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The effect of La on the synthesis and properties of a W-Ti alloy



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HIGHLIGHTS

- Synthesis of W-Ti alloy dispersed with in-situ La oxides is developed.
- Formation of La oxides can be influenced by secondary ball milling.
- A complex LaTiO₃/TiO₂ oxide is found as interaction of La₂O₃/TiO₂.
- · Material behavior is strongly affected by La-Ti-O oxides.

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ABSTRACT

A new method for the synthesis of tungsten-titanium alloys dispersed with in-situ RE-oxides was developed. In this paper, the addition of the rare earth element lanthanum in W-Ti alloys has been investigated. The results suggested that La plays an important role in influencing the formation of dispersed oxides, which can further change the microstructural evolution and mechanical properties of the W-Ti alloys. A complicated oxide particle was found in this study and has been identified as the LaTiO₃/TiO₂ structure, which could be associated with the interaction between La₂O₃ and TiO₂ during mechanical alloying. Nanoindentation was further used to measure local variations in the mechanical properties of the alloys, which could correspond to interfaces between the dispersed oxides and the tungsten matrix where the bond strengths are different form the bulk alloy.

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1. Introduction

Tungsten is one of candidates for use as plasma facing materials (PFMs) in future fusion reactors such as the divertor of ITER due to its high melting point, reduced activation, high sputtering resistance, high thermal conductivity, low thermal expansion and high temperature stability [1–4]. During plasma operation, intense thermal loads and transient events can generate a strong impact on the surface of plasma facing materials that cause the deterioration of materials. In addition, one of the major drawbacks of tungsten is its inherently high ductile-brittle transition temperature (DBTT). Therefore, it is highly important to develop new tungsten alloys with enhanced mechanical properties and reliable fabrication processes for a safe and longer service life of plasma facing materials. Among all tungsten alloys there has been a growing interest in W-Ti alloys. An addition of Ti as an alloying element to tungsten can change the dislocation core structure which lowers the ductile-brittle transition temperature (DBTT) and improves

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the Charpy impact energy [2]. It has also been proved that Ti addition can promote densification and grain refinement of tungsten materials [3,4]. In the past few years, nano-scale oxide dispersed particles are intentionally introduced into tungsten alloys where the interfaces between matrix and oxide particles act as sinks for the irradiation-induced defects [5,6]. A number of researchers have conducted studies in tungsten alloys dispersed with different types of nanoscale oxides or refractory carbides e.g. Y₂O₃, La₂O₃, Y₂Ti₂O₇ and TiC [7-11]. The presence of nano-dispersoids does not only play a role on irradiation resistance but also is important in determining high temperature strength, creep resistance, and ductility [12–16]. Mechanical alloying is an essential process for fabrication of oxide dispersion strengthening (ODS) alloys. One of the major problems is oxygen contamination during the milling process. It can be expected that the milled powders can be easily oxidized and reduce the ductility and toughness of the final products. In earlier work [17,18], we investigated W-Ti alloys dispersed with Y₂O₃ and Y₂Ti₂O₇ oxide particles. The results suggested that Ti can be oxidized during the ball milling process and forms a large number of TiO₂ oxide particles in the tungsten matrix. A complex TiO₂/Y₂Ti₂O₇ oxide structure is also observed. Therefore, further studies are necessary to take into account the reduction of oxygen

Table 1The detail of W, Ti and La metal powders.

Material	Average particle size	Purity
W	$2-6\mu m$	>99.95%
Ti	$\sim 48\mu m$	>99.90%
La	$\sim 48~\mu m$	>99.90%

contamination during processing. Rare earth elements have a high affinity with oxygen and most likely form in-situ RE oxides during mechanical alloying. The fine in-situ oxides can have good lattice coherence with the matrix and greatly increase the mechanical properties at elevated temperature and enhance irradiation resistance [19–21]. The effect of rare earth elements on pure tungsten has been reported previously [22] but information is very limited for different tungsten alloy systems. Therefore, in this paper, we further investigated the effect of the rare earth element La on microstructural evolution and mechanical properties of W-Ti alloys. A secondary ball milling method was also introduced in this work to understand the effect of La on the synthesis of the alloys. The focus of the present work is to develop a new method to synthesize W-Ti ODS alloys dispersed with in-situ La oxides. It is also important to clarify the role of La in the W-Ti alloy system and interaction of the La oxides with tungsten matrix.

