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Mo₂Ga₂C: A New Ternary Nanolaminated Carbide

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Abstract

We report the discovery of a new hexagonal Mo₂Ga₂C phase, wherein two Ga layers – instead of one – are stacked in a simple hexagonal arrangement in between Mo₂C layers. It is reasonable to assume this compound is the first of a larger family.

Introduction

The ternary M_{n+1}AX_n, or MAX, phases (where M is an early transition metal, A is an A group element mostly groups 13 and 14, X is C and/or N, and $n = 1$ to 3), phases are a large family, 70+, of nanolayered, machinable solids.^{1–3} There are approximately 50 M₂AX, or 211, phases, five M₃AX₂, or 312, phases and a growing number of M₄AX₃, of 413, phases since that structure was first established in Ti₃AlN₄. In all cases, the M_{n+1}AX_n unit cells are hexagonal – space group *P*6₃/*mmc* – with two formula units per unit cell. In these compounds, near close-packed M atoms are interleaved with a single layer of pure A-element; the X atoms occupy the octahedral sites in between the M atoms. In the 211's, every third layer is an A-group element, in the 312's every fourth layer, and in the 413's every fifth. Recently, these solids have attracted much attention due to their unusual and sometimes unique combination of properties.

Of special interest to this work is the first, and sole, Mo-containing MAX phase, Mo₂GaC, first synthesized in 1967 by reacting Mo and C powders with liquid Ga for four weeks at 850 °C in an evacuated quartz capsule.⁴ Superconducting behavior below 7 K has been reported.^{4,5} More recently a theoretical paper was published, predicting some of its properties.⁶ Compared to Nb₂GaC and V₂GaC, Mo₂GaC was predicted to have the highest bulk and lowest shear modulus.

In efforts to synthesize Mo₂GaC, an XRD peak around 9° 2θ suggested the possible existence of Mo₃GaC₂. However, since the latter is predicted to be highly unstable,⁵ further work described herein, led us to the discovery of a new phase: Mo₂Ga₂C, wherein two Ga layers – instead of one in Mo₂GaC and all other MAX phases – are stacked in a simple hexagonal arrangement in between Mo₂C layers.

The processing details can be found in the ESI.[†] In short, the new phase was produced in two forms: thin film and bulk. The thin films were grown by direct current magnetron sputtering of elemental targets on MgO(111) substrates. Bulk Mo₂Ga₂C samples were synthesized by first heating a 2 : 1 molar ratio of Mo:C powders in flowing Ar at 1000 °C for 12 h. The resulting, lightly sintered, Mo₂C compact was crushed into a powder and mixed with Ga in a 1 : 8 molar ratio. First, the Ga was heated to 45 °C to melt it and the Mo₂C powder was homogeneously mixed into the melt in a mortar and pestle, before the mixture was allowed to solidify. The mixture was then placed in a quartz tube that was evacuated using a mechanical pump and sealed. Then, the quartz tube was placed in an alumina furnace and heated at a rate of 10 °C min⁻¹ to 850 °C, and held at that temperature for 48 h. After furnace cooling, the powder was immersed in a 37 wt% HCl for 3 days to dissolve any residual Ga and Ga₂O₃, if present. A predominantly single phase powder, with 18 wt% Mo₂C, was obtained after separating the solution and washing the powders several times using deionized water and dried in air.

The morphologies of all the phases resulting from the bulk form were imaged in a scanning electron microscope (SEM) (Supra 50VP, Carl Zeiss AG, Germany) equipped with an energy dispersive spectroscope (EDS). X-ray diffraction, XRD, of the resulting powders was carried using a diffractometer (see the ESI[†] for details). High resolution scanning electron microscopy (HRSTEM) and X-ray energy dispersive spectroscopy (EDX) were performed with a double Cs corrected FEI Titan3 60–300 operated at 300 kV, equipped with the Super-X EDX system. Selected area electron diffraction (SAED) characterization was carried out using a FEI Tecnai G2 TF20 UT instrument operated at 200 kV, with a point resolution of 0.19 nm.

