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Orientation dependence of electron energy loss spectra and dielectric functions of Ti_3SiC_2 and Ti_3AlC_2

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Abstract

We employ monochromatized electron energy loss spectroscopy to study Ti_3SiC_2 and Ti_3AlC_2 . By probing individual grains aligned along different axes in bulk polycrystalline Ti_3SiC_2 and Ti_3AlC_2 , this approach enables determination of the anisotropy of the dielectric functions and an estimate of the free-electron lifetime in different orientations. The dielectric functions are characterized by strong interband transitions in the low energy region. The energies plasmon resonance were determined to be $\approx 5 \text{ eV}$ and exhibit a strong orientation-dependence. Our measurements show that the free-electron lifetimes are also highly orientation-dependent. These results suggest that scattering of carriers in MAX phases is very sensitive to composition and orientation.

Key words: Anisotropy, electronic structure, nanolaminate, conductivity, Drude-Lorentz model, EELS, MAX-phases

1. Introduction

MAX phases are hexagonal highly anisotropic ternary carbides and nitrides with a unique combination of properties half-way between metals and ceramics [1, 2, 3]. Their chemical composition is $M_{n+1}AX_n$ (n = 1 - 3), where M is an early transition metal, A is an element from groups 12 - 16,

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and X is carbon or nitrogen. Their crystal structure can be described as alternating layers of two transition metal planes forming octahedra cavities filled with carbon or nitrogen and layers of metal such as Al or Si. The MAX phases with n = 2 have a sequence of two MC octahedral and one metallic plane. This particular hexagonal structure is believed to confer unusual physical properties to these materials. MAX phases are potentially useful as high temperature components in aerospace application [1], for nuclear fuel confinement [4], electrofriction contacts [5], electromagnetic interference shielding [6], and more. Among the most important and studied ones are Ti₃SiC₂ and Ti₃AlC₂, the topic of the present study.

There is a strong need for improving the understanding of their electronic, thermal, and dielectric properties. The anisotropy in the electronic structure and its effect on conduction are very difficult to probe experimentally given the fact that MAX-phase single crystals have only been synthesized as thin films [2, 7, 8, 9, 10, 11] or as small (< 0.2 mm) free-standing crystals [12], not in bulk. Therefore, only very few experimental studies on the anisotropy in electronic structure or electrical properties of MAX phases exist [2, 13, 14, 15, 16].

Nevertheless, the anisotropy is important. For example, bulk samples of Ti_3SiC_2 exhibit negligible thermopower over a very wide temperature range, from 300 K to 850 K [17]. This astounding phenomenon has been explained from *ab initio* calculations of the band structure; these calculations predicted that two types of bands are the main contributors to the thermopower: a hole-like band in the *ab*-plane and an electron-like band along the *c* axis [18, 19]. For Ti_3SiC_2 , the predicted thermopower in the *c* direction was predicted to be twice that in the *a* direction, but with opposite sign. Therefore, the contributions in the *a*, *b*, and *c* directions would macroscopically cancel each other out in a randomly oriented bulk sample. It must be emphasized that this result was obtained under the assumption that the relaxation time can be approximated by a term proportional to the inverse of density of states at the Fermi level, which means that relaxation is isotropic.

Most theoretical studies discussing the issue of anisotropy in the conductivity of the MAX phases are calculations of the band structure, and often attempt to draw conclusions about conductivity based only on the electronic band structure. In reality, however, the conductivity also depends on electron-electron and electron-phonon interactions [20, 21] which are very difficult to model. It is therefore important to experimentally determine the dielectric functions, since they allow a determination of the free-electron lifetimes from the Drude-Lorentz model. In the present paper, we employ monochromatized electron energy loss spectroscopy to study Ti_3SiC_2 and Ti_3AlC_2 . By probing individual grains aligned along different axes in bulk polycrystalline Ti_3SiC_2 and Ti_3AlC_2 , this approach enables determination of the anisotropy of the dielectric functions and an estimate of the free-electron lifetime in different orientations.

2. Data acquisition and processing

Low-loss spectra were collected with a Zeiss Libra 200 TEM with a field emission gun and an electrostatic -filter type monochromator (MC) [22, 23, 24]. Acting as a bandpass filter, the MC sharply cuts out the high energy tail of the elastic peak[25]. This is particularly important for metallic materials, for which it is commonly difficult to separate correctly elastic and inelastic contributions.