2. Experimental procedure

Pure W, Ti and rare earth element of La powders used as the starting materials supplied by Gredmann Group, Taiwan. The detail of each element can be seen in Table 1. The material used in the present study is of composition W-2Ti-2La (in weight percent). Initially, the elemental powders of tungsten, titanium and lanthanum were milled for $4\,h,\,8\,h,\,16\,h$ and $24\,h$. The mechanical alloying process was carried out using a planetary ball mill (Retsch PM 100) with a speed of $350\,\mathrm{rpm}$ under an argon atmosphere. In the high-energy

ball mill, severe plastic deformation of different metallic powders leads to a rapid interaction between the Ti and La powders and this most likely forms intermediate phases during processing. Therefore, in this study, a secondary ball milling method was introduced by involving a two-step milling procedure. First, the starting materials of tungsten and titanium powders were ball milled for 4 h and 8 h. In this stage, it is expected that the Ti powders were dissolved into the W matrix as a solid solution element. The pre-milled powders of the W-Ti alloy system for 8 h of milling were then further secondarily ball milled with the rare earth element of the La powder for additional 4 h, 8 h, and 16 h. The two model alloys with different procedures will then be referred to as W2T2L-P (primary milling) and W2T2L-S (secondary milling). Milling experiments were carried out using a tungsten carbide grinding medium with a ball to powder ratio of 10:1. The mechanically alloyed powders were consolidated into green compacts with a pressure of 350 MPa and then were further sintered in a mixed hydrogen-argon atmosphere at 1723 K for 30 min. Microstructure evolution and phase characterization of the W-Ti model alloys with different milling times were examined using an X'PERT PRO X-ray diffractometer (XRD) and a Hitachi-4700 Scanning electron microscope (SEM). A further analysis of the complex dispersed oxides and phase identification was performed on a FEI Tecnai F20 G2 Field Emission Gun Transmission electron microscope (TEM). Nanoindentation tests (MTS Nanoindenter XP) were conducted to obtain the elastic modulus and hardness using the continuous stiffness measurement (CSM) method

3. Results and discussion

3.1. Characterization of the W-Ti-La alloyed powders

Fig. 1 shows the XRD spectra of the W2T2L-S powder sample as a function of milling time. For the sample with the secondary ball

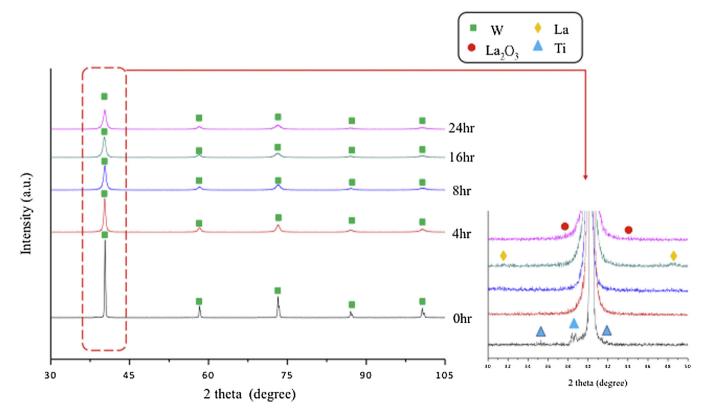


Fig. 1. XRD spectra of the W2T2L-S powders milled for different milling durations: 0 h, 4 h, 8 h, 16 h and 24 h.

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