Thermal and Electrical Properties of Nb_2AlC, $(Ti, Nb)_2AlC$ and Ti_2AlC

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The heat capacities, thermal-expansion coefficients, thermal and electrical conductivities of Nb_2AlC (actual Nb:Al:C mole fractions: 0.525 ± 0.005, 0.240 ± 0.002, and 0.235 ± 0.005, respectively), Ti_2AlC and $(Ti, Nb)_2AlC$ (actual Ti:Nb:Al:C mole fractions: 0.244 ± 0.005, 0.273 ± 0.005, 0.240 ± 0.003, and 0.244 ± 0.005, respectively) were measured as a function of temperature. These ternaries are good electrical conductors, with a resistivity that increases linearly with increasing temperatures. The resistivity of $(Ti, Nb)_2AlC$ is higher than the other members, indicating a solid-solution scattering effect. The thermal-expansion coefficients, in the 25 °C to 1000 °C temperature range, are comparable and fall in the narrow range of 8.7 to 8.9×10^{-6} K^{-1}, with that of the solid solution being the highest. They are all good conductors of heat, with thermal conductivities in the range between 15 to 45 W/m K at room temperature. The electronic component of the thermal conductivity is the dominant mechanism at all temperatures for Nb_2AlC and $(Ti, Nb)_2AlC$. The conductivity of Ti_2AlC, on the other hand, is high because the phonon contribution to the conductivity is nonnegligible.

I. INTRODUCTION

THE ternary carbides Nb_2AlC and Ti_2AlC belong to a larger class of solids with the general formula $M_{N+1}AX_N$, where $N = 1$ to 3, M is an early transition metal, A is an A-group (mostly III and IV A) element, and X is C or N. These, so-called MAX, phases are hexagonal ($P6_3/mmc$) layered compounds, wherein pure layers of the A-group elements are interleaved with $M_{N+1}X_N$ layers having the rock-salt structure.[1] Over the past few years, compounds have been shown to posses unusual combinations of properties.[1–16] They are readily machinable but quite stiff, resistant to thermal shock and damage tolerant. Some of them also exhibit some very attractive high-temperature properties.

Nowotny and co-workers[17,18] were the first to synthesize Nb_2AlC and Ti_2AlC. Later, they reported on the existence of a solid solution between the end members.[19] Recently, details on the synthesis and characterization of Ti_2AlC, Ti_2AIN, and $Ti_2AlC_{0.5}N_{0.5}$ were reported.[3,9] In the latter work,[9] it was shown that, whereas the mechanical properties and thermal-expansion coefficients (TECs) were affected by the formation of the solid solution, the electrical properties were much less altered.

In general, the MAX phases are good thermal conductors because they are good electrical conductors.[5,10,14] The phonon contribution to the total conductivity, with a few notable exceptions, Ti_2AlC being one of them, is small, despite the fact that these solids are elastically stiff and have high Debye temperatures.[6,16] The phonon contribution is suppressed because of two factors. First, the presence of small concentrations of point defects which, not unlike the binary near-stoichiometric MX phases, are potent phonon scatterers.[20,21] This is one reason, for example, $Ti_4AlN_{2.9}$, is a poorer phonon conductor than Ti_2AlC.[1] The second reason is more unique to the MAX phases in that the A element, by virtue of its being relatively loosely bound, acts as a rattler.[1] Rattlers, defined as atoms whose vibrational amplitudes are higher than other atoms in the structure, are also potent phonon scatterers.[22,23] This hypothesis explains why, for example, the phonon contribution to the total conductivity in Ti_3SiC_2 or Nb_2SnC is quite small.[1] As discussed subsequently in Section IIIC, Ti_2AlC is an exception because at room temperature roughly 50 pct of the heat conductivity is due to phonons.[1]

This article is a continuation of our efforts to fabricate and fully characterize all the known MAX phases.[1,17] Here, we report on the heat capacities, thermal expansion coefficients, TCE's, TECs, and thermal conductivities of Nb_2AlC, $(Ti, Nb)_2AlC$, and Ti_2AlC in the 200 °C to 1200 °C temperature range. We also measured their electrical resistivities down to 4 K.