Fig. 1 shows a θ – 2θ XRD pattern of a thin film sample, where the two peaks with highest intensities at $2\theta = 36.97^\circ$ and 78.64° can be assigned to the (111) and (222) diffractions, respectively, of the MgO substrate. The other nine peaks originate from phases in the thin film with a d spacing with a least common multiple of ~ 9.04 Å. The insets in Fig. 1, shows two XRD pole figures of the thin film sample acquired respectively at constant $2\theta = 34.26^\circ$ (inset (i) in Fig. 1) and 62.50° (inset (ii) in Fig. 1). At $2\theta = 34.26^\circ$, six poles can be seen at $\Psi = 87$ – 90° with 60° separations in between, which shows a six-fold symmetry in the in-plane directions with respect to the sample surface. This symmetry can be assigned to a phase from the film with an in-plane d spacing of ~ 2.62 Å. At $2\theta = 62.50^\circ$, three poles can be seen at $\Psi = 33$ – 39° with 120° separations in between, while two groups of six poles with 60° separations in between are observed at $\Psi = 81^\circ$ and $\Psi = 87$ – 90° , respectively. The poles at $\Psi = 33$ – 39° and 87 – 90° are assigned to MgO{220} from the MgO(111) substrate, which has a three-fold rotational axis along MgO[111]. The six-fold symmetric poles at $\Psi = 81^\circ$ can be assigned to a phase oriented in accordance with the in-plane orientation of the substrate, i.e. it is epitaxially grown on the MgO substrate.

[†] Electronic supplementary information (ESI) available: Synthesis of materials, and characterization techniques. See DOI: 10.1039/c5cc00980d

Fig. 1 XRD pattern of $\text{Mo}_2\text{Ga}_2\text{C}$ thin film sample. The two peaks with highest intensities at $2\theta = 36.97^\circ$ and 78.64° are those of the MgO substrate. The other 9 peaks come from the thin film and represent a series of interplanar spacing d with a least common multiple of $\sim 9.04 \text{ \AA}$. The pole figure labelled (i) was acquired at a constant $2\theta = 34.26^\circ$ – and the one labelled (ii) was acquired at $2\theta = 62.50^\circ$.

Fig. 2a and b are SAED patterns from the new compound. The phase has a hexagonal structure with a and c lattice parameters of $3.05 \text{ \AA}$ and $18.19 \text{ \AA}$, respectively. The possibility that those patterns originate from a 312 MAX structure (i.e., the hypothetical Mo_3GaC_2 which is not stable⁵) can be excluded for the following reasons. As noted above, the chemical composition analysis shown in Fig. 2c exhibits a different Mo/Ga ratio from that of a Mo_3GaC_2 phase. The structure is also not that of $\text{Ta}_2\text{S}_2\text{C}$ and $\text{Nb}_2\text{S}_2\text{C}$ phases, which have the same,^{7,8} or at least similar “221” stoichiometries, since they belong to the space group $R\bar{3}m1$. Furthermore the stacking observed in HRSTEM (below), is inconsistent with either of these sulphides or a 312 MAX phase.

Fig. 2 (a) Selected area electron diffraction of Mo₂Ga₂C thin films in $[11\bar{2}0]$ and, (b) $[10\bar{1}0]$ zone axes, (c) EDS spectrum; (d) HAADF images in the (d) $[11\bar{2}0]$ and, (e) $[10\bar{1}0]$ zone axes. Insets are corresponding atomic structure models indicating the corresponding positions of the Ga and Mo atoms in the stacking sequence.

To reveal the detailed structure, a Z contrast image was obtained using HRSTEM. Fig. 2c and d show the HRSTEM images with the beam aligned along the $[11\bar{2}0]$ and $[10\bar{1}0]$ zone axes, respectively. The Z contrast images show a double-layer structural feature. The bright and dark spots should correspond to the Mo and Ga atoms, respectively.

The corresponding EDX maps shown in Fig. 3(a–f) confirm the Mo–Mo–Ga–Ga–Mo–Mo layering. The simplest description of the structure is the following: start with a 211 MAX phase

structure and simply insert one extra Ga layer on top of the existing Ga. Surprisingly, the two Ga layer lie exactly on top of each other (*i.e.* not close-packed), an unusual arrangement indeed.

Fig. 3 (a) HAADF image and corresponding, (b) Mo, (c) Ga, and, (d) Mo and Ga maps, (e) HAADF image superimposed with Mo and Ga maps, (f) line scan along [0001] direction a $\text{Mo}_2\text{Ga}_2\text{C}$ film.