The energy resolution without monochromator was typically ≈ 0.65 eV. After monochromator filtering the energy resolution was 0.2 or 0.3 eV, depending on the width of the filtering slit, which is selected.

Within the present experimental conditions using a small aperture, the momentum transfer is almost purely perpendicular to the incident beam. Thus it is contained in the basal plane when the electron beam is along the c axis and in a prismatic plane containing the c and a axes when the beam is along a. In the latter case, the dielectric response is a mixture of the responses in the basal plane and along the six-fold axis whereas in the former the pure contribution in the basal plane is probed.

The full complex dielectric functions have been determined after Fourier-Log deconvolution of the energy loss spectra to remove the plural scattering and Kramers-Kronig transformation. As mentioned, it critically depends on the separation of the elastic and inelastic contribution from the signal. In this work, the incident peak was recorded without a sample in the same experimental conditions, rescaled to the high of the elastic peak of the spectrum to process and then subtracted. This procedure was found to be more accurate and less prone to introduce artifacts.

The experimental dielectric function $\varepsilon(\omega)$ was then fitted to a semiclassical Drude-Lorentz model, given by the equation

$$\varepsilon(\omega) = 1 - \frac{n_p e^2}{m\varepsilon_0(\omega^2 - i\omega\gamma_p)} + \frac{e^2}{m\varepsilon_0} \sum_j \frac{n_j}{(\omega_j^2 - \omega^2) - i\Gamma_j\omega},$$

where the first term in the right hand side is the free electrons plasmon contribution and the second one a sum over j possible interband transitions (IBTs) characterized by their energy ω_j , and a damping parameter Γ_j . The oscillator strength n_j is related to the intensity of the transition j. The plasmon intensity is related to the number of free electrons n_p and γ_p represents the plasmon damping parameter.

In our procedure, both real and imaginary parts of the dielectric function are fitted simultaneously. Such fits of the experimentally determined dielectric functions to the Drude-Lorentz model are used to identify the strong IBTs at low energy. It allows characterizing the plasmon resonance, especially the relaxation time which is not trivial to access from theory.

3. Results and discussion

3.1. Monochromator influence

Figure 1 shows the raw low loss spectra of Ti_3AlC_2 recorded with the MC off and on. Apart from a better visibility of the structures in the Ti M edge at 38 - 40 eV, the two spectra are very similar above approximately 10 eV. However, in the low energy range, the spectrum recorded with the MC is dramatically improved. One can see that there is a significant spectral intensity below 5 - 6 eV and down to 2 eV. This spectral intensity is clearly intrinsic in origin. When the spectrum is recorded without the MC, it is more difficult to decide whether the low loss intensity comes from the high energy tail of the elastic peak or from inelastic processes. Consequently, the MC provides reliable signal down to a lower value in an important energy region containing the visible light frequencies.

Figure 2 shows the dielectric functions determined by Fourier-Log deconvolution of plural losses and Kramers-Kronig transformation of the spectra in Fig. 1. The improvement given by the MC is even more obvious. Again, at high energies the difference is small. As Fig. 2 shows, the two sets of data are significantly different below 24 eV. The strong intensity that shows up on the ε_2 curve shows that strong IBTs are operative in the compound. The smoothing due to experimental loss of resolution transforms the shape of the dielectric function to a more free-electronlike behavior by reducing the importance of low energy IBTs. The crossing of ε_1 with the abscissa, which should characterize the position of the plasmon, is also slightly red-shifted in the dielectric functions determined using the MC compared to the dielectric functions determined without the MC.



Figure 1: Ti_3AlC_2 low loss raw spectra recorded with monochromator off (red) and on (blue). The electron beam is collinear with the **c** axis.

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Figure 2: Comparison of the dielectric functions extracted from spectra recorded with MC (full lines) and without MC (dashed lines). Spectra recorded for Ti_3AlC_2 with electron beam along the [001] axis.