II. EXPERIMENTAL PROCEDURE

Mixtures of graphite (−325 mesh, 99 pct), Al_4C_3 (−325 mesh, 99.0 pct+), Nb (1–5 μm, 99.8 pct), and Ti (−325 mesh, 99.5 pct) powders were used in this work. The purities, based on metal basis alone, are those specified by the supplier (Alpha Aesar, Ward Hill, MA). The processing details for the Ti_2AlC and the Nb-containing samples can be found in References 9 and 15, respectively. In all cases, the powders were mixed in the proper stoichiometry, cold isostatically pressed, sealed in glass under vacuum, and placed in a hot isostatic press (HIP). The Ti_2AlC samples were hipped at ≈40 MPa for 16 hours at 1300 °C and the Nb_2AlC and $(Ti, Nb)_2AlC$ samples at ≈100 MPa for 8 hours at 1600 °C. The samples were sliced, mounted, and polished down to a 1-μm diamond suspension for optical and scanning electron microscopy (SEM). The chemical composition was measured by electron probe X-ray microanalysis (EPMA), which was performed on a Cameca SX100 (Paris). Measurements were carried out using an accelerating voltage of 20 kV, and

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a probe current of 10 nA. The K_α lines from the following standards were used for the quantitative analysis: pure Ti for Ti metal and Fe_3C for C. The standard for Nb was Nb metal, where the L_α line was used instead.

The densities, δ, of the hipped samples were measured using Archimedes' method in water at ambient temperatures. The resistances were measured using a four-probe technique at 290, 273.15, 77, and 4.2 K. The coefficients of thermal expansions of bulk samples ($4 \times 4 \times 25$ mm^3) were measured under flowing Ar (25 mL/min) in the 25 °C to 1200 °C temperature range using a dilatometer (Unitherm, Anter Corp., Pittsburgh, PA). The thermal strains were measured, both on heating and cooling, at a rate of 2 °C/min. The dilatometer was precalibrated, and the error in the measurements is estimated to be $\approx$2 pct.

The heat capacities and thermal diffusivities, D, of Ti_2AlC were measured using the equipment and procedures described in Reference 5; the corresponding values for the other two samples were measured using a different calorimeter and a different laser-flash system. The former was measured using a differential scanning calirometer (DSC) (Netzsch, 404C, Selb, Germany). The DSC was calibrated using a sapphire crystal; the calibration and all measurements were carried out under Ar at a heating rate of 10 K/min. The samples were cylindrical (5-mm diameter, $\approx$1-mm thick). For the laser-flash technique, a flat cylindrical sample (11-mm diameter, 1-mm thick) was placed in a vacuum furnace (10^{-4} atm) and heated to a predetermined temperature; at which time, the front surface was pulsed, for 1 ms, with a 40 J pulse of a Nd:YAG laser. The increase in temperature on the rear surface was monitored with a pyrometer, and the temperature *vs* time curve was digitized and analyzed to estimate the half-time lag, $t_{1/2}$, between the initial and the stable final temperatures. The D values are calculated assuming, $D = 0.134\, d^2/t_{1/2}$. The Ds are then converted to thermal conductivities, k_{tot}, using the relationship: $k_{\text{tot}} = c_p D \delta$.

III. RESULTS AND DISCUSSION

A. *Microstructural Characterization*

The Ti_2AlC samples were fully dense, measured density, $\delta = 4.1$ Mg/m^3, with an average grain size of $\approx$25 μm. Optical and SEM micrographs of the resulting microstructure can be found elsewhere.[9] Backscattered SEM micrographs revealed that in addition to the Ti_2AlC matrix, two minority phases, most likely Al_2O_3 and Ti_3P,[9] with a total volume fraction of $\approx$4 vol. pct exist. The EPMA indicated that the final composition was close to the stoichiometric composition, Ti_2AlC.

