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Synthesis and tribological properties of high purity Ti_2SC nanolamellas by microwave hybrid heating

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ABSTRACT

Nearly pure Ti_2SC powder has been synthesized by microwave hybrid heating a mixture of TiS_2, Ti and C powders using Al powder as an additive. The recipes with different mole ratios of TiS_2:Ti:C:Al were examined at temperatures from 800 to 1050 °C. The purity of Ti_2SC was sensitive to the final temperature and raw material scale, and the addition of Al considerably reduced the calcining temperature. The nearly pure Ti_2SC powder can be fabricated reliably on a large scale by sintering the raw materials with a mole ratio of $1.1TiS_2$:2.9Ti:1.9C:0.3Al at temperatures from 900 to 1050 °C. The tribological properties of Ti_2SC as an additive in 150SN base oil were evaluated by a MMW-1A four-ball friction and wear tester. The results showed that under determinate conditions, the base oil containing 3 wt% Ti_2SC sample presented good tribology performance under a load of 300 N. The improved tribological properties of the Ti_2SC sample could be attributed to the formation of a tribofilm in the friction process.

1. Introduction

Ti_2SC, belongs to a special group of materials known as $M_{n+1}AX_n$ phases (where n = 1, 2, or 3, M is an early transition metal, A is a IIIA or IVA element and X is C or N, abbreviated as MAX), which are lately of higher interest because of their unique properties of ceramics and metals [1–5]. Among them, Ti_2SC, is a member of the novel 211 ternary compounds which exhibits outstanding ceramic properties such as a low density [6], high Vickers Hardness (8 ± 2 GPa) and Young's modulus (316 ± 2 GPa) [7]. Meanwhile, Ti_2SC possesses metallic properties including high electrical and thermal conductivities [8], good damage tolerance and easy machinability.

To date, several methods including hot pressing [7–12], spark plasma sintering [6], and so on have adopted to synthesize bulk Ti_2SC. However, the synthesis of high purity Ti_2SC powder was rarely reported. Recently, Chen et al. utilized Iron Disulfide or Iron Sulfide to synthesize Ti_2SC at 1500 °C in argon by Pulse-Electric-Current-Aided [13]. Zhu et al. prepared Ti_2SC powder by pressureless heating a mixture of TiS_2, Ti and C at 1600 °C for 2 h in argon [12]. Li and Liang et al. used S, Ti and C as starting materials to synthesize Ti_2SC powder at 2125 °C and 2410 °C by combustion synthesis [14,15]. These synthesis processes, however, usually require high energy ball milling and certain sintering equipment requirements, which lead to energy and time consuming, complicated productive process and low production efficiency [12–15].

Recently, microwave heating method, as a new route, could well supply the homogeneous heating in the sintered materials during the volumetric heating process, which has aroused researcher's interest because of its advantages, such as rapid heating rate, uniform heating, selective energy absorption and simple operating. Presently, highly purity Ti_3SiC_2 and Ti_3AlC_2 powders have been successfully synthesized by microwave heating [16,17]. Hence, it is reasonable to expect that the microwave heating method can provide a new approach to fabricate high-purity Ti_2SC powder. In addition, it was found that Ti_2SC has an excellent tribological property [11]. The relatively low coefficient and wear rate were obtained owing to the crystalline tribofilms formed on both contact surfaces. To the best of our knowledge, little work focuses on the tribological applications of Ti_2SC as lubrication additive.

In this work, microwave hybrid heating was introduced to synthesize highly pure Ti_2SC powder using $Ti/C/TiS_2/Al$ system. The tribological properties of Ti_2SC samples as additives in the 150SN base oil were also studied.

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2. Experimental procedure

The elemental Al was used first as an additive to synthesize high purity Ti_2SC powder. It is conceivable that the Al with a low melting point (663.4 °C) can form a liquid condition, which is able to induce a part of the raw materials to react beforehand. On the other hand, the Al may prevent S from entering into the Ti_2SC crystal lattice in terms of the attribute of the ternary carbides ($M_{n+1}AX_n$ phases, where n = 1, 2, or 3, M is an early transition metal, A is a IIIA or IVA element and X is C or N) [1].

Powders of Ti (99%, 200 mesh), S (99.5%, 100 mesh), graphite (98%, 100 mesh) and Al (99.9%, 100 mesh) were used as starting materials. Firstly, TiS_2 was obtained by heating the mixture of 1Ti:2.05S in the vacuous borax glass tubes at 600 °C for 12 h. Then, the as-synthesized TiS_2, Ti, C and Al powders with different molar ratios were mixed and compacted using a cylindrical steel die (diameter of 8 mm). The compacts were put in a small alumina crucible covering by thermal insulation materials to reduce the heat dissipation which was placed in the middle of large alumina crucible filled with microwave absorption material. The self-designed reactor which can be found elsewhere [18] was heated in a multimode microwave furnace (equipment type: RWS-3, frequency 2.45 GHz, maximum power of 3 kW) at 800–1050 °C (60 °C/min) for 3 min in Ar atmosphere. Finally, the samples were cooled to room temperature under Ar atmosphere, and the powders were obtained by crushing the sintered block.

Different mass fractions of the as-synthesized Ti_2SC powder from $1.1TiS_2$:2.9Ti:1.9C:0.3Al sintered at 1050 °C were dispersed in 150SN base oil *via* 3 h ultrasonication without any active reagent, and then the suspended oil samples were obtained. The tribological properties of the samples were measured using a MMW-1A four-ball friction and wear tester. The testing of friction reduction and wear resistance was conducted at rotating speeds of 600 r/min, and at a constant load of 300 N. The balls with a diameter of 12.7 mm used in the tests were fabricated from a GCr15 bearing steel, hardness of 61 HRC. The friction coefficient was recorded automatically with a strain gauge equipped with the tester. After the four-ball testing, the balls were cleaned using petroleum ether in an ultrasonic bath for 10 min, and stoved at 50 °C for 10 min.