Based on the Z-contrast images, the distance between adjacent Mo layers was estimated to be 2.27 Å. The separation between the Mo and Ga layers is shortest: 2.09 Å. The separation between the adjacent Ga layers at 2.64 Å is relatively high. Based on the measured data, initial atomic positions of the Mo and Ga atoms are found and used as input data to the Rietveld refinement below.

Fig. 4 shows a typical XRD pattern of the phase obtained after dissolving the unreacted Ga from the bulk sample. When this new phase was imaged in a HRSTEM (see above) its structure was found to be unlike any other MAX phase known, in that there were two Ga layers separating

the Mo₂C blocks. Making use of this insight, the XRD pattern was analyzed assuming the unit cell shown in the inset of Fig. 4.

Fig. 4 Powder XRD patterns indexed to Mo₂Ga₂C showing observed pattern (black crosses), Rietveld generated pattern (red line) and difference between the two (blue line). The black and green ticks below the pattern represent the peak positions of Mo₂Ga₂C phase, and Mo₂C phase, respectively. The χ^2 value was 4.93. Inset shows schematic of a unit cell where the Ga atoms are green, Mo are red and carbon are black.

To determine the composition of this new phase, elastic recoil detection analysis (ERDA) was carried out on a close to phase-pure thin film sample containing traces of Mo–C intermetallic phases, and the Mo, Ga and C contents, in at%, were found to be, respectively, 38.9%, 42.5%, 18.3%, with a trace amount of oxygen (0.34 at%). This composition is consistent with 2 : 2 : 1, within the error margins of the technique.

The results of the Rietveld analysis are summarized in Table 1. The space group assumed was that of the MAX phases: $P6_3/mmc$. The a and c -lattice parameters were calculated to be 3.03396(4) Å and 18.0814(3) Å, respectively. The overall temperature factor was calculated to be 0.22(3) Å² and 6.5(6)% of the sample was preferably oriented in the (001) direction. The presence of 19.8(4) wt% of Mo₂C was also found. When this phase was taken into account, the χ^2 value was 4.93. The occupancies for all atoms were fixed at 100%.

Table 1 Atomic positions in Mo₂Ga₂C determined from the Rietveld analysis of the XRD pattern shown in Fig. 1. The space group was $P6_3/mmc$. The a and c -lattice parameters were calculated to be 3.03396(4) Å and 18.0814(3) Å, respectively.

Element	x	y	z	Wyckoff positions
Mo	1/3	2/3	0.06571(11)	4f
Ga	1/3	2/3	0.68247(13)	4f
C	0	0	0	2a

A list of the *hkl* indices of the various peaks – theoretical and experimentally observed – and their intensities and d spacings are listed in Table S1 (ESI†), which shows generally good agreement between the theory and experiment. It should be noted here that since the simple hexagonal arrangement of the Ga atoms is somewhat unusual, other arrangements were tested, where the Ga layers were sheared with respect to each other. The χ^2 values in those cases were significantly higher than for the unit cell shown in the inset of Fig. 4.

The importance of this work lies beyond the discovery of a totally new phase, as exciting as that may be. After Kudielka and Rohde,⁹ discovered the first MAX phase – then referred to as a H phase – in 1960, Nowotny and co-workers discovered over 50 such phases, including the 312 phases.¹⁰ In 1999 Ti₄AlN₃ – the first 413 phase – was discovered;¹¹ since then over five 413 phases have been discovered, not including solid solutions. Based on this track record, it is quite reasonable to assume that Mo₂Ga₂C is the first of a new and distinct family of MAX-related phases.

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Supplementary Information for

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S1 Experimental Details:

a) Synthesis of the bulk Mo₂Ga₂C:

The starting materials used were commercial Mo (63 NS, Metco Inc., Anderson, SC), graphite (Grade 4827, Asbury Graphite Mills Inc., Asbury, NJ) powders and Ga shots (99.99%) (Roto Metals Inc., San Leandro, CA). First the Mo and graphite powders were weighed in a 2:1 molar ratio and placed in a plastic bottle and mixed for 24 h using agate balls as the milling media. The resulting, lightly sintered, Mo₂C compact was crushed into powder and mixed with Ga in a 1:8 molar ratio to form Mo₂Ga₂C. The mixture was then placed in a quartz tube that was evacuated using a mechanical vacuum pump and sealed. The quartz tube was then placed in an alumina tube furnace and heated at a rate of 10 °C/min to 850 °C, and held at that temperature for 48 h. After furnace cooling, the powder was immersed in a 37 wt. % HCl solution for 3 days to dissolve any residual Ga and Ga₂O₃ if present. Predominantly single phase Mo₂Ga₂C powder – with ≈ 20 wt.% Mo₂C – was obtained. The powders were then washed with deionized water several times and dried in air for further analysis.