3.2. Experimental complex dielectric responses

Figure 3 compares the dielectric responses of Ti_3AlC_2 recorded with the beam along [100] (lower panel) and [001] (upper panel). The anisotropy of the material is clearly reflected in these functions. Whereas the high energy behavior is essentially the same for both orientations, the low energy region exhibit marked differences. In that region, strong IBTs are present and significantly perturb the plasmon response. In the upper panel, the ε_1 curve corresponding to the case where the beam is along the c axis, crosses the abscissa four times (at 10.1, 11.7, 12.8, and 16.6 eV) but the corresponding curve for the [100] orientation has no zero-crossing. On both curves, the intensity of ε_2 is similar (between 2-3 at 10 eV and 1 at 15 eV). Thus, ε_2 never satisfies the condition that $\varepsilon_2 \ll 1$ when ε_1 vanishes, which is necessary for a true plasmon to exist. By observing the low energy shape of ε_2 , it is likely that the strong interband transitions are responsible for this behavior of the plasmon. When the electron probe is along the [001] axis, the IBTs are well visible as a clear maximum of ε_2 at 7.9 eV well separated from the plasmon and followed by a relatively sharp decrease.

In contrast, the IBTs for the [100] orientation appears more like a smooth modification of the slope above 10 eV and therefore closer and more coupled to the plasmon resonance. These observations suggest that the energy positions of the IBTs, which are themselves closely related to the topology of the band structure, are responsible for the appearance or disappearance of the zero of ε_1 .

The dielectric complex function of Ti₃SiC₂ displayed in Fig. 4 bears some similarities with the previous case of Ti₃AlC₂ in that on one orientation the ε_1 exhibits crossing with the abscissa axis and not on the other. However, the situation is reversed now since when the beam is along [001], ε_1 does not cross the zero (Fig. 4, upper panel), whereas it does in the [100] orientation (Fig. 4, lower panel). We observe two intersections of ε_1 at 14.7 and 17.6 eV with the horizontal axis for the later orientation. A well-defined maximum is also present at 10 eV in the ε_2 component of the dielectric function. The fact that no true plasmon (i. e. $\varepsilon_1 = 0$ and $\varepsilon_2 << 1$) is observed in the [001] orientation is likely to be due to the strong transition visible as a maximum of ε_2 at 11 eV.

3.3. Fit results

The fitted dielectric functions are represented as solid lines in Figs. 3 and 4. Despite the crude Drude-Lorentz model, the complex dielectric functions



Figure 3: Dielectric functions of Ti_3AlC_2 determined with the beam along (a) [100] (red dashed line with symbols) and (b) [001] (blue dashed line with symbols). The fits of the dielectric functions to a Drude-Lorentz model are shown as full lines. The black dashed boxes represent the fitting intervals.



Figure 4: Dielectric functions of Ti_3SiC_2 determined with the beam along (a) [100] (red dashed line with symbols) and (b) [001] (blue dashed line with symbols). The fits of the dielectric functions to a Drude-Lorentz model are shown as full lines. The black dashed boxes represent the fitting intervals.

are remarkably well reproduced. However, this simple model breaks down at low energy since on one hand the number of IBTs to introduce would diverge and on the other hand the signal can be recorded down to a certain limit in energy. With our experimental setting we estimate that the signal is safely recorded down to approximately 0.5 eV. Nevertheless, the low energy boundary of the fitting window were set 1 to 2 eV as a minimum. The high energy limit could be extended up to 65 eV or more. It is always desirable to limit the number of adjustable parameters in the fits to constrain the model to physically sound parameters only. Therefore, we have used the minimum number of IBT compatible with a good quality fit of our experimental data. We have found that 4 and 3 IBTs were needed for Ti_3AlC_2 and Ti_3SiC_2 , respectively (Table 1). One additional oscillator was also required to account for the Ti M edge at approximately 45 eV (Table 2).

Compound	Energy	Oscillator strength	Lifetime
	(eV)		(eV^{-1})
Ti_3AlC_2 [001]	6.58	155	0.09
	9.18	113	0.16
	13.7	27.7	0.27
	18.9	45.2	0.98
Ti_3AlC_2 [100]	5.29	128	0.091
	9.15	49.7	0.15
	13.6	56.8	0.16
	20.2	63.8	0.089
Ti_3SiC_2 [001]	5.06	17.5	0.12
	10.9	27.1	0.29
	15.2	261	0.11
Ti_3SiC_2 [100]	6.31	250	0.056
	9.67	98.1	0.15
	14.5	60.7	0.16

Table 1: Interband transitions for Ti_3AlC_2 and Ti_3SiC_2 determined with the beam along [001] and [100].