The phase composition of synthesis samples was identified with X-ray diffraction experiments conducted on a Rigaku D/max-Ultima IV X-ray diffractometer using Cu Kα radiation. A scanning electron microscopy with an energy dispersive spectroscopy (SEM, XL30 S-FEG, Japan) was used to investigate the morphology of synthesized Ti_2SC sample and the wear scars. The wear scars widths were determined by an optical microscope (BH200M, China).

3. Results and discussion

3.1. Synthesis of Ti_2SC

Fig. 1 shows the results of XRD analyses for the samples microwave sintered at 1050 °C for 3 min with the different amount of additive Al. It was found that Ti_2SC, as the major phase, with large amount of TiC, TiS and graphite was formed in the sample with no additive Al shown in Fig. 1(a). As different amount of additive Al was added in the composition, the content of impurities (TiC, TiS and graphite) decreases with increase in additional Al. When the amount of additive Al was equal to 30 at% (Fig. 1d), purity Ti_2SC was obtained together with small amount of TiC and trace amount of graphite. Further increasing the amount of additive Al more than 30 at% (Fig. 1e), the content of impurities (TiC, TiS and graphite) increased which contributed little to the productivity of Ti_2SC. So the amount of additive Al to be 30 at% was enough for sintering of sample.

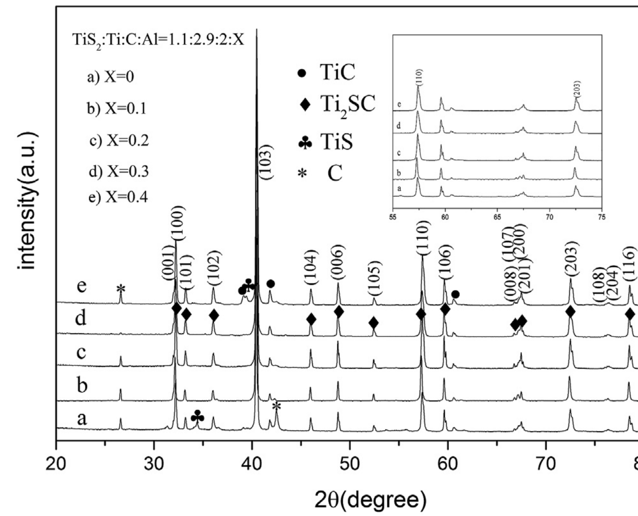

Fig. 1. XRD patterns of samples with different amount of additive alumina sintered by microwave. hybrid heating at 1050 °C for 3 min at Ar atmosphere. The inserted image was an enlarged part of the XRD pattern in the range of 55°–75° (2θ).

One can see from the inserted image that (110) and (203) peaks of Ti_2SC shifted towards a low-angle direction when amount of additive Al was 10 at% (b). According to the Bragg's law, some increase in the lattice parameters of Ti_2SC should be caused, indicating the possibility of Al atoms diffusing into the lattices of Ti_2SC. When amount of additive Al was more than 10 at% (c-e), slight shifts of the (110) and (203) peaks towards a large-angle direction were found which suggested Al atoms substituted for S atoms in Ti_2SC and formed a solid solution between Ti_2SC and Ti_2AlC. It was confirmed by calculating the lattice parameter with the approximate a_0 value of about 3.06 of $Ti_2S_xAl_yC$ solid solution from Vegard's law: $a_{Ti_2S_xAl_yC} = xa_{Ti_2SC} + ya_{Ti_2AlC}$. The proportion of $x = 0.82$, $y = 0.14$ (atom fraction) can be obtained by EDS in Fig. 6h, and the lattice parameters of Ti_2SC and Ti_2AlC could be found in literature [1].

In Fig. 1, the TiC impurity always existed, and its content increased when the amount of additive Al was equal to 40 at% (In Fig. 1e). With increasing temperature, the amount of TiC impurity also increased as shown in Fig. 2, thus, a highly pure Ti_2SC product cannot be obtained. The recent investigation [12] has shown that the reaction between Ti and C led to the formation of TiC. Probably, reducing the content of C in the starting materials may be favorable for the synthesis of high-purity Ti_2SC. Thus, the content of C in the starting materials would be investigated in subsequent experiments.

XRD patterns of samples with different amount of additive graphite in the composition synthesized at 1050 °C for 3 min are shown in Fig. 3. When the powder ratio was $1.1TiS_2$:2.9Ti:2C:0.3Al, except Ti_2SC, TiC and un-reacted graphite were also observed. The amount of impurities (TiC and graphite) decreased gradually with decrease in graphite in the composition. As the powder ratio was $1.1TiS_2$:2.9Ti:1.9C:0.3Al, Ti_2SC was found to be main crystalline phases, and graphite was presented as minor phase. With further decreasing the amount of graphite (Fig. 3d), however, contents of TiS and TiC phases were both increased while the intensity of Ti_2SC peaks was getting weaker. Based on the results mentioned above, further work focused on the samples with the molar ratio of TiS_2:Ti:C:Al = 1.1:2.9:1.9:0.3.

As shown in Fig. 4, specimen was synthesized by microwave hybrid heating using $1.1TiS_2$:2.9Ti:1.9C:0.3Al powders mixtures at