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Quantitative Analysis of Cementite in Steel by Atom Probe Tomography

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Atom probe tomography (APT) is a very powerful tool for the quantitative analysis of local composition of nano-scale features in steel. In particular, the quantitative analysis of carbon atoms in steel is intentionally required because carbon atoms play a very important role in controlling microstructures and mechanical properties. However, it was reported that an apparent carbon composition of cementite showed slightly different values from a stoichiometric composition of 25 at% [1]. It is not easy to analyze carbon composition in steel because carbon atoms are detected as various molecular ions in addition to single ions [2].

In this paper, the 3DAP performance of quantitative analysis of carbon atoms in steel was investigated through the composition analysis of spherical cementite (Fe_3C). The specimens were prepared by annealing heavily drawn pearlitic steel wires (Fe- 4.2C- 0.2Si- 0.29Mn- 0.22at.% Cr) at 500°C. Fig. 1 shows a TEM bright-field image of an annealed wire (500°C x 5h), observed in the direction perpendicular to the drawn axis. By annealing at 500°C, mixtures of unstable carbides can be avoided and cementite of stoichiometric composition can be obtained. Atom probe measurements were carried out using an energy-compensated 3DAP (Oxford Nanoscience Ltd.) under conditions with the specimen temperature being 20-90K, the pulse fraction being 15-25 %, and the pulse frequency being 1.5k-20k Hz. Fig. 2 shows the mass spectrum of a specimen containing a cementite region. According to the method of assignment used by Sha *et al*.[2], peaks of 6, 6.5, 12 and 13 were assigned to one single carbon ion (C1), peaks of 18, 18.5 and 36 to three ions (C3), and a peak of 24.5 to four ions (C4). However, different from the Sha's assignment, we assigned a peak of 24 to two ions (C2). Our assignment leads to the lowest carbon composition. Fig. 3 shows the specimen temperature and pulse fraction dependences of apparent carbon compositions in cementite and unwindowed noise. The apparent carbon composition decreased with increase in the temperature, which was interpreted by the increment of larger molecular ions. However, it is difficult to explain carbon compositions higher than 25at.%, obtained under low temperature conditions. Normally, quantitative performance increases with decrease in the temperature because preferential evaporation or retention is prevented [3-5]. On the other hand, the apparent composition did not changed with pulse fraction. The unwindowed noise corresponds to the amount of field evaporation while DC voltage is only applied, and it increased with increase in the temperature and with decrease in the pulse fraction. These results suggest that field evaporation rates of iron and carbon atoms in cementite intercrossed around 65K. Fig. 4 shows the temperature dependence of the apparent carbon composition calculated using our model where both pre-exponential factor and activation energy of evaporation rates are fitting parameters.

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Fig.1. TEM bright-field image of an annealed wire (500°C x 5hr).

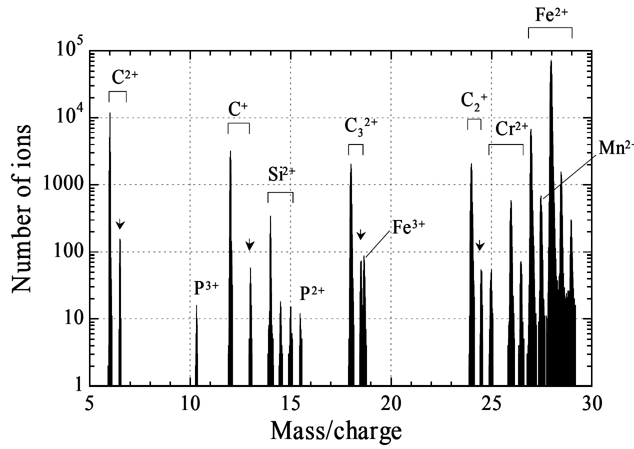
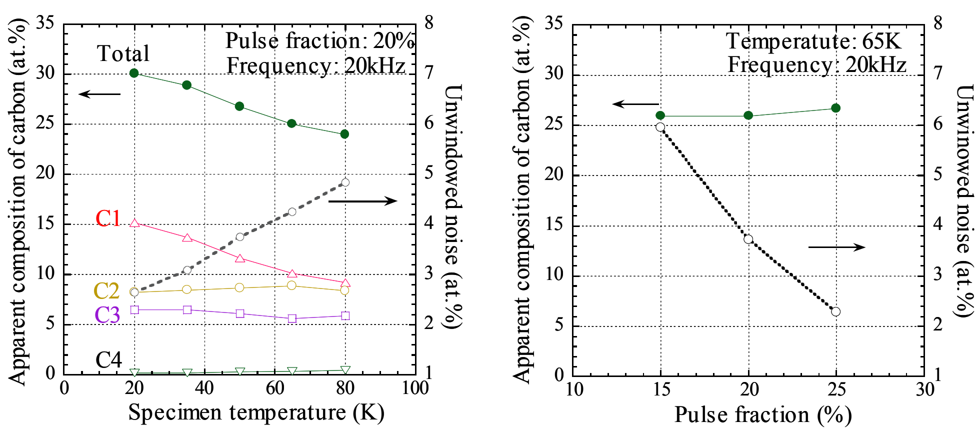


Fig.2. Mass spectrum of one million-ion data set containing ferrite and cementite. The arrows indicate ions involving ^{13}C isotope.



(a) (b)

Fig.3. Temperature (a) and pulse fraction (b) dependences of apparent carbon compositions and unwindowed noise in cementite.

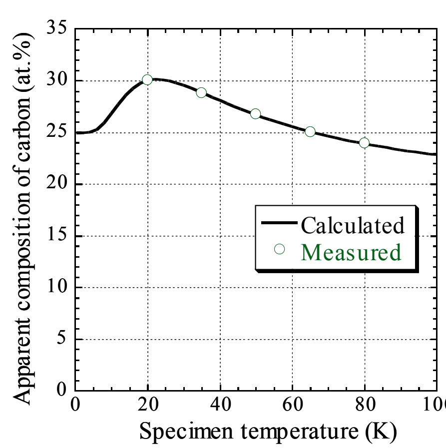


Fig.4. Apparent carbon composition calculated using our model.

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