To obtain phase-pure samples of Nb_2AlC, over a dozen different compositions were explored.[15] The nominal composition that resulted in predominantly single-phase samples was $Nb_{1.95}Al_{1.05}C_{0.95}$. However, the actual Nb, Al, and C mole fractions, as determined by EPMA were 0.525 ± 0.005, 0.240 ± 0.002, and 0.235 ± 0.005, respectively. If one assumes the Nb sites to be fully occupied, the resulting compound is best represented as $Nb_{2.00}Al_{0.91}C_{0.89}$. In other words, it is postulated that vacancies exist on the Al and C sites. The resulting predominantly single-phase samples contained $\approx$2 vol pct Al_2O_3 and $\approx$ 1 vol pct Nb-carbides and aluminides. The grains were equiaxed with an average grain size of $\approx$14 μm. The samples were fully dense, with a measured density ($\delta = 6.37 \pm 0.02$ Mg/m^3) that is 98% of value calculated from the lattice parameters.

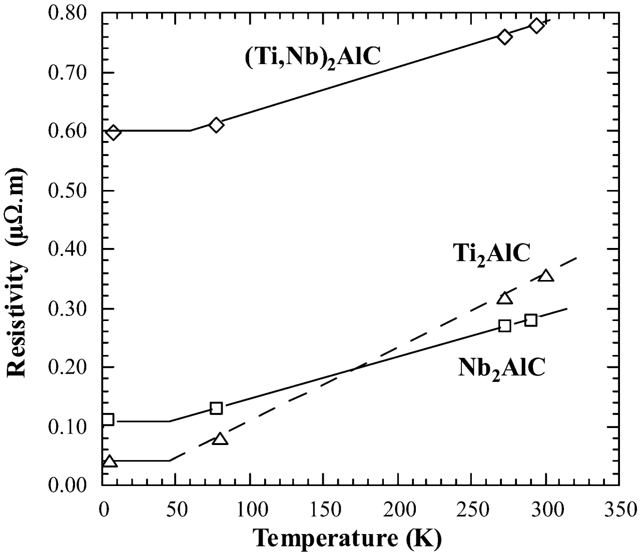


Fig. 1—Temperature dependence of resistivity in the 4.2 to 300 K range.

The starting composition for the solid solution was (Ti, Nb)$_2$AlC with a Ti:Nb ratio of 1:1. The resulting material was predominantly single phase with $\approx$2 vol pct Al_2O_3 and $\approx$1 vol pct of Nb-Ti carbides and aluminides. According to EPMA results, the actual Ti, Nb, Al, and C mole fractions were 0.244 ± 0.005, 0.273 ± 0.005, 0.240 ± 0.003, and 0.244 ± 0.005, respectively. Once again, assuming the Nb sites are fully occupied, the resulting material can best be represented as $(Ti_{0.47}, Nb_{0.53})_2Al_{0.93}C_{0.94}$. The a and c lattice parameters, 3.077 Å and 13.79 Å, respectively, are in good agreement with previous work.[19] The samples were fully dense; the measured density (5.3 ± 0.05 Mg/m^3) is in excellent agreement with theoretical value calculated from the lattice parameters, *viz.* 5.28 Mg/m^3.[19] The grains are platelike, with an average size of 50 to 60 μm.

B. *Electrical Properties*

The temperature dependencies of the resistivities, ρ, are plotted in Fig. 1. Like all MAX phases [1], ρ increases linearly with increasing temperatures and can be represented by:

