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The study of electron energy loss spectroscopy of Ni-4.8at%Ti alloy

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Abstract. An Ni-4.8at%Ti alloy was prepared and the electron energy loss spectroscopy (EELS) of nickel was collected. The *d*-electron occupancy of Ni is measured from the white-line intensity of EELS. It was found that relative to pure Ni the *d*-electron occupancy of Ni in Ni-4.8at%Ti alloy did not change significantly.

Introduction

Electronic sturcture is very important to the property of the alloys. In recent years both simulations and experiments are employed to explore the electronic structure in alloys. The *d*-electron occupancy is frequently concerned during compounds formation in the literatures, such as the *d*-electron occupancy of Ni or Fe. The techniques of XPS, XAS (X-Ray absorption spectroscopy), EELS (electron energy loss spectroscopy) and *ab initio* calculations have all been used to measure the change of electron occupancy. And the change in *d*-electron occupancy is related to the change in the property of the alloys.

EELS is usually equiped in transition electron microscope, which allows it can in-situ study the electronic structure of the alloys. For transition elements, the excitations of 2*p* core electrons in an atom to unoccupied states forms L_2 and L_3 absorption edges. The peaks are known as "white-line" [1]. The change of "white-line" intensity is proportional to the change of the *d*-electron occupancy [2-11] for 3*d* and 4*d* metals [9]. EELS is a tool to measure the change of *d*-electron occupancy of transition metals.

There is a lot of study on the Ti-Ni system alloys which possess lots of interesting mechanical and chemical properties, such as shape memory [12-16], hydrogen storage [17-18], and large negative energies of alloy-formation [19]. These properties have the origin in the electronic structure in essence. Therefore the study of the electronic structures of Ti-Ni systeim alloys is important and lots of work has been carried on the change of *d*-electron occupancy in the alloys.

In this paper we focus on the EELS of Ni in Ni-4.8at%Ti alloy and the change of L edge including the white-line intensity of Ni EELS in the alloy. The EELS of Ni in the alloy was acquired and the change of *d*-electron occupancy was measured from the white-line intensity of EELS.

Experimental details

In this work, Ni-4.8at%Ti alloy was prepared by arc-melting under an argon atmosphere. Samples of pure Ni and Ni-4.8at%Ti alloy were thinned down to electron transparency by twin jet electropolishing, then the samples was cleaned by ion milling to remove the possible thin oxide layer on the surface. The EELS experiments were performed on a field emission gun transmission electron microscope (JEOL 2010F) equipped with a GIF 678 system and EDS (Energy Dispersive X-Ray Spectroscopy) at 200 kV. EDS was used to check the composition of the alloys. After examining the oxygen edge and making sure that the surface of sample was clean, EELS of Fe in each alloy was acquired in similar thickness areas. The microscope was operated in diffraction mode with the camera length of 80 mm and a spectrometer collection aperture of 3 mm. The energy resolution is typically 1.4 eV estimated from the width of half maximum of the zero-loss peak. The detector dark current and gain variation were corrected. The pre-edge backgrounds of all spectra were subtracted by fitting with the power-law function and then deconvoluted by the Fourier-ratio method.

Results and discussion

EELS of Ni in Pure Ni and Ni-4.8at%Ti alloy are shown in Fig. 1. The L_3 and L_2 edges can be seen easily in the spectra. The L_3 and L_2 edges appear due to the excitations of $2p$ core electrons in an atom to the unoccupied states according to the dipole selection rule. The L_3 edge is about 855 ev and L_2 edge is about 872 eV. The EELS are aligned at the threshold position of the L_3 edge and the energy was set as zero. The EELS of Ni in the two alloys are quite similar respectively.

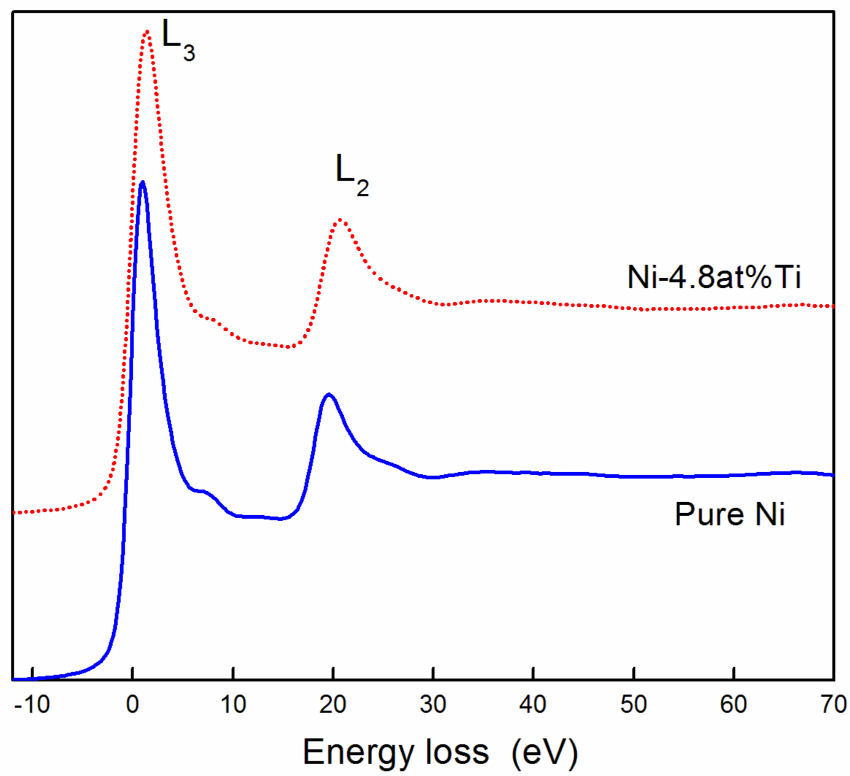

Fig. 1. EELS of Ni in pure Ni and Ni-4.8at%Ti alloy. The threshold position of the L_3 edge is taken as 0 eV.

To accurately measure the change of *d*-electron occupancy of Ni in the alloys, Pearson's method [9] was performed. Firstly, a step function is simulated to model the continuum background; Secondly, subtract the step function from L edges and obtain the white-line intensity; Thirdly, normalized the white line intensity and get *d*-electron occupancy of Fe according to the following formular [9].

$$I_{3d} = 10.8(1 - 0.10n_{3d}) \quad (1)$$

I_{3d} is the normalized intensity and n_{3d} is the *d*-occupancy of Fe atom.

The calculated *d*-electron occupancy of Ni in pure Fe is 8.804 and *d*-electron occupancy of Ni in Ni-4.8at%Ti alloy is 8.618. The difference is as small as 0.186 implying that the change of *d*-electron occupancy of Ni is not significant.

The change of *d*-electron occupancy of Ni in the Ti-Ni system compounds has attracted most interest for a long time, and the experiments such as saturation magnetic moments [20], X ray emission experiments [21], and SCF- Xα calculation [22] have been carried out to measure it. Recent results of XPS, XAS and EELS showed the change of *d*-electron occupancy of Ni is not significant in TiNi compound relative to pure Ni [23-28]. Our results showed that the change of *d*-electron occupancy of Ni in the Ni based solid-solution alloys containing Ti is very small also, so the LCN approximation may be accepted if only the change of *d*-electron occupancy of Ni is taken into account.

Summary

In summary, we collected electron energy loss spectroscopy (EELS) of Ni in Ni-4.8at%Ti and pure Ni and found that the *d*-electron occupancy of Ni did not change significantly when added into pure Ni. So local charge neutrality may be accepted when alloying.

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