X-ray powder diffraction data for Mn_4C

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We report on the X-ray diffraction data and unit-cell parameters of Mn_4C, which has a cubic perovskite-type structure with $a = 3.8726$ Å and unit-cell volume $V = 58.1$ Å^3. The measured lines were indexed and are consistent with the space group $Pm\bar{3}m$ (No. 221). © *2019 International Centre for Diffraction Data*. [doi:10.1017/S0885715619000265]

Key words: Mn_4C, X-ray powder diffraction

I. INTRODUCTION

Mn_4C has a cubic perovskite-type crystal structure, with Mn atoms at the corners and the face centers and a C atom at the body center (Tagawa and Motizuki, 1991; Si *et al*., 2018a, 2018b). It is difficult to prepare Mn_4C because it is unstable. A γC solid solution with a composition that seemed to be about Mn_4C was produced via a peritectoid reaction (Isobe, 1951). However, the composition of the carbide Mn_4C was thought to be more accurately represented by the formula $Mn_{23}C_6$ (Kosolapova, 1971). Morgan found that an induction melted alloy with a nominal composition of Mn_4C was weakly ferromagnetic after water quenching. About 70% was separated with a magnet and the products were considered to be Mn_4C (Morgan, 1954). However, the Mn_4C reported by Morgan was soon suggested to be Mn_4(C, O) (Kuo and Persson, 1954; Benz *et al*., 1973). Moreover, the similar water quenching of Mn-C alloys with varied compositions invariably gave a mixture of $Mn_{23}C_6$ and Mn (Karen *et al*., 1991). Most later researchers have tended to believe that Mn_4C is unstable at room temperature and difficult to prepare. The constitutional binary phase-diagram of Mn-C contains three stoichiometric carbides ($Mn_{23}C_6$, Mn_5C_2, Mn_7C_3) at room temperature and two carbides (Mn_3C and hexagonal close-packed ε-Mn_4C_{1-x}) at high temperatures (Tang *et al*., 2018). We recently successfully obtained cubic Mn_4C powder at room temperature (Si *et al*., 2018a, 2018b, Yao *et al*., 2019). The Mn_4C powder shows unconventional thermo-enhanced magnetic properties at temperatures up to 590 K (Si *et al*., 2018a, 2018b). In this work, the X-ray diffraction data for the Mn_4C powder at room temperature were collected in detail.

II. EXPERIMENTAL

A. Synthesis

Commercial Mn and graphite in a nominal composition of $Mn_{4.23}C$ were melted using argon plasma. The high vapor pressure of Mn may result in Mn loss during melting at high temperatures. Excess Mn was added to compensate for Mn evaporation during the melting process. The products were ground into powder followed by repeated magnetic separation using a magnet.

B. Powder data collection and processing

A small amount of the powder collected using the magnet was mounted on a glass specimen holder. The X-ray powder diffraction patterns were recorded with a Rigaku D/max 2500 diffractometer equipped with a Cu-target X-ray tube (40 kV and 30 mA). A divergence slit of 5 mm was used. The scan range was $2\theta = 25°$–$130°$ with a step size of 0.01° and a scan speed of 5° min^{-1}. The data were collected at room temperature (~300 K). MDI JADE 6.5 software was used to eliminate the $K\alpha_2$ component. The top of the peaks was used to determine the peak positions and intensities of the diffraction peaks (Table I). The *d*-values were calculated using Cu$K\alpha_1$ radiation ($\lambda = 1.5406$ Å). Rietveld refinements were performed with the FULLPROF package (Rodriguez-Carvajal, 2001).