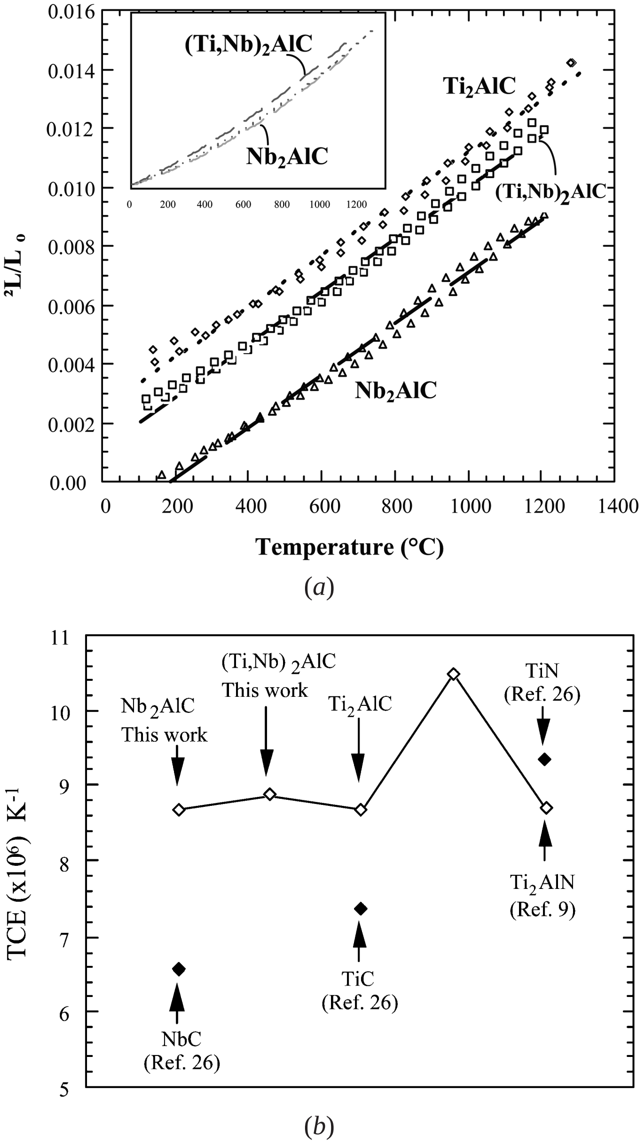
$$\rho = \rho_0[1 + \alpha(T - T_{\text{ref}})]$$

where ρ_0, α, and T are, respectively, the resistivity at the reference temperature, T_{ref}, the temperature coefficient of resistivity, and the temperature in degrees Kelvin. Least squares fits of the results, shown in Figure 1, yield the values listed in Table I. In all cases, the reference temperature is 300 K and $R^2 > 0.999$. Note that these relationships are only valid for temperatures $>$70 K.

The resistivity of the solid solution is significantly higher than its end members, which implies that solid-solution scattering is occurring in this system. In contrast, when the resistivities of Ti_2AlN, $Ti_2AlN_{0.5}C_{0.5}$, and Ti_2AlC are compared, the solid solution appears to fall on the line joining the end members.[9] It, thus, appears that the effect of substitutions on the M sites on the electrical conductivity is greater

Table I. Values of ρ_0 ($\mu\Omega$m) and α (K)$^{-1}$ and the Coefficients of a Second-Order Polynomial Assuming $\Delta L/L_0 = A + B\,T(K) + CT^2$. The Conductivity Results are Only Valid for Temperatures >70 K and the Thermal Expansion in the 400–1500 K Temperature Range.

Compound	ρ_0 ($\mu\Omega$m)	α (K)$^{-1}$	Ref. Temp.	A	B	C	R^2
Nb_2AlC	0.29	0.0024	300 K	-1.6×10^{-3}	-4.0×10^{-6}	2.4×10^{-9}	>0.99
(Ti, Nb)$_2$AlC	0.78	0.001	300 K	-1.8×10^{-3}	-4.5×10^{-6}	2.34×10^{-9}	>0.99
Ti_2AlC	0.36	0.0035	300 K	-1.4×10^{-3}	-3.3×10^{-6}	2.7×10^{-9}	>0.99



(*a*)

(*b*)

Fig. 2—(*a*) Temperature dependence of thermal expansions upon heating and cooling. The data for the solid solution are shifted upward by a strain of 0.002 and those for Ti_2AlC by 0.004. Least-squares fits of the results are shown by lines superimposed on the data. The inset superimposes the results of the polynomial fits (text and Table I). (*b*) Comparison of TCEs of select M_2AX phases and those of their corresponding near-stoichiometric binary carbides, MX.

than those on the X sites. This conclusion is based on only two solid solutions, and consequently, its general validity for the other MAX phases must await further work.

When compared to the residual resistivities of the other MAX phases measured to date,[1] the values reported here for Nb_2AlC are relatively high. Such high values are consistent with a structure in which the concentration of defects, in general, and point defects, in particular, are large, *i.e.*, consistent with the proposed structure, $Nb_{2.00}Al_{0.91}C_{0.89}$.

