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EELS Study of the Effect of Temperature on Ti L23 White Lines

In the study of changes in electronic structure of transition metal-based martensitic transformations, one is frequently addressed to calculate the L_3/L_2 ratios as well as the normalized total white-line areas, in order to measure e.g. electron transfer among different atomic sites. Sometimes it is argued that it is not clear if changes in white lines intensity are due to changes in electronic structure or are affected by changes in temperature.

In this work, EELS spectra were acquired from thin titanium foils at 20, 100, 200, 300 and 500°C. Samples were polished to a thickness less than 5 µm and then ion-milled. They were immediately placed in a heating sample holder and introduced in the transmission electron microscope, in order to avoid oxidation.

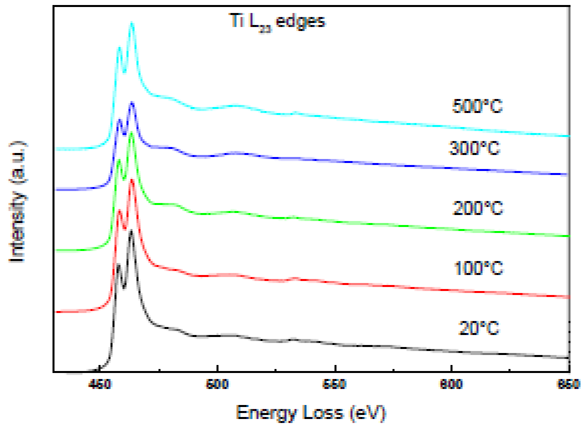
Electron energy loss spectra were obtained using a Gatan Parallel Electron Energy Loss Spectrometer (PEELS model 766) attached to a Philips CM-200 transmission electron microscope. Spectra were acquired in diffraction mode with 0.3 eV/ch dispersion, an aperture of 3 mm and a collection semi-angle of about 10 mrad. The resolution of the spectra was determined by measuring the full width at half-maximum (FWHM) of the zero- loss peak and this was typically close to 1.5 eV, when the TEM was operated at 200 kV The EELS spectra were corrected for dark current and readout noise. The channel to channel gain variation was minimized by normalizing the experimental spectrum with independently obtained gain spectrum of the spectrometer.

Next, spectra were background- subtracted by fitting the pre-edge backgrounds with a power-law function and then Fourier- Ratio deconvoluted to remove multiple scattering components.

Figure 1 shows Ti L_{23} ionization edges for all temperatures tested, where spectra have been shifted up for clarity. In all spectra appears a small bump around 530 eV, indicating the presence of small amounts of oxygen, these signals are, however, sufficiently small so as to neglect them.

To obtain L_3/L_2 ratios and normalized total white line intensities (L_{23}), we closely followed the work by Pearson et al [1]. The normalized white-line intensity is defined as the integrated intensity of the L_2 and L_3 white lines divided by the integrated intensity in a normalization window 50 eV in width beginning 50 eV past the L_3 edge onset. Figure 2 shows an example of this procedure.

Results obtained are summarized in Table 1. It is noted that values for L_3/L_2 remain unchanged, within experimental accuracy, up to 200°C and L_{23} remains the same for 20°C and 100°C. At higher temperatures, however, results must be taken with caution.



Ti L_{23} edges at different temperatures

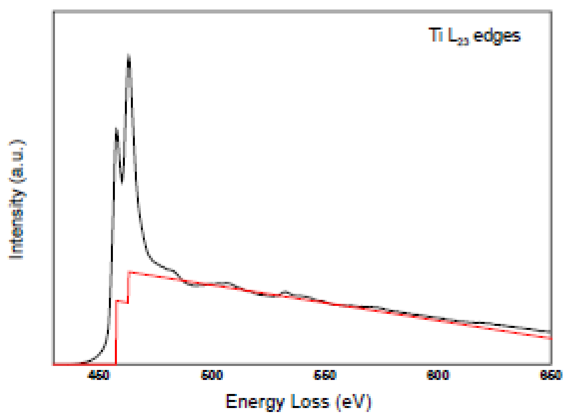


FIG. 2. Isolation of Ti L_{23} white lines

TABLE 1. Normalized total white lines areas L_{23} and white lines ratio L_3/L_2 for Ti at different temperatures

Temperatura (°C)	L_3/L_2	L_{23}
20	0.66	0.71
100	0.60	0.71
200	0.60	0.53
300	0.86	0.37
500	0.77	0.50

References

[1] D.H. Pearson, C.C. Ahn and B. Fultz, Phys. Rev. B 47 (1993) 8471