b) Thin film synthesis:

Direct current magnetron sputtering was used to grow Mo-Ga-C thin films were synthesized. The films were co-deposited from three elemental targets, Mo (3-inch, 99.95% purity, SCOTECH Ltd.), Ga (2-inch, 99.99999% purity, 5N Plus UK Ltd.) and C (3-inch, 99.99% purity, SCOTECH Ltd.) with respective powers 40 W, 18 W and 200 W and at ~0.5 Pa Ar with a background pressure in the range of 10⁻⁷ Pa. Due to its low melting point (~ 30°C), the Ga source was kept in a concave stainless steel crucible right below the substrate, in line with previously developed procedures. [A. Petruhins, A. S. Ingason, M. Dahlqvist, A. Mockute, M. Junaid, J. Birch, J. Lu, L. Hultman, P. O. Å. Persson and J. Rosen, Phys. Status Solidi RRL, vol. 7, no. 11, pp. 971-974, 2013]. The Mo and C targets were tilted +35° and -35° away from the horizontal position of the Ga target, co-focusing onto the rotating substrate.

The thin films were grown on MgO(111) substrates (10×10×0.5 mm³, Latech Ltd.) that were ultrasonically cleaned sequentially in acetone, ethanol and isopropanol for 10 minutes at each stage. Before deposition, the substrate was heated to 560 °C at the base pressure, followed by a 10 minutes pre-sputtering with the same powers for deposition. A shutter was inserted to blind the substrate from the target's line-of-sight when pre-sputtering, and was afterwards removed directly to start the deposition at the same substrate temperature set-point (560°C).

S2 Characterization Details:

a) Details of XRD Experimental Parameters and Refinement Conditions

XRD patterns were obtained with a diffractometer (Rikagu Smartlab, Tokyo, Japan), with a step size of 0.02° in the 3°-120° 2θ range with a step time of 7 s with a 10x10 mm² window slit. Scans were made with Cu K α radiation (40 kV and 44 mA).

Rietveld refinement of the XRD diffractograms was carried out using the FULLPROF code [J. Rodriguez-Carvajal, Phys. B 192 (1993) 55–69]. The refinement was carried out from 8.5° to 120° 2θ . Refined parameters were: five background parameters, scale factors from which relative phase fractions are evaluated, Y profile parameter for peak width, lattice parameters (LPs), the overall thermal factor, preferred orientation and atomic positions for all phases. The experimental and those calculated from the Rietveld refinement are summarized in Table S1.

Table S1: X-ray (Cu K α) powder diffraction data for Mo₂Ga₂C.

Peak Number	h	k	l	2θ	I calculated	I observed	d_{hkl}
1	0	0	2	9.775	440.1	460.1	9.040678
2	0	0	4	19.623	12.9	0	4.520339
3	0	0	6	29.619	8.7	0	3.013559
4	1	0	0	34.095	639.6	646.7	2.627484
5	1	0	1	34.465	87.6	79.1	2.600175
6	1	0	2	35.552	4.6	15.7	2.523087
7	1	0	3	37.303	1439.2	1607.4	2.408563
8	1	0	4	39.644	22.3	31.1	2.271614
9	0	0	8	39.853	480.5	513.7	2.26017
10	1	0	5	42.493	1128.4	1308.5	2.125649
11	1	0	6	45.779	16.4	83.9	1.980435
12	1	0	7	49.44	91.9	86.8	1.842001
13	0	0	10	50.43	3.1	0	1.808136
14	1	0	8	53.43	361.3	375.3	1.713455
15	1	0	9	57.718	0.3	0	1.595959