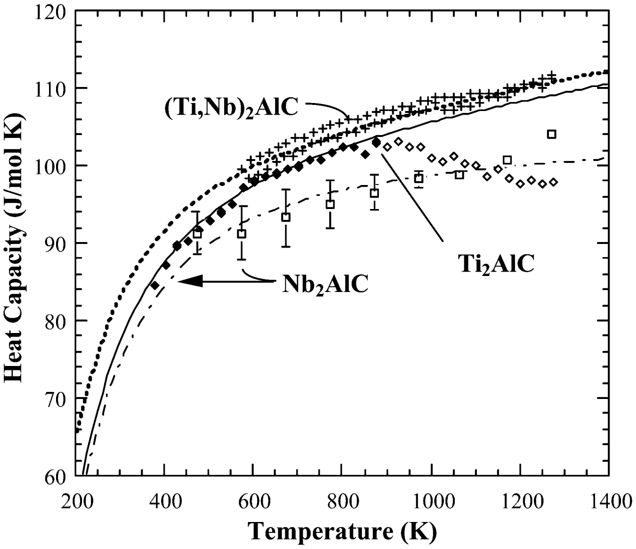


Fig. 3—Temperature dependence of heat capacities. The data were fit to the Debye model, which results in the various lines superimposed on the data. For Ti_2AlC, the data points above ≈900 K were not used in the fit (text). To obtain the results listed in Table III, the lines were fit to a third-order polynomial.

C. *Thermal Properties*

The thermal expansions (on heating and cooling) of the two end members are almost indistinguishable, and both are slightly lower than the solid solution (Figure 2). Least squares fitting of all points (heating and cooling) yields the following TECs: $8.7 \times 10^{-6}K^{-1}$ for Nb_2AlC, $8.7 \times 10^{-6}K^{-1}$ for Ti_2AlC, and $8.9 \times 10^{-6}K^{-1}$ for the (Ti, Nb)$_2$AlC. The R^2 values in all cases was >0.98. Given the curvature, a better fit can be obtained by a second order polynomial; the coefficients of which are also listed in Table I. Here, the R^2 values were >0.995. It follows that, as in the case of $TiAlC_{0.5}N_{0.5}$, the solid solution somewhat destabilizes the structure at higher temperatures,[9] the effect in this case is much more subtle, however (inset in Figure 2(a)). In contrast to the Sn and Pb-containing (211) phases,[11] there is little correlation between the TECs of the MAX phases and their respective binary near-stoichiometric MX phases (Figure 2(b)).

The temperature dependence of the heat capacities, c_p, of the end members and the solid solution are shown in Figure 3. The solid and dotted lines in Figure 3 represent the best

Table II. Coefficients of Third-Order Polynomial Used to Fit the c_p Results after They Were Fit to the Debye Model. The Relevant Equation is: $c_p = A + B\,T(K) + CT^2 + DT^3$.

Compound	A	B	C	D	Temp. Range (K)
Ti_2AlC	58.1	0.10	-7.0×10^{-5}	1.8×10^{-8}	300–1600
$(Ti, Nb)_2AlC$	74.8	0.06	-4.3×10^{-5}	1.2×10^{-8}	300–1600
Nb_2AlC	59.2	0.09	-7.3×10^{-5}	2.0×10^{-8}	300–1600

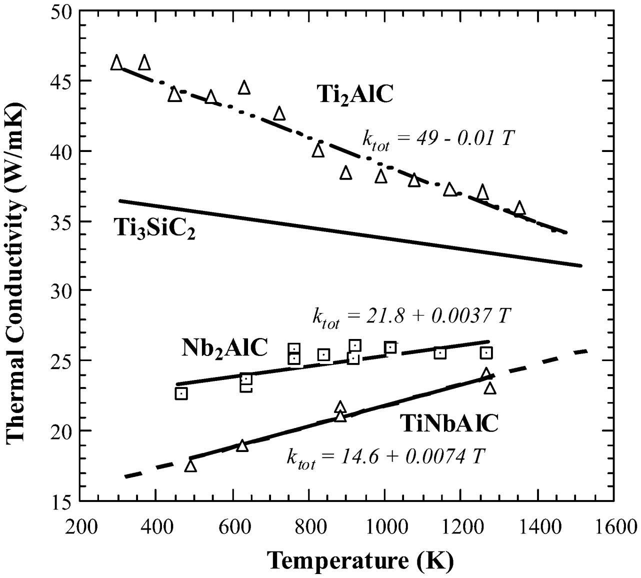


Fig. 4—Temperature dependence of thermal conductivity. The results for Ti_3SiC_2[5] are also included for comparison.