III. RESULTS AND DISCUSSION

The X-ray powder diffraction patterns of the Mn_4C powder are shown in Figure 1, which are quite similar to the powder diffraction patterns of Mn_4N, as seen in PDF 01-089-4804 (ICDD, 2010), indicating similar structure of Mn_4C to that of Mn_4N. Therefore, most of the reflection peaks could be indexed with cubic perovskite-type Mn_4C with lattice parameters of $a = 3.8726$ Å, unit-cell volume V = 58.1 Å^3, and space group $Pm\bar{3}m$ (No. 221). The lattice parameter of one Mn_4C sample has been estimated to be 3.8682 Å in our previous work (Si *et al*., 2018a). We speculate that the stoichiometry of Mn/C may to some extent have an effect on the exact lattice parameters. The Mn:C ratio of the precursor in this work is 4.23:1, which is slightly larger than that of our previous work (Si *et al*., 2018a, 2018b). It seems the precursor stoichiometry has an effect on the stoichiometry of Mn_4C and thus the lattice parameters. Of course, the evaporation loss variation of Mn during high-temperature melting would affect the stoichiometry of the final product Mn_4C. Several weak diffraction peaks were indexed with Mn (PDF 32-0637) and $Mn_{23}C_6$ (PDF 28-0646) impurities (ICDD, 2010), which are more

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TABLE I. X-ray powder diffraction data for Mn_4C, which shows a lattice parameter $a = 3.8726$ Å, unit-cell volume $V = 58.1$ Å^3, and space group $Pm\bar{3}m$.

$2\theta_{obs}$ (deg.)	d_{obs} (Å)	$(I/I_0)_{obs}$	h	k	l	$2\theta_{calc}$ (deg.)	d_{calc} (Å)	$\Delta 2\theta$ (deg.)
32.650	2.7394	28	1	1	0	32.675	2.7383	0.025
40.310	2.2347	1000	1	1	1	40.304	2.2358	−0.006
46.901	1.9349	675	2	0	0	46.883	1.9363	−0.018
52.878	1.7294	21	2	1	0	52.817	1.7319	−0.061
58.280	1.5813	23	2	1	1	58.316	1.5810	0.036
68.490	1.3684	348	2	2	0	68.470	1.3692	−0.02
73.279	1.2903	12	3	0	0	73.271	1.2909	−0.008
77.881	1.2252	38	3	1	0	77.952	1.2246	0.071
82.550	1.1673	278	3	1	1	82.554	1.1676	0.004
87.131	1.1173	82	2	2	2	87.107	1.1179	−0.024
91.640	1.0738	18	3	2	0	91.642	1.0741	0.002
96.050	1.0358	23	3	2	1	96.188	1.0350	0.138
105.380	0.9682	54	4	0	0	105.429	0.9681	0.049
110.109	0.9395	12	3	2	2	110.192	0.9392	0.083
115.170	0.9122	15	4	1	1	115.106	0.9128	−0.064
120.220	0.8883	113	3	3	1	120.227	0.8884	0.007
125.631	0.8658	128	4	2	0	125.630	0.8659	−0.001

Only the peaks in the range of $2\theta = 25°$–$130°$ are presented. The d-values were calculated using Cu$K_{\alpha 1}$ radiation ($\lambda = 1.5406$ Å).

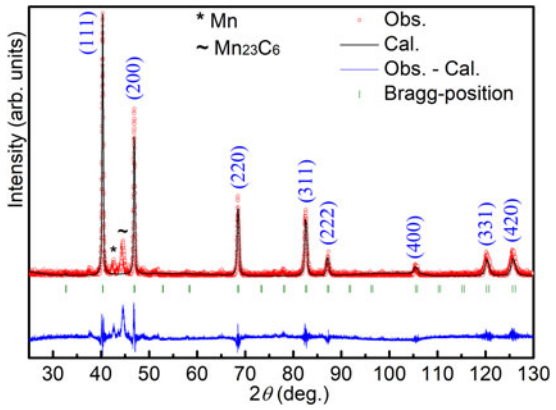


Figure 1. (Color online) X-ray powder diffraction patterns of Mn_4C using Cu$K\alpha$ radiation.

stable than Mn_4C and are difficult to remove completely by magnetic separation. The presence of these impurities has also been observed in our previous work. The X-ray powder diffraction data for the compound are given in Table I.

SUPPLEMENTARY MATERIAL

The supplementary material for this article can be found at https://doi.org/10.1017/S0885715619000265

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CONFLICTS OF INTEREST

The authors have no conflicts of interest to declare.

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