Due to the additional electron of its A element, Ti_3SiC_2 holds on more electron per formula unit in its valence band and thus one less empty state in the conduction band available to promote excited electrons. This might be

an explanation for the fact that fitting the dielectric signal of Ti_3SiC_2 requires one less IBT than that of Ti_3AlC_2 . This observation, however, does not allow to draw any conclusions since both the occupied and unoccupied density of states are involved in the process. Looking in more details the table 1, one finds that three IBTs are present in all compounds all orientation at 5 - 6 eV, 9 - 10 eV and 13 - 15 eV. Therefore, these transition could be ascribe to the Ti - C hybrids, although the last one is probably also influenced by the *A* element. Finally, a transition at 19 - 20 eV seems specific of Ti_3AlC_2 . Further identification of the transitions requires theoretical spectra to be calculated from *ab initio* modelization.

Despite that the Ti-M edge is also modeled by an oscillator, it can be seen from Table 2 that the energies and lifetimes are very close from on experiment to the other. There is a tendency that the Ti-M edge is more enhanced in the [001] orientation. In this specific crystallographic orientation the momentum transfer is mainly in the basal plane, a configuration which may enhance some channeling effect of the excited electron.

Compound	Energy	Oscillator strength	Lifetime
	(eV)		(eV^{-1})
Ti_3AlC_2 [001]	45.15	255	0.065
Ti_3AlC_2 [100]	45.7	177	0.079
Ti_3SiC_2 [001]	48.3	324	0.055
$\mathrm{Ti}_3\mathrm{SiC}_2$ [100]	45.7	199	0.066

Table 2: Ti M_{45} edge Ti_3AlC_2 and Ti_3SiC_2 determined with the beam along [001] and [100].

Finally, this procedure allows the extraction of the free electrons lifetimes and plasmon energies as listed in the Table 3 for Ti₃SiC₂ and Ti₃AlC₂ in both crystallographic orientations (beam along [001] or [100]). These plasmon energies are located well below the maximum of the main peak of the loss function, which is generally considered as being the plasmon resonance. Preliminary *ab initio* calculations indicate that the plasmon resonance are located at such a low energy. Thus, the plasmon resonance is shifted in the 15 - 20 eV range due to the presence of the strong interband transitions. Our measurements show that the lifetime of free electrons are strongly orientation dependent. The anisotropy is somewhat smaller for Ti₃SiC₂.

Compound	$\mathrm{Ti}_{3}\mathrm{AlC}_{2}$	$\mathrm{Ti}_{3}\mathrm{AlC}_{2}$	$\mathrm{Ti}_3\mathrm{SiC}_2$	$\mathrm{Ti}_3\mathrm{SiC}_2$
	[001]	[100]	[001]	[100]
Energy (eV)	6.23	4.56	2.14	5.11
Lifetime (eV^{-1})	0.44	1.8	5.77	1.16

Table 3: Plasmon positions and free-electron lifetime for Ti_3AlC_2 and Ti_3SiC_2 determined with the beam along [001] and [100].

4. Conclusion

The dielectric function of the Ti_3SiC_2 and Ti_3AlC_2 compounds have been determined from HR-EELS using a monochromatized TEM. Strong interband transitions dominate the dielectric functions at low energy. Furthermore, the dielectric functions of Ti_3AlC_2 and Ti_3SiC_2 exhibit a marked anisotropy. Thus, the local-probe EELS measurements employed here can reveal the anisotropy of the dielectric properties of the MAX phases at a microscopic scale. This behavior is difficult to obtain from macroscopic measurements due to the lack of suitable single crystal samples. It needs to be stressed that recent ab-initio calculations of the dielectric functions of Ti_3SiC_2 , Ti_4AlN_3 [26] and V_4AlC_3 [27] do not account for the anisotropy in the dielectric properties.

The plasmon energies are determined to be close to 5 eV or below, whereas the plasmon peaks are found at much higher energies due to the presence of the IBTs. This peak exhibit a significant orientation-dependence with a shift larger than 1 eV in Ti_3AlC_2 . A much less pronounced shift is observed in Ti_3SiC_2 . Similarly, the electron lifetimes are highly orientation-dependent, which is expected to have important consequences on the transport properties of the MAX phases. These results are still preliminary since the overall accuracy is limited as stated in Sec. 3.3. Better values should be obtainable e. g. using more advanced signal processing for multiple losses deconvolution. This work also call for further theoretical analysis from *ab initio* modeling.

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