fits of the data to the Debye model. Once generated, the Debye curve was, in turn, fitted to a third-order polynomial, the coefficients of which are listed in Table II.

The reason for the decrease in c_p at temperatures higher than 900 K for Ti_2AlC is not clear at this time, but it is most probably related to the loss of Al from the sample. Recent work on Ti_2InC showed that a much more severe decrease in c_p with increasing temperatures was related to the loss of In.[24] The data points for temperatures >900 K (open diamonds in Figure 3) were, therefore, not used for the fitting of the results (solid line). Based on the c_p results, it is unlikely that the other two compositions lost Al; why that is the case is not clear at this time. From the figure, it is clear that at higher temperatures, the heat capacities of the solid solution are highest, followed by those of Ti_2AlC, and finally Nb_2AlC. The slight increase in the c_p data at temperatures >1200 K are probably due to oxidation from residual oxygen in the Ar gas.

The effect of temperature on the thermal conductivities, k_{tot}, is shown in Figure 4. The thermal conductivities of the Nb-containing ternaries increase slightly with increasing temperatures; conversely, k_{tot} of Ti_2AlC decreases. Least-squares fits of the data, shown as straight lines in Figure 4, yield.

$$k_{tot}\,(Nb_2AlC) = 21.8 + 0.0037\,T \quad R^2 = 0.61$$

$$k_{tot}\,(Ti, Nb)_2AlC = 14.4 + 0.0074\,T \quad R^2 = 0.94$$

$$k_{tot}\,(Ti_2AlC) = 49 - 0.01\,T \quad R^2 = 0.90$$

Because both k_{tot} and ρ are known, the Wiedmann–Franz law:

$$k_e = L_o\,T/\rho$$

where $L_o = 2.45 \times 10^{-8}\ W\Omega/K^2$, can be used to decouple the electronic, k_e, and phonon, k_{ph}, contributions to k_{tot}. This expression has been shown to be valid for many of the MAX phases explored to date.[1] The results are summarized in Table III for two temperatures. Based on these results the following points are salient.

1. The Ti_2AlC has the highest k_{tot} because of its high k_{ph}. Increasing the temperature reduces k_{ph} by about 60 pct.
2. At all temperatures, $(Ti, Nb)_2AlC$ has the lowest k_{tot} because both k_{ph} and k_{tot} are lower than the end members presumably because of solid-solution scattering. Increasing the temperature to 1300 K decreases k_{ph} by ≈50 pct.
3. Because for Nb_2AlC, the impossible result that $k_e > k_{tot}$, is obtained, one must conclude that $L_o < 2.45 \times 10^{-8}\ W\Omega/K^2$. This is not unique to Nb_2AlC but is true for other Nb-containing MAX and MX phases, such as NbC[25] and Nb_2SnC[1,14] (last two rows in Table III). Furthermore, given that it is unlikely that k_{ph} of the solid solution is greater than that of Nb_2AlC, the value for the latter must be greater than the former, *i.e.*, >7 W/m K. Using that value for Nb_2AlC implies that L_o reduces to L with $L \approx 1.5 \times 10^{-8}$. Using this value at 1300 K yields the values listed in Table III (entries with double asterisks). These values are in good agreement with the values obtained for Nb_2SnC (Table III), arrived at using slightly different assumptions.[1] They are also in good agreement with the values of k_{ph} calculated for $(Ti, Nb)_2AlC$ using the Wiedmann–Franz law (*i.e.*, assuming $L_o = 2.45 \times 10^{-8}\ W\Omega/K^2$).
4. For the Nb-containing phases, where k_{ph} is small, and more or less constant with temperature, the slope of k_{tot} *vs* temperature is positive because their residual resistivities are high.*[1] The slope for the solid solution is higher because its residual resistivity is higher (Figure 1). For Ti_2AlC, the slope is negative, probably because k_{ph} decreases with increasing temperatures (Table III).

