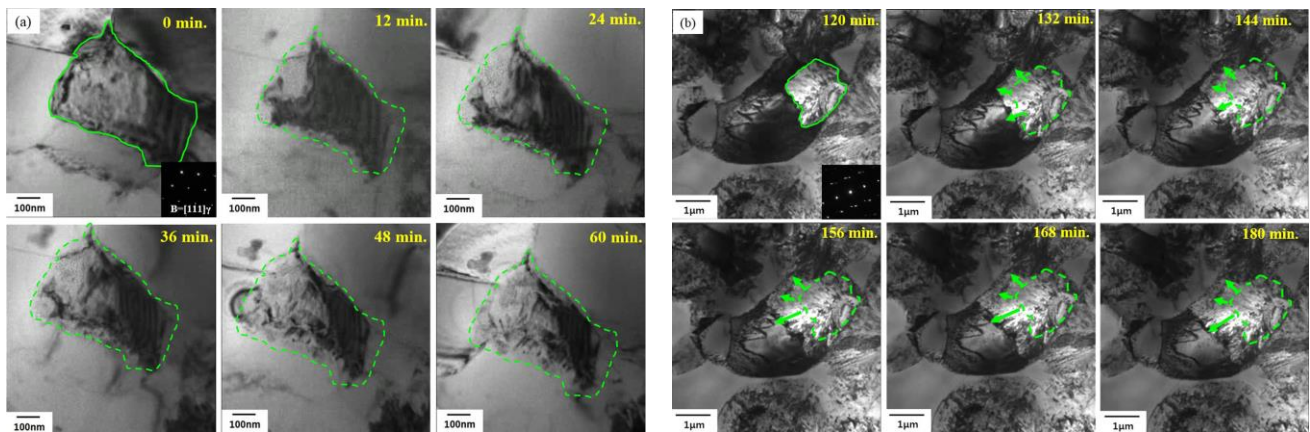


Figure 1. *in situ* heating TEM images at 500 °C showing the morphological evolution of austenite decomposition; (a) nano-sized austenite and (b) micro-sized austenite.

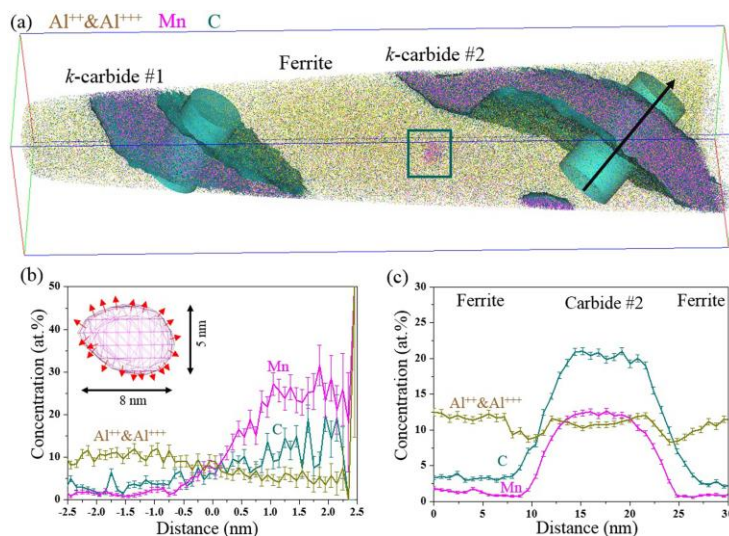


Figure 2. (a) 3-D reconstructed map of Mn, C, and Al exhibiting two k-carbides and ferrite formed by austenite decomposition; (b) proxigram of cluster and (c) 1-D profiles along the cylinder.