Peak Number	<i>h</i>	<i>k</i>	<i>l</i>	2θ	<i>I</i> calculated	<i>I</i> observed	<i>d</i> _{hkl}
16	1	1	0	61.032	530.3	492.5	1.516979
17	0	0	12	61.49	14.2	12.1	1.50678
18	1	1	2	61.979	20.9	20.1	1.496064
19	1	0	10	62.282	7.1	5.8	1.489519
20	1	1	4	64.771	4.4	1.3	1.438156
21	1	0	11	67.114	174	128.1	1.393528
22	1	1	6	69.29	6.6	16.4	1.354988
23	2	0	0	71.795	68.6	56.8	1.313742
24	2	0	1	72.014	8.5	7.2	1.310288
25	1	0	12	72.217	7.8	6.3	1.307101
26	2	0	2	72.668	1	1	1.300087
27	0	0	14	73.228	2	1.6	1.291525
28	2	0	3	73.755	185.1	165.8	1.283603
29	2	0	4	75.265	2.7	2.5	1.261544
30	1	1	8	75.404	432.9	431.9	1.259573
31	2	0	5	77.193	185.9	182.1	1.234785
32	1	0	13	77.604	181.9	193.4	1.229266
33	2	0	6	79.529	3.4	4.6	1.204282
34	2	0	7	82.267	17.9	24.4	1.17099
35	1	1	10	83.031	5.5	9	1.162145
36	1	0	14	83.3	0.9	1.5	1.159069
37	2	0	8	85.405	90.3	89.9	1.135807
38	0	0	16	85.942	46.6	49.1	1.130085
39	2	0	9	88.945	0.2	0.1	1.099527
40	1	0	15	89.347	26.2	18.9	1.095625
41	1	1	12	92.199	28.2	34.5	1.069042
42	2	0	10	92.898	2.8	3.7	1.062824
43	1	0	16	95.804	48.9	45.5	1.038136
44	2	0	11	97.284	74.5	80.4	1.026246
45	0	0	18	100.14	1.4	5.9	1.00452
46	2	1	0	101.728	63.2	69.1	0.993096
47	2	1	1	101.941	5.5	5.8	0.991601
48	2	0	12	102.138	3.7	4.2	0.990218
49	2	1	2	102.579	1.6	2.2	0.987158
50	1	0	17	102.762	5.5	7.5	0.985896
51	1	1	14	103.128	8.9	10	0.983394
52	2	1	3	103.647	190.3	177	0.979883
53	2	1	4	105.15	2.8	2.3	0.969963
54	2	1	5	107.098	211.4	218.3	0.957642
55	2	0	13	107.519	104.8	111.9	0.95506

Peak Number	<i>h</i>	<i>k</i>	<i>l</i>	2θ	<i>I</i> calculated	<i>I</i> observed	d_{hkl}
56	2	1	6	109.508	5.6	16.2	0.943201
57	1	0	18	110.362	0.8	5.4	0.938286
58	2	1	7	112.403	19.4	37	0.926948
59	2	0	14	113.517	1.1	2.6	0.921001
60	2	1	8	115.822	132.4	125.5	0.9092
61	1	1	16	109.508	5.6	16.2	0.943201
62	0	0	20	110.362	0.8	5.4	0.938286
63	1	0	19	112.403	19.4	37	0.926948
64	2	1	9	113.517	1.1	2.6	0.921001

b) TEM, XRD and Structural Characterization

The TEM specimens were prepared by mechanic polishing followed by ion thinning down to electron transparency. High resolution scanning electron microscopy (HRSTEM) and X-ray energy dispersive spectroscopy (EDX) were performed with a double C_s corrected FEI Titan3 60–300 operated at 300 kV, equipped with the Super-X EDX system. Selected area electron diffraction (SAED) characterization was carried out using a FEI Tecnai G2 TF20 UT instrument operated at 200 kV with a point resolution of 0.19 nm. Structural characterization of the thin films was performed through X-ray diffraction (XRD). The system utilized was a Panalytical Empyrean MRD with a $Cu K\alpha$ source. The measurements performed were symmetric (θ - 2θ) scans obtained by employing a hybrid mirror and a 0.27° parallel plate collimator in the incident and the diffracted beam side, respectively.

Fig. S1 Scanning electron microscope micrograph of Mo_2Ga_2C powders showing typical MAX phase platelet morphology.

Fig. S2 XRD pattern of Mo₂Ga₂C bulk sample, the triangle markers represent peaks for free Gallium.