It is important to point out that the primary purpose of the preceding exercise is not to determine the phonon contribution to k_{tot} precisely but rather to make the point that for the Nb-containing phases, k_{ph}, is quite small, both in relation to k_e and in absolute terms. Given the low thermal expansions, relatively high Debye temperatures and presumably

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*If k_{ph} is not a function of temperature, then dk_{tot}/dT has the same sign as the intercept of the electrical resistivity *vs* T curves at 0 K. Thus, if the intercept is high, the slope is positive.

Table III. Summary of Thermal Conductivity Results Obtained in This Work; Also Included are Literature Results for TiC, Nb_2SnC, and NbC

	k_{total} at		300 K		1300 K	
Compound	300 K	1300 K	k_e	k_{ph}	k_e	k_{ph}
$TiC_{0.96}$	14.4[27]	33.4[27]	7.35 (50 pct)	7 (50 pct)	—	—
TiC_x	33[26]	39[26]	12[26] (36 pct)	21 (62 pct)	24 (66 pct)	15 (38 pct)
Ti_2AlC	46	36	20 (43 pct)	26 (57 pct)	19.7 (55 pct)	16.3 (45 pct)
$(Ti, Nb)_2AlC$	16.6	24	9.4 (56 pct)	7.2 (43 pct)	20.3 (85 pct)	3.7 (16 pct)
Nb_2AlC	23	27	26* (>100 pct)	—	32* (>100 pct)	—
	—	—	15.6** (77 pct)	>7.4 (32 pct)	20** (73 pct)	>4 (26 pct)
NbC_x	14[25]	—	21[25]* (>100 pct)	—	—	—
Nb_2SnC	17.5[14]	30.5	18** (>100 pct)	—	25 (82 pct)	5 (18 pct)

*Since $k_e > k_{tots}$, the Lorenz number must be $<2.45 \times 10^{-8}$ WΩ/K (text).

**Assuming Lorenz number $= 1.5 \times 10^{-8}$ WΩ/K (text).

high stiffnesses, this is a somewhat surprising result. It is, however, in agreement with notion that the *A* atoms act as rattlers in these structures.[1] Clearly, Ti_2AlC is an exception presumably because the Al atoms are strongly bound in that structure, at least at lower temperatures. However, based on the TEC data, it can be argued that the Al atoms are as well bound in Nb_2AlC as they are in Ti_2AlC. Rietveld analysis of high-temperature neutron or X-ray diffraction would be invaluable in answering this question and is indicated.

A second source of phonon scattering is point defects. It is well established that such defects are potent scatterers of phonons in near-stoichiometric MX phases, such as TiC_x and NbC_x.[20,21] This is especially true here, when it is recalled that, if one assumes vacancies on the A and X sublattices, the resulting stoichiometries are $Nb_{2.00}Al_{0.91}C_{0.89}$ and $Ti_{0.94}Nb_{1.06}Al_{0.93}C_{0.94}$. These comments notwithstanding, it is hereby acknowledged that more work is needed to sort out some of these issues. Most indicated at this time would be low-temperature thermal-conductivity measurements and neutron diffraction studies at elevated temperatures, as well as a systematic characterization of the thermal and electrical properties as a function of stoichiometry.

To date, over a dozen MAX phases have been synthesized and characterized.[1] The highest k_{tot} in the 300 to 1400 K temperature range belongs to Ti_2AlC. Finally, it is worth noting that the microstructural stability of Nb_2AlC is excellent; the grain size of HIP samples at 1600 °C for 8 hours is $<\approx 15$ μm.[15] These characteristics indicate that this compound is potentially an excellent candidate for high-temperature applications. However, unless its oxidation resistance in air, which is poor,[28] can be enhanced, its use will have to be confined to nonoxidizing atmospheres.

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